The Effects of Particle Mesh and Temperature on Pyrolysis Spirulina platensis Residue (SPR): Pyrolysis Yield and Bio-Oil Properties			
Tanggal	Author	Editor	Bukti
28-Sep-21	SUBMIT ACKNOLEDGMENT	SUBMIT ACKNOLEDGMENT	Lampiran 1
01-Apr-22		REVIEW REVISE	Lampiran 2
15-Apr-22		Diminta mengisi response to reviewer	Lampiran 3
15-Apr-22		Response to Reviewer	Lampiran 4
15-Apr-22	Response to Reviewer		Lampiran 5
23 Mei 2022		Diminta REVISE 2-PLAGIARISME	Lampiran 6
23 Mei 2022	Upload revisi ke 2		Lampiran 7
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15 Juni 2022		Accepted	Lampiran 9
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26 Juni 2022		[AJChE] Copyediting Completed	Lampiran 11
27 Juni 2022		COPY EDIT REQUEST	Lampiran 12
28 Juni 2022		[AJChE] Copyediting Review Acknowledgement	Lampiran 13
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30 Juni 2022		[AJChE] New Issue Published and the recent Quartile of ASEAN J Chem Eng	Lampiran 15
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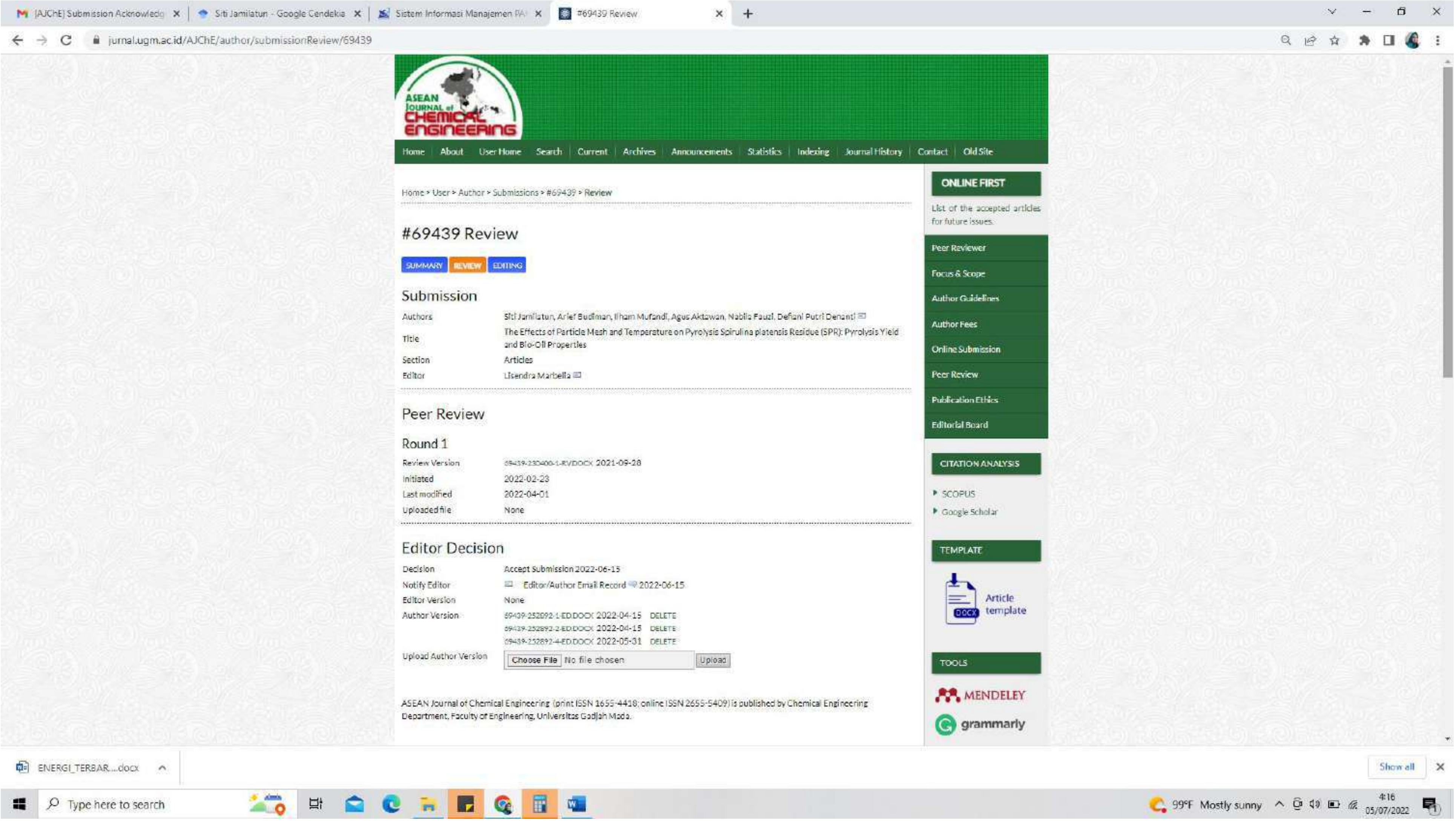
LAMPIRAN 1: SUBMIT ACKNOLEDGMENT



[AJChE] Submission Acknowledgement

Lisendra Marbelia, S.T., M.Sc., Ph.D. <ajche.ft@ugm.ac.id>
kepada saya

Sel, 28 Sep 2021 17.13

Siti Jamilatun:

Thank you for submitting the manuscript, "Effect of Particle Size and Temperature on Pyrolysis Spirulina Platensis Residue (SPR): Pyrolysis Yield and Bio-Oil Properties" to ASEAN Journal of Chemical Engineering. With the online journal management system that we are using, you will be able to track its progress through the editorial process by logging in to the journal web site:

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Lisendra Marbelia, S.T., M.Sc., Ph.D.
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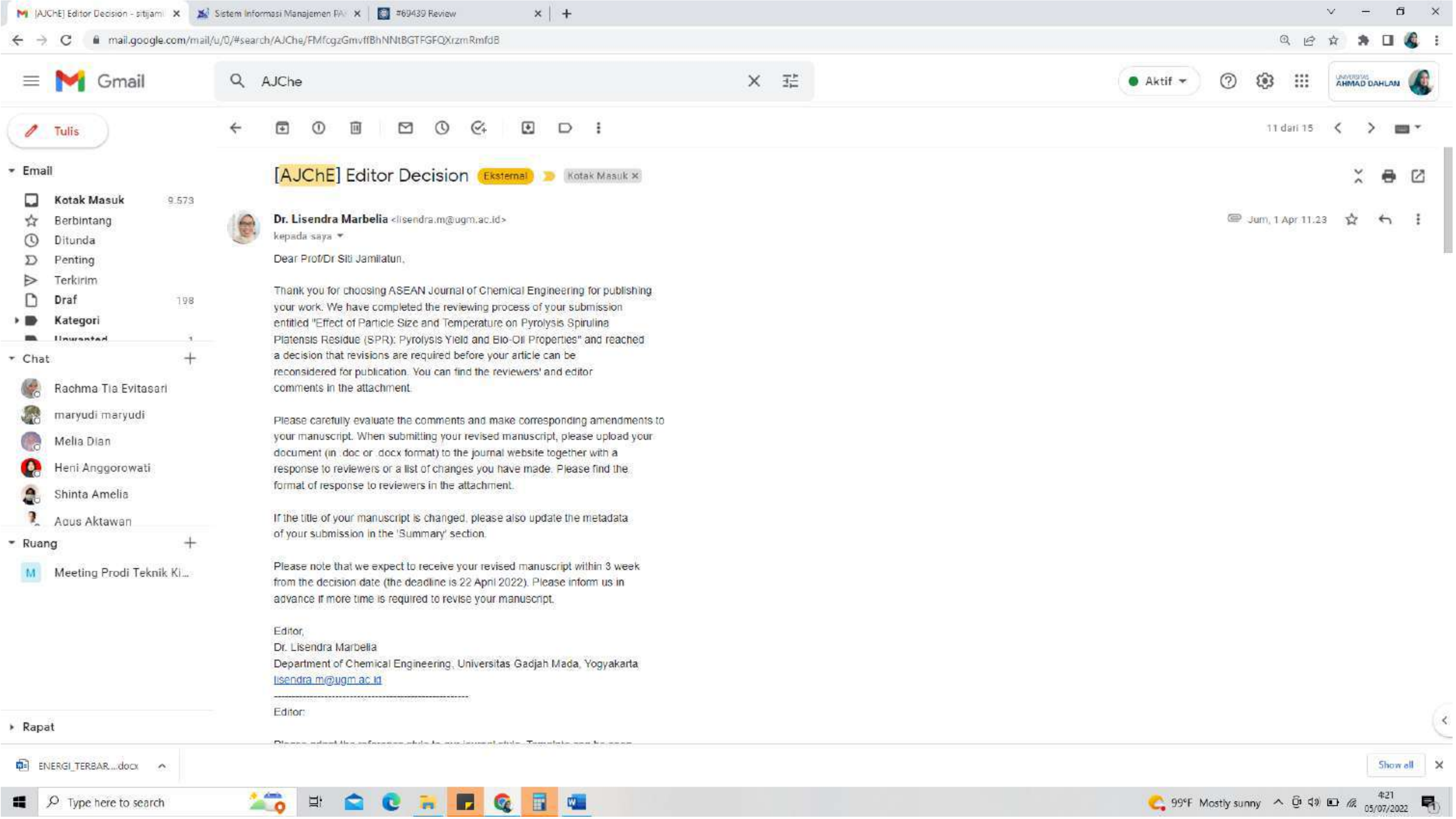
LAMPIRAN 2- HASIL REVIEW REVISE



[AJChE] Editor Decision

Dr. Lisendra Marbelia <lisendra.m@ugm.ac.id>
kepada saya
Jum, 1 Apr 11.23

Dear Prof/Dr Siti Jamilatun,

Thank you for choosing ASEAN Journal of Chemical Engineering for publishing your work. We have completed the reviewing process of your submission entitled "Effect of Particle Size and Temperature on Pyrolysis Spirulina Platensis Residue (SPR): Pyrolysis Yield and Bio-Oil Properties" and reached a decision that revisions are required before your article can be reconsidered for publication. You can find the reviewers' and editor comments in the attachment.

Please carefully evaluate the comments and make corresponding amendments to your manuscript. When submitting your revised manuscript, please upload your document (in .doc or .docx format) to the journal website together with a response to reviewers or a list of changes you have made. Please find the format of response to reviewers in the attachment.

If the title of your manuscript is changed, please also update the metadata of your submission in the 'Summary' section.

Please note that we expect to receive your revised manuscript within 3 week from the decision date (the deadline is 22 April 2022). Please inform us in advance if more time is required to revise your manuscript.

Editor,
Dr. Lisendra Marbelia
Department of Chemical Engineering, Universitas Gadjah Mada, Yogyakarta
lisendra.m@ugm.ac.id

Editor:

[AJChE] Editor Decision - sitijami | Sistem Informasi Manajemen PA | #69439 Review

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lisendra.m@ugm.ac.id

Editor:

Please adapt the reference style to our journal style. Template can be seen in our website
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The research gap and novelty of the current paper is not very clear. Please elaborate the sentences/paragraph in the introduction part, to be able clearly state the novelty of the current study.
In the methodology, some sentences in the bio-oil properties need to be revised/rearranged.
Part a, measure of bio-oil density, this is using density bottle? Then density is mass divided by volume. Please simplify the sentence.
Part b, how color is investigated or recorded? By taking note and pictures?
Part c, just write the info on the pH meter brand.
Part d, is there any further info on the bio-oil flammability measurement techniques? References? Write it clearly.
Fig 7 and Fig 8 were shifted. Please check it and put high quality images for the figure.

Reviewer A:

Dear authors,

I have reviewed your manuscript. Your topic is interesting. I have some comments for your manuscript. Please check it in the word file attached.

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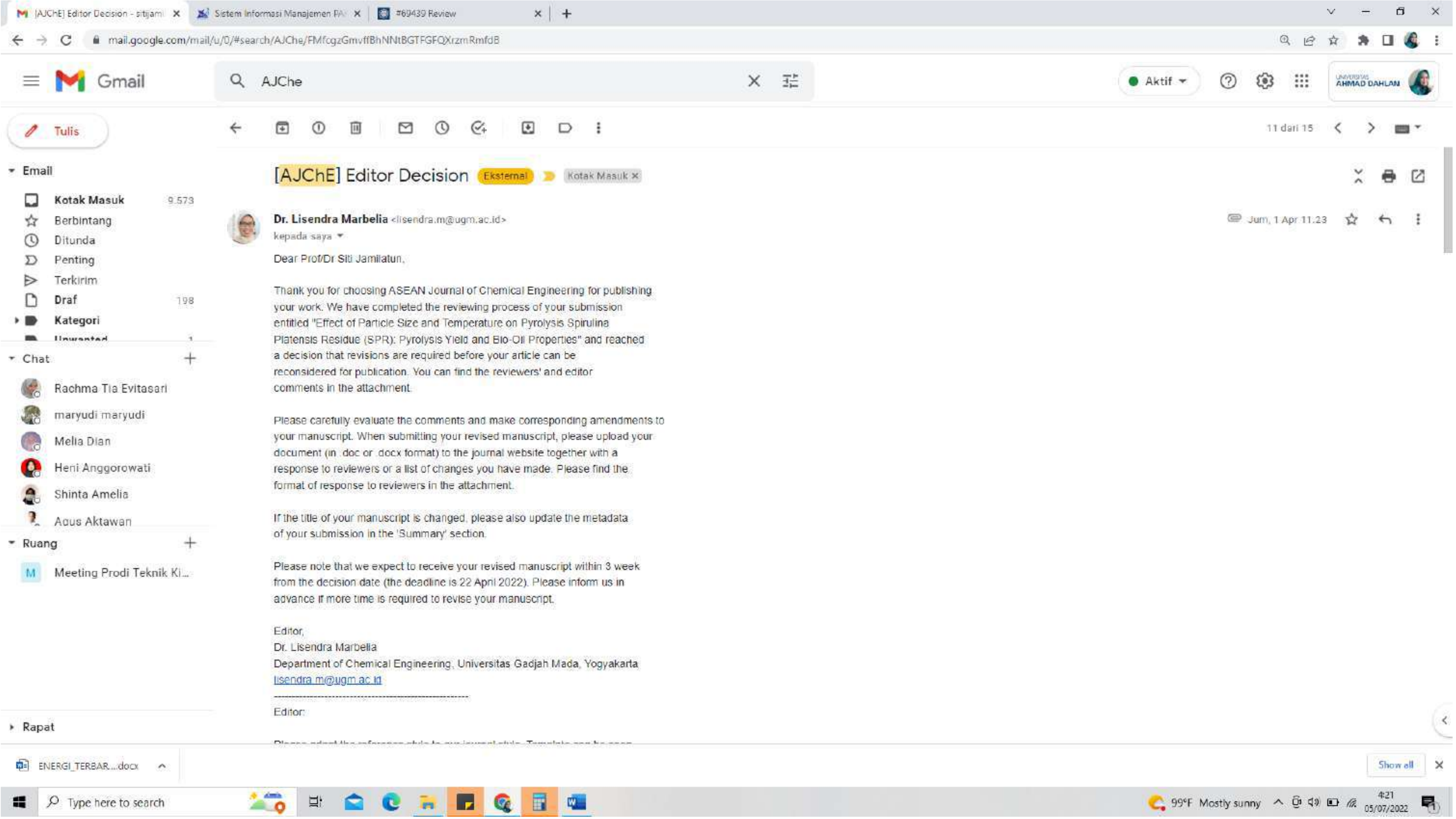
LAMPIRAN 2- HASIL REVIEW REVISE



[AJChE] Editor Decision

Dr. Lisendra Marbelia <lisendra.m@ugm.ac.id>
kepada saya

Jum, 1 Apr 11.23

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Please carefully evaluate the comments and make corresponding amendments to your manuscript. When submitting your revised manuscript, please upload your document (in .doc or .docx format) to the journal website together with a response to reviewers or a list of changes you have made. Please find the format of response to reviewers in the attachment.

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Editor,
Dr. Lisendra Marbelia
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Editor:

ISIChem 2021: The manuscript acceptance information_B4_AJChe

Eksternal | Kotak Masuk

isichem Tekkim <isichem@live.undip.ac.id>

kepada saya, aprilina

Sen, 20 Sep 2021 09.22

Dear. Prof/Dr/Sir/Mr/Mrs,

On behalf of the 2nd International Symposium on Chemical Engineering 2021 organizing committee, we would like to inform you that your manuscript has finished the review process. In accordance with the review results, we are pleased to inform you that **your manuscript has been accepted to be presented in 2nd ISIChem 2021 and selected to be published in ASEAN Journal of Chemical Engineering (AJChe)**. Through this letter, we would also like to deliver the review result.

High similarity rating: 27 % (It has to be improved, below 20%) - proof is attached
Figure 2-6 quality have to be improved (decide to use color, or black and white, and don't use outer box)
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Check the use of English. Proof-read again.

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Please do not hesitate to contact us if you need any further information.
Finally, we would like to take this opportunity to give great gratitude for your participation in the ISIChem 2021.

Best regards,
Conference chair
Dr. Aprilina

4 Lampiran

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Effect of Particle Size and Temperature on Pyrolysis *Spirulina Platensis Residue* (SPR): Pyrolysis Yield and Bio-Oil Properties

Microalgae is the third generation of biomass as renewable energy, a future energy source for making bio-oil. This study aims to examine the biomass from microalgae *Spirulina platensis residue (SPR)* using the pyrolysis process, to investigate the effect of particle size and temperature on the pyrolysis process, to determine the bio-oil properties, including density, pH, color, flame power, and conversion. A fixed bed reactor was installed with several configurations: outer diameter of 44 mm, inner diameter of 40 mm, and reactor height of 600 mm. The temperature processes are installed from 300-600°C combined with a 14-16 °C/minute heating rate. Fifty grams of SPR with the particle size variation (80 and 140 mesh) are delivered to the reactor. From the experiment results, the particle size 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, char, gas, water phase, pyrolysis, and *Spirulina platensis residue*.

INTRODUCTION

Microalgae is the third generation of biomass as renewable energy, a future energy source [1], [2]. In addition, microalgae can overgrow in wastewater with a high CO_2 level [3], dairy effluent [4] to reduce the cost of production, piggery wastewater [3], and can overgrow in open ponds [5]. Microalgae cultivation can reduce the greenhouse effect by using photobioreactors [6], [7]. There are many advantages of microalgae as follows: high lipid content [8], carbon hydrate [9], protein, and other chemicals [10], [11]. According to

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[12], the development of microalgae has many advantages over other biomass energy sources, such as a high rate of biomass production, not competing with food, and not requiring large areas of land for its growth. Microalgae contain lipids and fatty acids, which can be the essential ingredients of biofuels [13], [14]. Lipids produced from microalgae have C16 and C18, which can be esterified to produce high-quality biodiesel [15], [16]. The oil content in microalgae can exceed 80% by weight of dry biomass [17], [18].

As a renewable energy source, microalgae can be converted into three types: bio-oil, biochar, and non-condensable gas by using the pyrolysis process [19], [20]. The pyrolysis method is a process of thermal decomposition of organic matter at ambient temperature. There are high calorific values from microalgae such as Chlorella Vulgaris residue (24.57−35.10 MJ/kg), Spirulina platensis residue (20.46-33.62 MJ/kg), *Chlorella sorokiniana* CY1 residue (20.24 MJ/kg), and *Nannochloropsis Oceanica* residue (32.33−39 MJ/kg) [21]. As a comparison, these calorific values are higher than bio-oil produced from lignocellulose such as coconut shell (21.28 MJ/kg) and sugarcane bagasse [22]. Each pyrolysis product has its benefits if further processed; for example, bio-oil and gas can be used as marine material. The water phase is used as an additive for food preservation and contains components as a supplement. In contrast, char can be used as an adsorbent in food, sewage treatment, chemistry, etc.

The Making of bio-oil from microalgae by extraction produces solid residues that can be reused as a source of raw materials to produce biofuels [23]. This solid residue can be called *Spirulina platensis residue* (SPR), which contains high carbohydrates and protein. Using solid residues of microalgae of types with low lipid content, such as Spirulina sp. (4-9% lipids) as a raw material for pyrolysis is very beneficial because it can optimize the yield of bio-oil products by almost 40% [24]. The drawback of bio-oil from microalgae is the oxygenated and nitrogenated content is still high, which causes instability in its use.

Recently, many researchers have been conducted in the field of microalgae to convert a valuable product. [25] reported similar observations in their experiment from C. Vulgaris microalgae that containing: 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, heating rate, residence time, size and shape, catalyst, etc. [26]–[29]. The temperature factor is very crucial and essential because it can affect the composition of each product. Based on [30], the operating temperature increases beyond the optimum temperature, secondary cracks will occur, which causes a decrease in bio-oil production and an increase in gas production.

This research aimed to investigate particle size and temperature process's effect on pyrolysis from microalgae from the above explanation. The particle size of 80 mesh and 140 mesh was applied in the system, while the temperature was performed at 300, 400, 500, 550, and 600°C.

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The reactor was installed in a fixed-bed reactor with an outer diameter of 44 mm, an inner diameter of 40 mm, and a reactor height of 600 mm.

RESEARCH METHODOLOGY

Materials

This section summarizes the primary raw material of this research was obtained from solid residues of Spirulina platensis and extracted with methanol (CH_2OH). Based on a previous study [31], the ultimate and HHV from SPR were analyzed to know the characteristic of the SPR. 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%, carbon of 41.36%. The hydrogen of 6.60%, the nitrogen of 7.17%, and oxygen of 35.33%. HHV ~~(Higher Heating Value)~~ of SPR is 18.21 MJ/kg. SPR as raw material in this research was used 50 grams with the difference of size includes 140 mesh 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

Spirulina platensis residue (SPR) was obtained from the Chemical Engineering UGM Eco Mini Plant laboratory. SPR is mashed using ground or mashed using a blender. Then, sieving the SPR using a sieve with a size of 80 and 140 mesh. 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 [32,33]. 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 with an inner diameter of 40 mm, an outer diameter of 44 mm, and 600 mm. The temperature control was needed to know the relationship between temperature and time and the relationship between time and heating rate. The heater and thermocouple were used as the temperature control in this research.

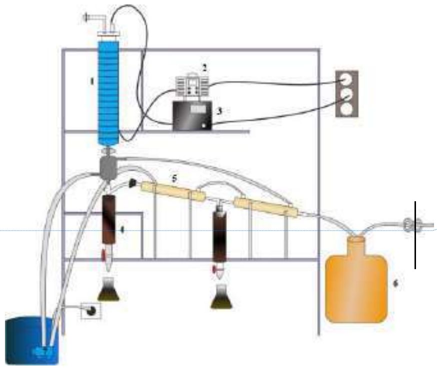

Fig. 1: Pyrolysis equipment

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 and 140 mesh. Then the reactor is

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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 natural materials, the reactor must be refrigerated. Repeat the process for SPR with a particle size of 140 mesh with a temperature range of 300–600ºC. 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:

$$Y_L = \frac{W_L}{W_M} x\ 100\ \% \quad \text{.....................} \quad (1)$$

$$Y_B = \frac{W_B}{W_M} x\ 100\ \% \quad \text{.....................} \quad (2)$$

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

$$Y_C = \frac{W_C}{W_M} x\ 100\ \% \quad \text{.......................} \quad (4)$$

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

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

$$X = \frac{(W_B+W_{WP}+W_G)}{w_M} x100\ \% \quad \text{......} \quad (7)$$

In this case, Y_L Y_B, Y_{WP}, Y_C Y_G and are the yields of the liquid, bio-oil, water phase, and gas, respectively; W_M, W_L, W_B, W_{WP}, and W_C are the weight of the SPR sample, the importance of the liquid product, bio-oil, water phase, and char, respectively, whereas X is conversion.

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, weigh the bottle that is filled with distilled water. Finally, calculate the density with the following formula:

$$\text{Volume of bottle} = \frac{(\text{Weight of bottle+distilled water})-(\text{weight of empty bottle})}{\text{Density of distilled water}}$$

$$\text{Density} = \frac{(\text{Product weight}-\text{empty bottle weight})-(\text{Empty bottle weight})}{\text{Bottle Volume}} \quad \text{..........} \quad (8)$$

b. Bio-oil color

The colors of bio-oil in this section are investigated and recorded

c. pH

A variety of tools have been used for measuring the pH of bio-oil. pH meter used to measure pH 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

The effect of temperature and particle size on bio-oil yield

A visual representation of this result can be seen in Figure 2. This researcher is carried out using a temperature process from 300-600ºC and particle-sized of 80 mesh and 140 mesh. The results of each

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experiment showed that the yield of bio-oil at a particle size of 80 mesh and in the temperature ranges: 300, 400, 500, 550, and 600ºC was 12.54, 19.62, 19.66, 13.96, and 14.12%. Meanwhile, the yield of bio-oil at 140 mesh and at processing temperatures: 300, 400, 500, 550, and 600ºC was 14.9, 22.24, 22.92, 15.18, and 15.66%. Based on these results, the bio-oil yield was influenced by the particles size SPR as a raw material in this research.

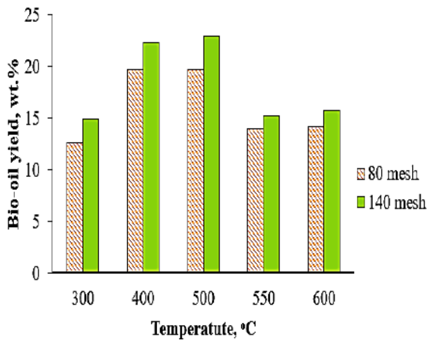

Fig. 2: Effect of temperature and SPR particle size on bio-oil yield

Based on Figure 3, During the reaction temperature process, the bio-oil increases from temperature 300 to 500ºC. The bio-oil yield decreases from temperature 500 to 600ºC. This condition takes place in both particle sizes of biomass 80 mesh and 140 mesh. The optimum reaction temperature of SPR occurs at temperatures 400 and 500ºC. This result is consistent with previous research [34]–[36]. 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. Following the experiment from [37], [38], the pyrolysis process increased with the increase of the thermal cracking reaction, which this condition made the increase in the gas and liquid production. The decomposition of biomass leads to the formation of phenol and CH_4 [39]. Hence this temperature, the maximum bio-oil is formed.

The influence of particle size on bio-oil yield is identified as the larger the particle size, the lower the bio-oil yields. This result is relevant with previous research from [40], who also reported the effect of particle size 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 influenced the increase of the particle size in the particles. At a certain time, 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. Small particles have sufficient surface area to interact with pyrolysis media to form volatile products, thus leaving the biomass matrix without experiencing secondary reactions [41]. Furthermore, it can be said that the particle size of the material is directly related to the area of the contact area, including affecting the heat transfer rate and mass.

The effect of temperature and particles sized on the water phase.

The effect of temperature and SPR particle size on the yield water phase can be seen in Figure 3. respectively. The water phase with a particle size of 80 mesh was obtained 21.08, 22.40, 23.10, 25.10, and

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26.06% with the temperature of 300, 400, 500, 550, and 600ºC, respectively. At the same time, the water phase with a particle size 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 greater the temperature, the water phase yield value increased; this is because the high temperature can convert the water phase that is in the SPR optimally [42]. Next, also secondary decomposition reactions occur when the pyrolysis temperatures are very high [27]. This result was relevant with [43], 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 500ºC. According to [44], the larger the SPR particle size used, the lower the yield water phase is obtained. Because the particle size 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.

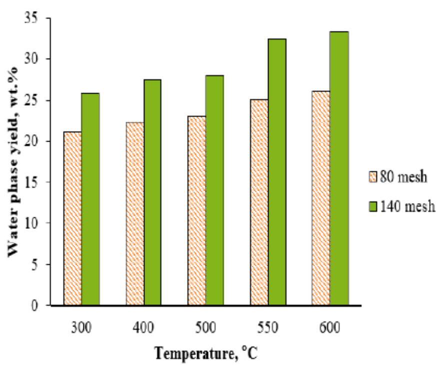

Fig. 3: Effect of temperature and SPR particle size on water phase yield

The effect of temperature and particles sized on char yield.

Figure 4 shows the effect of temperature and SPR particle size on biochar yield. The experiment result indicated that the yield of biochar with a particle size of 80 mesh was 39.14, 30.12, 27.90, 27.68, and 24.1%, respectively. In comparison, the biochar yield with a particle size 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 600ºC. In Figure 4, it can be seen that the biochar yield was gradually decreased when the temperature was too high and the particle of 80 mesh and 140 mesh. These results are relevant with [45]. In general, moisture and hydration water loss takes place at a temperature under 250ºC. In contrast, the temperature above 150ºC decompose and transform into a vapor containing organic compounds and gases [46], [47]. In addition, the decreasing biochar yield from low to high temperature was affected by organic matter decomposed when the pyrolysis was at high temperature [48].

The effect of particle size on char yield indicated that the char yield depended on particle size. The larger particle was not removed and need more time to heat transfer limitation between particle and supply heat. In addition, the larger biomass particle was support for the secondary charcoal [49]. This study is by [31] the more significant the SPR particle size used, the greater the biochar yield obtained. An increase in particle size was affected by the larger temperature gradient in the particles.

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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 [42], [50].

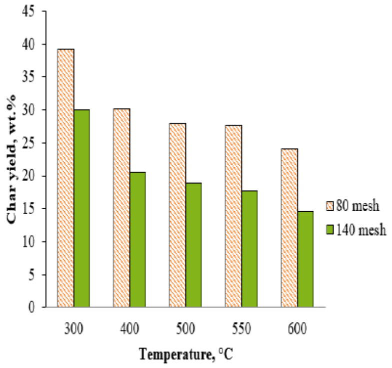

Fig. 4: Effect of temperature and SPR particle size on char yield

The effect of temperature and particles sized on gas yield

Figure 5 shows the effect of temperature and biomass particles on gas yield. The experiment indicated that the gas yield with a particle size of 80 mesh was 27.24, 27.86, 29.34, 33.26%, respectively. At the same time, the gas yield with a particle size 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 600ºC. In Figure 6, it can be seen that the higher the temperature, the greater the yield of gas. According to [50], 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 with [43], who reported the effect of biomass particles size from Chinese tallow trees. They were investigated that the small particle takes place reasonable heat transfer rate causing higher overall temperature. According to [51], the larger the SPR particle size used, the smaller the gas yield obtained. This phenomenon can occur because, at large particle sizes, the decomposition process is less than optimal due to the small contact area. It can affect the low speed of heat and mass transfer to convert SPR into gas.

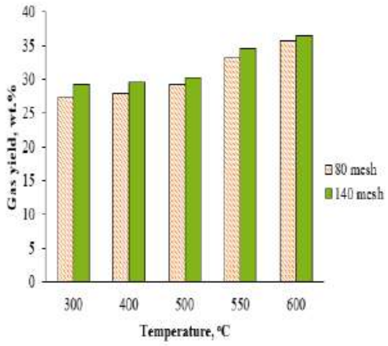

Fig. 5: Effect of temperature and SPR particle size on gas yield

Bio-oil conversion

Fig 6 shows the conversion of pyrolysis product with a particle size of 80 mesh and 140 mesh under temperature process at 300, 400, 500, 550, and 600ºC. The experiment results from the biomass particle size 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 size of 140 mesh with the same temperature set was 70.02, 79.42, 81.16, 82.28, and 85.44%, respectively. In Figure 7, it can be seen that the higher the temperature, the pyrolysis product conversion increased. This result was relevant with [52]. They reported the conversion in the heating rate of 10, 20, and 50ºC/min at a temperature ranging from 200 to 530ºC. The larger the SPR particle size used, the smaller the conversion of pyrolysis products obtained. From Figure 7, it can be concluded that the relationship between temperature, particle size, and conversion of pyrolysis products, namely an increase in pyrolysis temperature. A smaller SPR particle size causes thermal decomposition to work more effectively, decreasing the SPR weight.

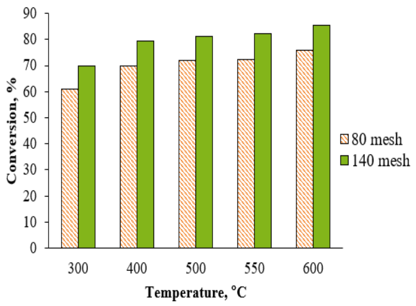

FIG. 6: Effect of temperature on various amounts of catalyst on conversion

Bio-Oil Spirulina platensis residue (SPR) properties: Density of Bio-oil

There are given in Table 1. the bio-oil density with particle size (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 which have a low percentage of heavy molecular substances, their density tends to be lighter. The chemical compound in the fuel with high molecular weight will complicate the evaporation process in the engine combustion chamber and tend not to burn completely. Previous research from [53] explained why bio-oil density is in the range of 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 fuel, namely the lower density, increased the ignition of the fuel. Hence the fuel easily burned because the calorific value was high [54].

Table 1. Density of bio-oil

Density of Bio-oil (gram/ml)		
Temperature (°C)	Particle size	(mesh)
	80	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 the different particles of 80 mesh and 140 mesh. 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 size of 80 mesh at a temperature of 550°C with a pH level of 8.9. In comparison, the particle size of 140 at a temperature of 550°C has obtained a pH level of 9.0. Relevant works by these researchers [20] are the leading methods on the pH of microalgae bio-oil at an alkaline pH of 8-9.9. In addition, previous research from [55] reported similar observations in bio-oil pH from *Chlorella Sp* of 9.33 of and *Nannochoropsis* of 9.93. The pH alkaline of bio-oil was affected by the presence of nitrogen in the bio-oil.

Commented [R33]: What kind of catalyst?

Commented [R34]: Please explain: what is the importance of measuring the bio-oil pH? What pH should bio-oil be?

There are several ways of improving the bio-oil, including hydrotreating, hydrocracking, steam reforming, and others where compounds with high molecular weight were split into alkane compounds [56]. [57] 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 bio-oil produced at a temperature of 300ºC – 400ºC, the flame power was relatively slow (slow), in contrast to the bio-oil produced at a temperature of 500ºC – 600ºC, its flame power was somewhat moderate (medium). 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 that 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 [54], [58]. The color of bio-oil from [58] was dark-brown, while the bio-oil from [54] 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.

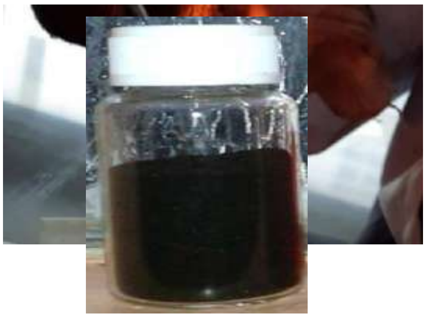

Fig. 8: Color of bio-oil

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 size greatly was affected the pyrolysis yield. The higher the pyrolysis temperature, the higher the conversion, and the bigger the particle size, the lower the yield of bio-oil, water phase, and gas for all temperatures tested. However, this is inversely proportional to the solid product, namely, char, where the larger the particle size, the greater the yield of char. The highest yield of bio-oil products was at 500ºC with a particle size of 140 mesh of 22.92%. The optimal temperature for SPR pyrolysis is at 500ºC. Product conversion is around 60-85%. The bio-oil product has properties such as a density of around 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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Response to Reviewer

Article Title: "The Effects of Particle Size and Temperature on Pyrolysis *Spirulina platensis* residue (SPR): Pyrolysis Yield and Bio-Oil Properties"

REVIEWER A

No.	Reviewer comment	Authors' response
1	Please explain the difference between char, charcoal and biochar	• When used as nouns, char means a charred substance. When used as verbs, char means to burn something to charcoal. • Biochar is a carbon-rich solid that is derived from biomass (organic matter from plants) that is heated in a limited oxygen environment. • Charcoal is also a carbon-rich solid that is derived from biomass in a similar manner. Charcoal is generally intended for heating or cooking, and is commonly associated with barbequing. Biochar and charcoal are made using a process called pyrolysis, in which the source material – in this case, the carbon-containing material – is exposed to high temperatures in the absence of oxygen so that it thermally decomposes into char or carbon-rich solids.
2	what is the importance of measuring the bio-oil pH? What pH should bio-oil be?	Physical properties such as density, pH, flammability are important parts to be analyzed, because with physical properties data we can assess and determine whether pyrolysis bio oil has the potential to be used as liquid fuel. pH is an important physical property to operate the machine, where the

		pH value of the biooil has the potential to cause corrosion in the engine. The low pH of biooil is not good for liquid fuel because it can accelerate the corrosion of the engine, so further treatment needs to be done. Previous research from [5] explained why bio-oil pH is in the range of 8-9
3		

REVIEWER B

No.	Reviewer comment	Authors' response

AJChE 20XX, Vol. XX, No. X, XX – XX

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

Microalgae is the third generation of biomass as renewable energy, a future energy source for making bio-oil. This study aims to examine the biomass from microalgae *Spirulina platensis* residue (SPR) using the pyrolysis process, to investigate the effect of particle size and temperature on the pyrolysis process, to determine the bio-oil properties, including density, pH, color, flame power, and conversion. A fixed bed reactor was installed with several configurations: outer diameter of 44 mm, inner diameter of 40 mm, and reactor height of 600 mm. The temperature processes are installed from 300-600°C combined with a 14-16 °C/minute heating rate. Fifty grams of SPR with the particle size variation (80 and 140 mesh) are delivered to the reactor. From the experiment results, the particle size and temperature process influenced by bio-oil yield, water phase, gas yield, bio char 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, bio char, gas, water phase, pyrolysis, and *Spirulina platensis* residue.

INTRODUCTION

Microalgae is the third generation of biomass as renewable energy, a future energy source [20], [49]. In addition, microalgae can overgrow in wastewater with a high CO_2 level [2], dairy effluent [15] to reduce the cost of production, piggery wastewater [2], and can overgrow in open ponds [30]. Microalgae cultivation can reduce the greenhouse effect by using photobioreactors [17], [35]. In a photobioreactor, photosynthesis of microalgae takes place. Where photosynthesis is the process of converting

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CO_2 in the air into O_2 with the help of light. The criterion by which the success rate of the process becomes is the amount of O_2 obtained. The greater the amount of O_2 produced, the more CO_2 is converted, and this indicates that the photosynthesis process in the photobioreactor is effective in reducing the concentration of CO_2 in the air to prevent global warming [17] [41]. There are many advantages of microalgae as follows: high lipid content [12], carbon hydrate [36], protein, and other chemicals [32], [53]. According to [11], the development of microalgae has many advantages over other biomass energy sources, such as a high rate of biomass production, not competing with food, and not requiring large areas of land for its growth. Microalgae contain lipids and fatty acids, which can be the essential ingredients of biofuels [26], [57]. Lipids produced from microalgae have C16 and C18, which can be esterified to produce high-quality biodiesel [7], [48]. The oil content in microalgae can exceed 80% by weight of dry biomass [34], [35].

As a renewable energy source, microalgae can be converted into three types: bio-oil, bio char, and non-condensable gas by using the pyrolysis process [5], [19]. The pyrolysis method is a process of thermal decomposition of organic matter at ambient temperature. There are high calorific values from microalgae such as *Chlorella vulgaris* residue (24.57−35.10 MJ/kg), *Spirulina platensis* residue (20.46-33.62 MJ/kg), *Chlorella sorokiniana* CY1 residue (20.24 MJ/kg), and *Nannochloropsis oceanica* residue (32.33−39 MJ/kg) [52]. As a comparison, these calorific values are higher than bio-oil produced from lignocellulose such as coconut shell (21.28 MJ/kg) and sugarcane bagasse [29]. Each pyrolysis product has its benefits if further processed; for example, bio-oil and gas can be used as marine material. The water phase is used as an additive for food preservation and contains components as a supplement. In contrast, bio char can be used as an adsorbent in food, sewage treatment, chemistry, etc.

The Making of bio-oil from microalgae by extraction produces solid residues that can be reused as a source of raw materials to produce biofuels [39]. This solid residue can be called *Spirulina platensis* residue (SPR), which contains high carbohydrates and protein. Using solid residues of microalgae of types with low lipid content, such as Spirulina sp. (4-9% lipids) as a raw material for pyrolysis is very beneficial because it can optimize the yield of bio-oil products by almost 40% [51]. The drawback of bio-oil from microalgae is the oxygenated and nitrogenated content is still high, which causes instability in its use.

Recently, many researchers have been conducted in the field of microalgae to convert a valuable product. [22] reported similar observations in their experiment from *Chlorella vulgaris* microalgae that containing: 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, heating rate, residence time, size and shape, catalyst, etc [10], [13], [18], [58]. The

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temperature factor is very crucial and essential because it can affect the composition of each product. Based on [40], the operating temperature increases beyond the optimum temperature, secondary cracks will occur, which causes a decrease in bio-oil production and an increase in gas production.

This research aimed to investigate particle size and temperature process's effect on slow pyrolysis of microalgae *Spirulina platensis* with range heating rate used is 14-16 °C/menit. The factors that affect the pyrolysis process of *Spirulina platensis* are reaction time, material size, pyrolysis temperature, heating rate, material content. [46]
.
The particle size of 80 mesh and 140 mesh was applied in the system, while the temperature was performed at 300, 400, 500, 550, and 600°C. The reactor was installed in a fixed-bed reactor with an outer diameter of 44 mm, an inner diameter of 40 mm, and a reactor height of 600 mm.

RESEARCH METHODOLOGY

Materials

This section summarizes the primary raw material of this research was obtained from solid residues of *Spirulina platensis* and extracted with methanol (CH_3OH). Based on a previous study [44], 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%, carbon of 41.36%. The hydrogen of 6.60%, the nitrogen of 7.17%, and oxygen of 35.33%. HHV of SPR is 18.21 MJ/kg. SPR as raw material in this research was used 50 grams with the difference of size includes 140 mesh 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

Spirulina platensis residue (SPR) was obtained from the Chemical Engineering UGM Eco Mini Plant laboratory. SPR is mashed by pounding or using a blender. Then, sieving the SPR using a sieve with a size of 80 and 140 mesh. 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 [4], [45]. 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 with an inner diameter of 40 mm, an outer diameter of 44 mm, and 600 mm. The temperature control was needed to know the relationship between temperature and time and the relationship between time and heating rate. The heater and thermocouple were used as the temperature control in this research.

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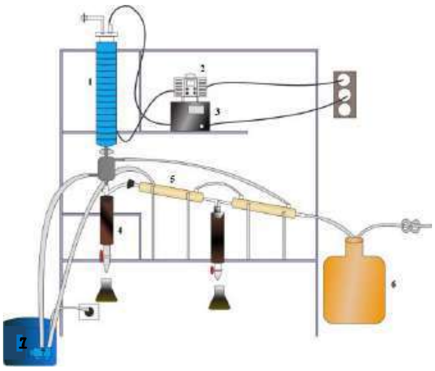

Fig. 1: Pyrolysis equipment

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 size of 140 mesh with a temperature range of 300–600ºC. 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:

$$Y_L = \frac{W_L}{W_M} x\ 100\ \% \qquad (1)$$

$$Y_B = \frac{W_B}{W_M} x\ 100\ \% \qquad (2)$$

$$Y_{WP} = \frac{W_{WP}}{W_M} x\ 100\ \% \qquad (3)$$

$$Y_C = \frac{W_C}{W_M} x\ 100\ \% \qquad (4)$$

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

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

$$X = \frac{(W_B + W_{WP} + W_G)}{W_M} x100\ \% \qquad (7)$$

In this case, Y_L, Y_B, Y_{WP}, Y_C, and Y_G are the yields of the liquid, bio-oil, water phase, bio char, and gas, respectively; W_M, W_L, W_B, W_{WP} and W_C are the weight of the SPR sample, the importance of the liquid product, bio-oil, water phase, and char, respectively, whereas X is conversion.

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, weigh the bottle that is filled with distilled water. Finally, calculate the density with the following formula:

$$\text{Density } (\rho) = \frac{\text{mass of biooil}}{\text{Volume of biooil}} \qquad (8)$$

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b. Bio-oil color

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

c. pH

Measurement of the pH of bio-oil using a pH meter by dipping the pH_meter into bio-oil. by dipping the pH

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

The effect of temperature and particle size on bio-oil yield

A visual representation of this result can be seen in Figure 2. This research is carried out using a temperature process from 300-600°C and particle-sized of 80 mesh and 140 mesh. The results of each experiment showed that the yield of bio-oil at a particle size 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 yield of bio-oil at 140 mesh and at processing temperatures: 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 particles size SPR as a raw material in this research.

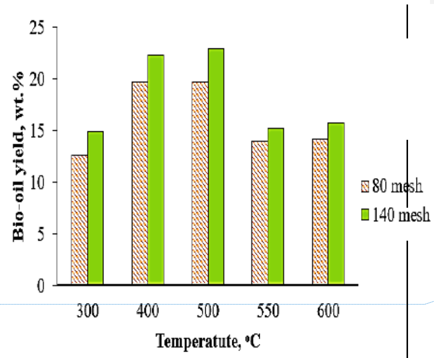

Fig. 2: Effect of temperature and SPR particle size on bio-oil yield

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 takes place in both particle sizes 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 [6], [31], [38]. 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. Following the experiment from [47], [54], the pyrolysis process increased with the increase of the thermal cracking reaction, which this condition made the increase in the gas and liquid production. The decomposition of biomass leads to the formation of phenol and CH_4 [42]. 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. [45].

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The influence of particle size on bio-oil yield is identified as the larger the particle size, the lower the bio-oil yields. This result is relevant with previous research from [21], who also reported the effect of particle size 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 of the particle size in the particles. At a certain time, the core temperature is lower than the surface of the biomass particles, which can cause an increase in bio char, while the production of gas and bio-oil decreases. Small particles have sufficient surface area to interact with pyrolysis media to form volatile products such us gas, thus leaving the biomass matrix without experiencing secondary reactions [1]. Furthermore, it can be said that the particle size 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 size, 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 larger 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 sizes.

The effect of temperature and particles sized on the water phase.

The effect of temperature and SPR particle size on the yield water phase can be seen in Figure 3. respectively. The water phase with a particle size of 80 mesh was obtained 21.08, 22.40, 23.10, 25.10, and 26.06% with the temperature of 300, 400, 500, 550, and 600ºC, respectively. At the same time, the water phase with a particle size 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 greater the temperature, the water phase yield value increased; this is because the high temperature can convert the water phase that is in the SPR optimally [46]. Next, also secondary decomposition reactions occur when the pyrolysis temperatures are very high [13]. This result was relevant with [9], 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 500ºC. According to [43], the larger the SPR particle size used, the lower the yield water phase is obtained. Because the particle size 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.

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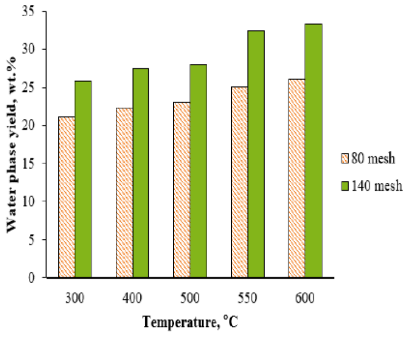

Fig. 3: Effect of temperature and SPR particle size on water phase yield

The effect of temperature and particles sized on bio char yield.

Figure 4 shows the effect of temperature and SPR particle size on bio char yield. The experiment result indicated that the yield of bio_char with a particle size of 80 mesh was 39.14, 30.12, 27.90, 27.68, and 24.1%, respectively. In comparison, the bio_char yield with a particle size 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 600ºC. In Figure 4, it can be seen that the bio_char yield was gradually decreased when the temperature was too high and the particle of 80 mesh and 140 mesh. These results are relevant with [56]. In general, moisture and hydration water loss takes place at a temperature under 250ºC. In contrast, the temperature above 150ºC decompose and transform into a vapor containing organic compounds and gases [23], [24]. In addition, the decreasing biochar yield from low to high temperature was affected by organic matter decomposed when the pyrolysis was at high temperature [14].

The effect of particle size on char yield indicated that the char yield depended on particle size. The effect of particle size on the yield of bio char shows that the yield of charcoal depends on the particle size. 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 In addition, the larger biomass particle was In addition, the larger biomass particle was support for the secondary charcoal [27]. This study is by [44] the more significant the SPR particle size used, the greater the biochar yield obtained. An increase in particle size was affected by the larger 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 [37], [46].

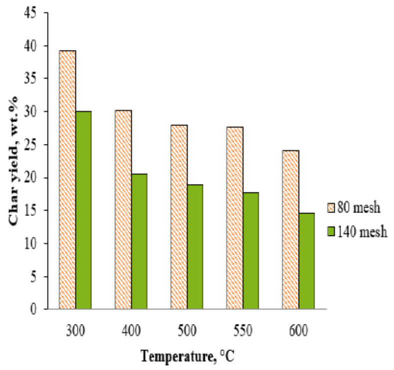

Fig. 4: Effect of temperature and SPR particle size on char yield

The effect of temperature and particles sized on gas yield

Figure 5 shows the effect of

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Commented [NF42R41]: In file response to reviewer temperature and biomass particles on gas yield. The experiment indicated that the gas yield with a particle size of 80 mesh was 27.24, 27.86, 29.34, 33.26%, respectively. At the same time, the gas yield with a particle size 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 600ºC. In Figure 6, it can be seen that the higher the temperature, the greater the yield of gas. According to [37], 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 with [9], who reported the effect of biomass particles size from Chinese tallow trees. They were investigated that the small particle takes place reasonable heat transfer rate causing higher overall temperature. According to [55], the larger the SPR particle size used, the smaller the gas yield obtained. This phenomenon can occur because, at large particle sizes, the decomposition process is less than optimal due to the small contact area. It can affect the low speed of heat and mass transfer to convert SPR into gas.

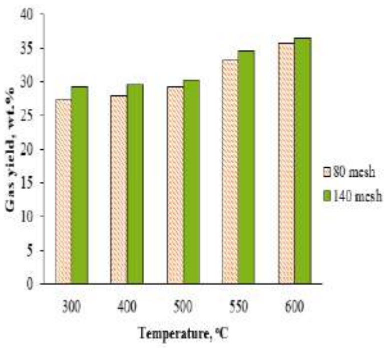

Fig. 5: Effect of temperature and SPR particle size on gas yield

Bio-oil conversion

Fig 6 shows the conversion of pyrolysis product with a particle size of 80 mesh and 140 mesh under temperature process at 300, 400, 500, 550, and 600ºC. The experiment results from the biomass particle size 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 size of 140 mesh with the same temperature set was 70.02, 79.42, 81.16, 82.28, and 85.44%, respectively. In Figure 7, it can be seen that the higher the temperature, the pyrolysis product conversion increased. This result was relevant with [33]. They reported the conversion in the heating rate of 10, 20, and 50ºC/min at a temperature ranging from 200 to 530ºC. The larger the SPR particle size used, the smaller the conversion of pyrolysis products obtained. From Figure 7, it can be concluded that the relationship between temperature, particle size, and conversion of pyrolysis products, namely an increase in pyrolysis temperature. A smaller SPR particle size causes thermal decomposition to work more effectively, decreasing the SPR weight.

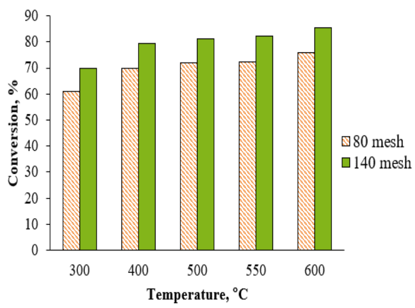

FIG. 6: Effect of temperature on various amounts of particle size on conversion

Bio-Oil Spirulina platensis residue (SPR) properties: Density of Bio-oil

There are given in Table 1. the bio-oil density with particle size (80 mesh and 140 mesh) and temperature settings of 300, 400, 500, 550, and 600ºC. 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 which have a low percentage of heavy molecular substances, their density tends to be lighter. The chemical compound in the fuel with high molecular weight will complicate the evaporation process in the engine combustion chamber and tend not to burn completely. Previous research from [53] explained why bio-oil density is in the range of 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 fuel, namely the lower density, increased the ignition of the fuel. Hence the fuel easily burned because the calorific value was high [16].

Table 1. Density of bio-oil

Density of Bio-oil (gram/ml)		
Temperature (ºC)	Particle size (mesh)	
	80	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 the different particles of 80 mesh and 140 mesh. The temperature process was set at 550ºC. This analysis was to know the pH level in bio-oil by using a pH meter. The experiment result found that the particle size of 80 mesh at a temperature of 550ºC with a pH level of 8.9. In comparison, the particle size of 140 at a temperature of 550ºC has obtained a pH level of 9.0. Relevant works by these researchers [5~~20~~] are the leading methods on the pH of microalgae bio-oil at ~~an alkaline~~ pH of 8-9.9. In addition, previous research from [8~~55~~] reported similar observations in bio-oil pH from *Chlorella* s~~S~~*p* of 9.33 of and *Nannochoropsis* 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 were split into alkane compounds [50~~56~~]. [25~~57~~] 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

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Previous research from [5] explained why bio-oil pH is in the range of 8-9 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 thatThe flame power of bio oil for temperatures of 300-600 ºC is in the slow category (lights up for more than 6 seconds). This is due to the high content of phenol in the biooil. it is necessary to do further treatment to increase the flame power of the biooil . The flame of bio-oil in this research can be . The flame of bio-oil in this research can be . The flame of bio-oil in this research can be . The flame of bio-oil in this research can be . 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 that 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 [16], [28]. The color of bio-oil from [28] was dark-brown, while the bio-oil from [16] 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 size greatly was affected the pyrolysis yield. The higher the pyrolysis temperature, the higher the conversion, and the bigger the particle size, the lower the yield of bio-oil, water phase, and gas for all temperatures tested. However, this is inversely proportional to the solid product, namely, bio char, where the larger the particle size, the greater the yield of bio char. The highest yield of bio-oil products was at 500ºC with a particle size of 140 mesh of 22.92%. The optimal temperature for SPR pyrolysis is at 500ºC. Product conversion is around 60-85%. The bio-oil product has properties such as a density of around 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.

ACKNOWLEDGMENTS

The author would like to thank the research funding assistance through the Research Fund Scheme Internally through the Research and Community Service

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Institute, Universitas Ahmad Dahlan Yogyakarta.

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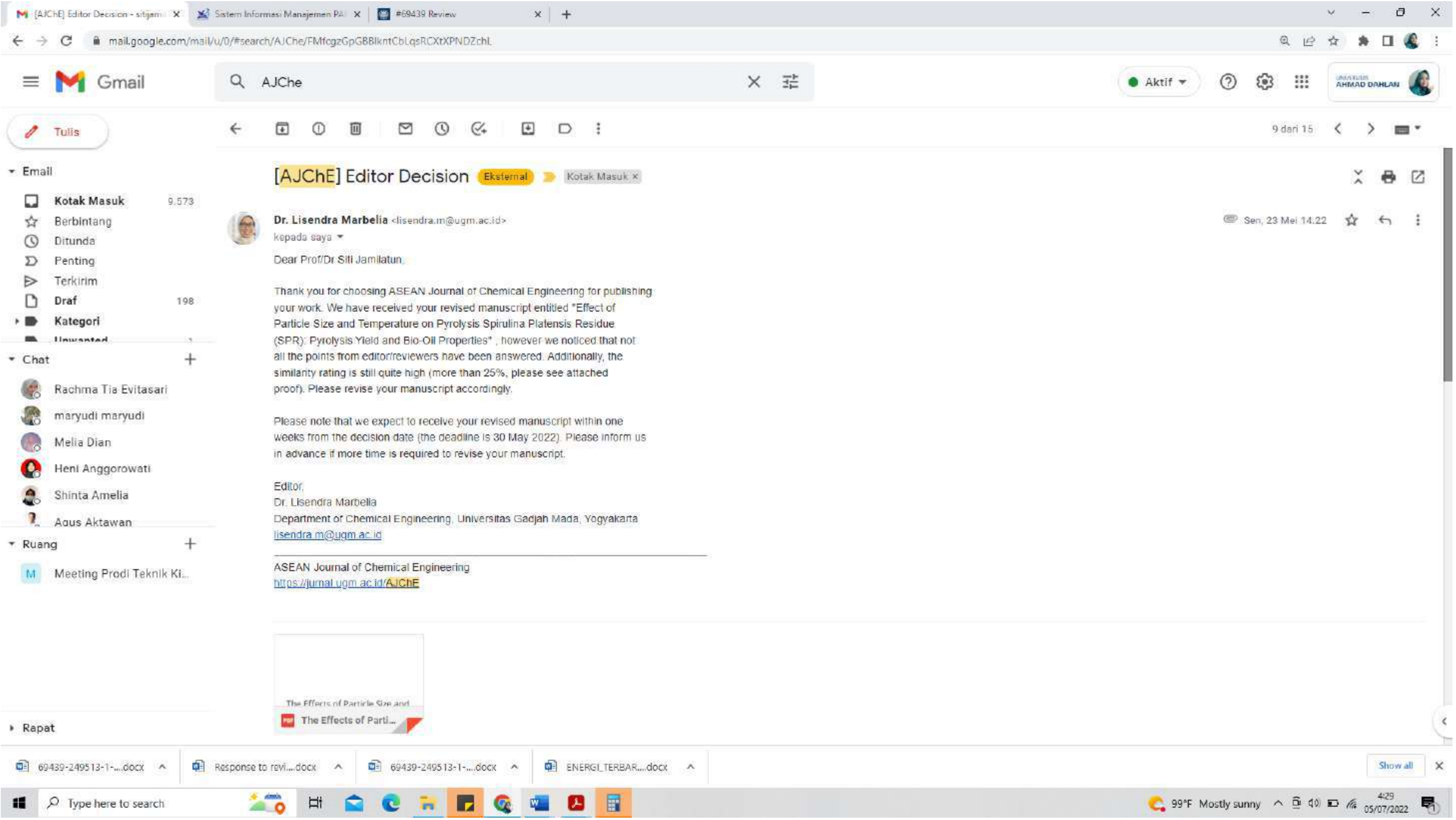

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The Effects of Parti...

The Effects of Particle Size and Temperature on Pyrolysis Spirulina Platensis Residue (SPR): Pyrolysis Yield and Bio-Oil Properties

by Ajche Ajche

AJChE 20XX, Vol. XX, No. X, XX – XX

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

Microalgae is the third generation of biomass as renewable energy, a future energy source for making bio-oil. This study aims to examine the biomass from microalgae *Spirulina platensis* residue (SPR) using the pyrolysis process, to investigate the effect of particle size and temperature on the pyrolysis process, to determine the bio-oil properties, including density, pH, color, flame power, and conversion. A fixed bed reactor was installed with several configurations: outer diameter of 44 mm, inner diameter of 40 mm, and reactor height of 600 mm. The temperature processes are installed from 300-600°C combined with a 14-16 °C/minute heating rate. Fifty grams of SPR with the particle size variation (80 and 140 mesh) are delivered to the reactor. From the experiment results, the particle size and temperature process influenced by bio-oil yield, water phase, gas yield, bio char 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, bio char, gas, water phase, pyrolysis, and *Spirulina platensis* residue.

INTRODUCTION

Microalgae is the third generation of biomass as renewable energy, a future energy source [20], [49]. In addition, microalgae can overgrow in wastewater with a high CO_2 level [2], dairy effluent [15] to reduce the cost of production, piggery wastewater [2], and can overgrow in open ponds [30]. Microalgae cultivation can reduce the greenhouse effect by using photobioreactors [17], [35]. In a photobioreactor, photosynthesis of microalgae takes place. Where photosynthesis is the process of converting

CO_2 in the air into O_2 with the help of light. The criterion by which the success rate of the process becomes is the amount of O_2 obtained. The greater the amount of O_2 produced, the more CO_2 is converted, and this indicates that the photosynthesis process in the photobioreactor is effective in reducing the concentration of CO_2 in the air to prevent global warming [17] [41]. There are many advantages of microalgae as follows: high lipid content [12], carbon hydrate [36], protein, and other chemicals [32], [53]. According to [11], the development of microalgae has many advantages over other biomass energy sources, such as a high rate of biomass production, not competing with food, and not requiring large areas of land for its growth. Microalgae contain lipids and fatty acids, which can be the essential ingredients of biofuels [26], [57]. Lipids produced from microalgae have C16 and C18, which can be esterified to produce high-quality biodiesel [7], [48]. The oil content in microalgae can exceed 80% by weight of dry biomass [34], [35].

As a renewable energy source, microalgae can be converted into three types: bio-oil, bio char, and non-condensable gas by using the pyrolysis process [5], [19]. The pyrolysis method is a process of thermal decomposition of organic matter at ambient temperature. There are high calorific values from microalgae such as *Chlorella vulgaris* residue (24.57−35.10 MJ/kg), *Spirulina platensis* residue (20.46-33.62 MJ/kg), *Chlorella sorokiniana* CY1 residue (20.24 MJ/kg), and *Nannochloropsis oceanica* residue (32.33−39 MJ/kg) [52]. As a comparison, these calorific values are higher than bio-oil produced from lignocellulose such as coconut shell (21.28 MJ/kg) and sugarcane bagasse [29]. Each pyrolysis product has its benefits if further processed; for example, bio-oil and gas can be used as marine material. The water phase is used as an additive for food preservation and contains components as a supplement. In contrast, bio char can be used as an adsorbent in food, sewage treatment, chemistry, etc.

The Making of bio-oil from microalgae by extraction produces solid residues that can be reused as a source of raw materials to produce biofuels [39]. This solid residue can be called *Spirulina platensis* residue (SPR), which contains high carbohydrates and protein. Using solid residues of microalgae of types with low lipid content, such as Spirulina sp. (4-9% lipids) as a raw material for pyrolysis is very beneficial because it can optimize the yield of bio-oil products by almost 40% [51]. The drawback of bio-oil from microalgae is the oxygenated and nitrogenated content is still high, which causes instability in its use.

Recently, many researchers have been conducted in the field of microalgae to convert a valuable product. [22] reported similar observations in their experiment from *Chlorella vulgaris* microalgae that containing: 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, heating rate, residence time, size and shape, catalyst, etc [10], [13], [18], [58]. The temperature factor is very crucial and essential because it can affect the composition of each product. Based on [40], the operating temperature increases beyond the optimum temperature, secondary cracks will occur, which causes a decrease in bio-oil production and an increase in gas production.

This research aimed to investigate particle size and temperature process's effect on slow pyrolysis of microalgae *Spirulina platensis* with range heating rate used is 14-16 °C/menit.. The factors that affect the pyrolysis process of *Spirulina platensis* are reaction time, material size, pyrolysis temperature, heating rate, material content. [46]. The particle size of 80 mesh and 140 mesh was applied in the system, while the temperature was performed at 300, 400, 500, 550 and 600°C. The reactor was installed in a fixed-bed reactor with an outer diameter of 44 mm, an inner diameter of 40 mm, and a reactor height of 600 mm.

RESEARCH METHODOLOGY

Materials

This section summarizes the primary raw material of this research was obtained from solid residues of *Spirulina platensis* and extracted with methanol (CH_3OH). Based on a previous study [44], 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%, carbon of 41.36%. The hydrogen of 6.60%, the nitrogen of 7.17%, and oxygen of 35.33%. HHV of SPR is 18.21 MJ/kg. SPR as raw material in this research was used 50 grams with the difference of size includes 140 mesh 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

Spirulina platensis residue (SPR) was obtained from the Chemical Engineering UGM Eco Mini Plant laboratory. SPR is mashed by pounding or using a blender. Then, sieving the SPR using a sieve with a size of 80 and 140 mesh. 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 [4], [45]. 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 with an inner diameter of 40 mm, an outer diameter of 44 mm, and 600 mm. The temperature control was needed to know the relationship between temperature and time and the relationship between time and heating rate. The heater and thermocouple were used as the temperature control in this research.

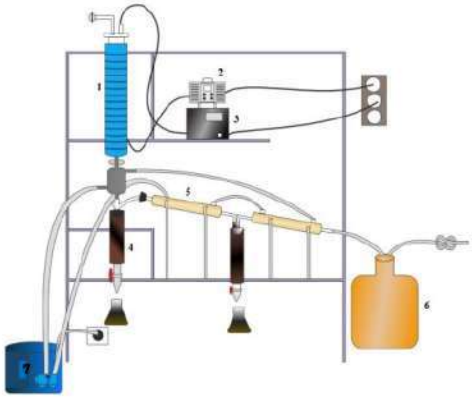

Fig. 1: Pyrolysis equipment

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 size 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:

$$Y_L = \frac{W_L}{W_M} x\, 100\ \% \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

$$Y_B = \frac{W_B}{W_M} x\, 100\ \% \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

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

$$Y_C = \frac{W_C}{W_M} x\, 100\ \% \quad \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

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

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

$$X = \frac{(W_B + W_{WP} + W_G)}{W_M} x100\ \% \quad \ldots\ldots (7)$$

In this case, Y_L, Y_B, Y_{WP}, Y_C, and Y_G are the yields of the liquid, bio-oil, water phase, bio char, and gas, respectively; W_M, W_L, W_B, W_{WP} and W_C are the weight of the SPR sample, the importance of the liquid product, bio-oil, water phase, and char, respectively, whereas X is conversion.

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, weigh the bottle that is filled with distilled water. Finally, calculate the density with the following formula:

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

b. Bio-oil color

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

c. pH

Measurement of 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

The effect of temperature and particle size on bio-oil yield

A visual representation of this result can be seen in Figure 2. This research is carried out using a temperature process from 300-600°C and particle-sized of 80 mesh and 140 mesh. The results of each experiment showed that the yield of bio-oil at a particle size 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 yield of bio-oil at 140 mesh and at processing temperatures: 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 particles size SPR as a raw material in this research.

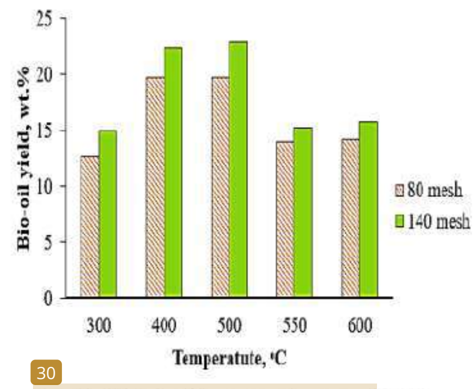

Fig. 2: Effect of temperature and SPR particle size on bio-oil yield

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 takes place in both particle sizes 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 [6], [31], [38]. 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. Following the experiment from [47], [54], the pyrolysis process increased with the increase of the thermal cracking reaction, which this condition made the increase in the gas and liquid production. The decomposition of biomass leads to the formation of phenol and CH_4 [42]. 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. [45].

The influence of particle size on bio-oil yield is identified as the larger the particle size, the lower the bio-oil yields. This result is relevant with previous research from [21], who also reported the effect of particle size 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 of the particle size in the particles. At a certain time, the core temperature is lower than the surface of the biomass particles, which can cause an increase in bio char, while the production of gas and bio-oil decreases. Small particles have sufficient surface area to interact with pyrolysis media to form volatile products such us gas, thus leaving the biomass matrix without experiencing secondary reactions [1]. Furthermore, it can be said that the particle size 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 size, 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 larger 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 sizes.

The effect of temperature and particles sized on the water phase.

The effect of temperature and SPR particle size on the yield water phase can be seen in Figure 3. respectively. The water phase with a particle size of 80 mesh was obtained 21.08, 22.40, 23.10, 25.10, and 26.06% with the temperature of 300, 400, 500, 550, and 600°C, respectively. At the same time, the water phase with a particle size 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 greater the temperature, the water phase yield value increased; this is because the high temperature can convert the water phase that is in the SPR optimally [46]. Next, also secondary decomposition reactions occur when the pyrolysis temperatures are very high [13]. This result was relevant with [9], 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 [43], the larger the SPR particle size used, the lower the yield water phase is obtained. Because the particle size 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.

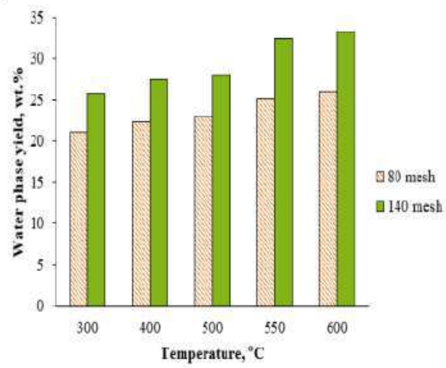

Fig. 3: Effect of temperature and SPR particle size on water phase yield

The effect of temperature and particles sized on bio char yield.

Figure 4 shows the effect of temperature and SPR particle size on bio char yield. The experiment result indicated that the yield of bio char with a particle size of 80 mesh was 39.14, 30.12, 27.90, 27.60, and 24.1%, respectively. In comparison, the bio char yield with a particle size 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 600ºC. In Figure 4, it can be seen that the bio char yield was gradually decreased when the temperature was too high and the particle of 80 mesh and 140 mesh. These results are relevant with [56]. In general, moisture and hydration water loss takes place at a temperature under 250ºC. In contrast, the temperature above 150ºC decompose and transform into a vapor containing organic compounds and gases [23], [24]. In addition, the decreasing biochar yield from low to high temperature was affected by organic matter decomposed when the pyrolysis was at high temperature [14].

The effect of particle size on char yield indicated that the char yield depended on particle size. The effect of particle size on the yield of bio char shows that the yield of charcoal depends on the particle size. 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 support for the secondary charcoal [27]. This study is by [44] the more significant the SPR particle size used, the greater the biochar yield obtained. An increase in particle size was affected by the larger 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 [37], [46].

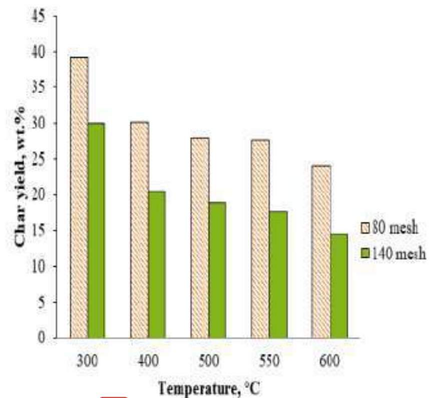

Fig. 4: Effect of temperature and SPR particle size on char yield

The effect of temperature and particles sized on gas yield

Figure 5 shows the effect of temperature and biomass particles on gas yield. The experiment indicated that the gas yield with a particle size of 80 mesh was 27.24, 27.86, 29.34, 33.26%, respectively. At the same time, the gas yield with a particle size 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 600ºC. In Figure 6, it can be seen that the higher the temperature, the greater the yield of gas. According to [37], 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 with [9], who reported the effect of biomass particles size from Chinese tallow trees. They were investigated that the small particle takes place reasonable heat transfer rate causing higher overall temperature. According to [55], the larger the SPR particle size used, the smaller the gas yield obtained. This phenomenon can occur because, at large particle sizes, the decomposition process is less than optimal due to the small contact area. It can affect the low speed of heat and mass transfer to convert SPR into gas.

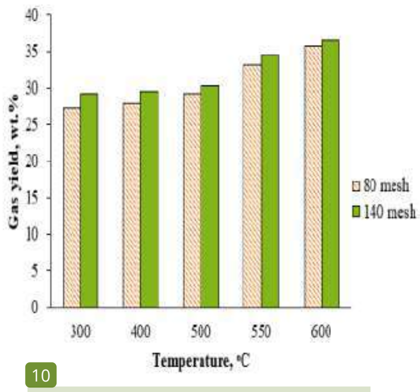

Fig. 5: Effect of temperature and SPR particle size on gas yield

Bio-oil conversion

Fig 6 shows the conversion of pyrolysis product with a particle size of 80 mesh and 140 mesh under temperature process at 300, 400, 500, 550, and 600°C. The experiment results from the biomass particle size 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 size of 140 mesh with the same temperature set was 70.02, 79.42, 81.16, 82.28, and 85.44%, respectively. In Figure 7, it can be seen that the higher the temperature, the pyrolysis product conversion increased. This result was relevant with [33]. 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 size used, the smaller the conversion of pyrolysis products obtained. From Figure 7, it can be concluded that the relationship between temperature, particle size, and conversion of pyrolysis products, namely an increase in pyrolysis temperature. A smaller SPR particle size causes thermal decomposition to work more effectively, decreasing the SPR weight.

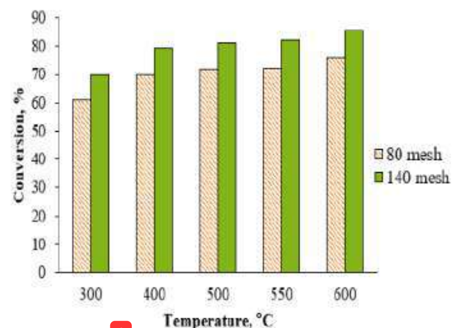

FIG. 6: Effect of temperature on various amounts of particle size on conversion

Bio-Oil Spirulina platensis residue (SPR) properties: Density of Bio-oil

There are given in Table 1. the bio-oil density with particle size (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 which have a low percentage of heavy molecular substances, their density tends to be lighter. The chemical compound in the fuel with high molecular weight will complicate the evaporation process in the engine combustion chamber and tend not to burn completely. Previous research from [53] explained why bio-oil density is in the range of 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 fuel, namely the lower density, increased the ignition of the fuel. Hence the fuel easily burned because the calorific value was high [16].

Table 1. Density of bio-oil

Density of Bio-oil (gram/ml)		
Temperature (°C)	Particle size (mesh)	
	80	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 the different particles of 80 mesh and 140 mesh. 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 size of 80 mesh at a temperature of 550°C with a pH level of 8.9. In comparison, the particle size of 140 at a temperature of 550°C has obtained a pH level of 9.0. Relevant works by these researchers [5] are the leading methods on the pH of microalgae bio-oil at pH of 8-9.9. In addition, previous research from [8] reported similar observations in bio-oil pH from *Chlorella sp* of 9.33 of and *Nannochoropsis* 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 were split into alkane compounds [50]. [25] 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 thatThe flame power of bio oil for temperatures of 300-600 °C is in the slow category (lights up for more than 6 seconds). This is due to the high content of phenol in the biooil. it is necessary to do further treatment to increase the flame power of the biooil. The flame of bio-oil in this research can be seen in Figure 7.

Figure 7: Flame power of bio-oil

Color of Bio-oil

Based on the experiment results, the color of the bio-oil was blackish-brown that 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 [16], [28]. The color of bio-oil from [28] was dark-brown, while the bio-oil from [16] 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 size greatly was affected the pyrolysis yield. The higher the pyrolysis temperature, the higher the conversion, and the bigger the particle size, the lower the yield of bio-oil, water phase, and gas for all temperatures tested. However, this is inversely proportional to the solid product, namely, bio char, where the larger the particle size, the greater the yield of bio char. The highest yield of bio-oil products was at 500°C with a particle size 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 around 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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The Effects of Particle Size and Temperature on Pyrolysis Spirulina Platensis Residue (SPR): Pyrolysis Yield and Bio-Oil Properties

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

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 [20], [49]. In addition, microalgae can overgrow in wastewater with a high CO_2 level [2], dairy effluent [15] To reduce the cost of production, piggery wastewater [2] and can overgrow in open ponds [30]. Microalgae cultivation can reduce the greenhouse effect by using photobioreactors [17], [35]. In photobioreactor, photosynthesis of microalgae takes place. Photosynthesis converts CO_2 in the air into O_2 with the help of light. The criterion by which the success rate of the process becomes is the amount of O_2 obtained. The greater the amount of O_2 produced, the more CO_2 is converted. This condition indicates that the photosynthesis process in the photobioreactor effectively reduces the concentration of CO_2 in the air and prevents global warming [17] [41]. There are many advantages of microalgae as follows: high lipid content [12], carbon hydrate [36], protein, and other chemicals [32], [53]. According to [11], 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 [26], [58]. Lipids produced from microalgae have C16 and C18, which can be esterified to produce high-quality biodiesel [7], [48]. Microalgae can have an oil concentration of up to 80% by mass of dry biomass [34], [35].

The pyrolysis process converts microalgae into three forms of sustainable energy: bio-oil, charcoal, and non-condensable gas. [5], [19]. The pyrolysis technique is a method for decomposing organic materials at room temperature using heat. Jamilatun (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 (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 [52]. 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 [29]. 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 [39]. *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% [51]. The drawback of bio-oil from microalgae is the oxygenated and nitrogenated content is still high, which causes instability in its use.

Recently, much research has been conducted on microalgae to convert a valuable products. [22] 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, [10], [13], [18], [59]. The temperature factor is crucial and essential because it can affect the composition of each product. Based on [40], 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. [46]. 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 the primary raw material of this research was obtained from solid residues of *Spirulina platensis* and extracted with methanol (CH_3OH). Based on a previous study [44], 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 of 6.60%, the nitrogen of 7.17%, and oxygen of 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 mesh 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 with a size of 80 and 140 mesh. 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 [4], [45]. 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.

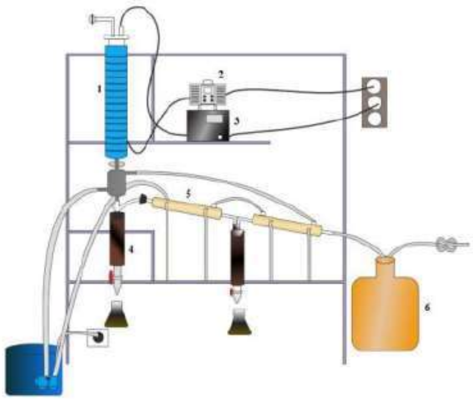

Fig. 1: Pyrolysis equipment

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:

$$Y_L = \frac{W_L}{W_M} x\, 100\ \% \quad \text{....................} \quad (1)$$

$$Y_B = \frac{W_B}{W_M} x\, 100\ \% \quad \text{....................} \quad (2)$$

$$Y_{WP} = \frac{W_{WP}}{W_M} x\, 100\ \% \quad \text{...............} \quad (3)$$

$$Y_C = \frac{W_C}{W_M} x\, 100\ \% \quad \text{.....................} \quad (4)$$

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

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

$$X = \frac{(W_B + W_{WP} + W_G)}{W_M} x 100\ \% \quad \text{......} \quad (7)$$

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:

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

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.

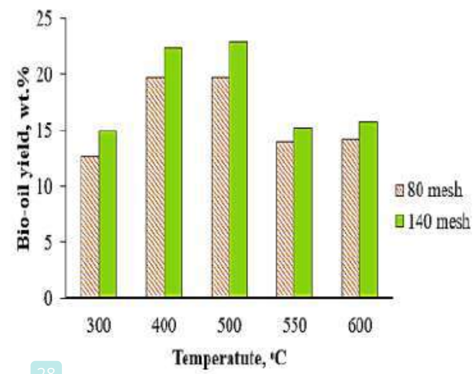

Fig. 2: Effect of temperature and SPR particle mesh on bio-oil yield

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 [6], [31], [38]. 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. Following the experiment from [47], [55], 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 CH_4 [42]. 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 [45].

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 [21], 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. Puny 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 [1].

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 600ºC, 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 [46]. Next, also secondary decomposition reactions occur when the pyrolysis temperatures are very high [13]. This result was relevant to [9], 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 500ºC. According to [43], 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.

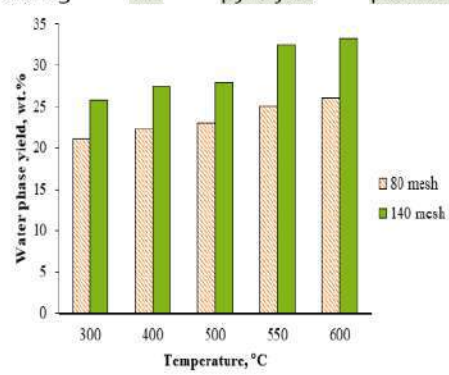

Fig. 3: Effect of temperature and SPR particle mesh on water phase yield

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 [57]. 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 [23], [24]. In addition, the decreasing biochar yield from low to high temperature was affected by organic matter decomposed when the pyrolysis was at high temperature [14].

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 [27]. This study is by [44] 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 [37], [46].

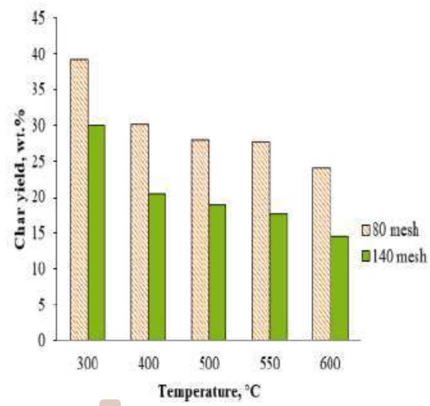

Fig. 4: Effect of temperature and SPR particle mesh on char yield

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 [37], 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 [9], 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 [56], 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.

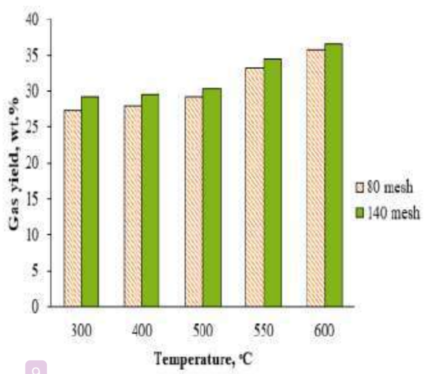

Fig. 5: Effect of temperature and SPR particle mesh on gas yield

Bio-oil conversion

Fig 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 [33]. 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.

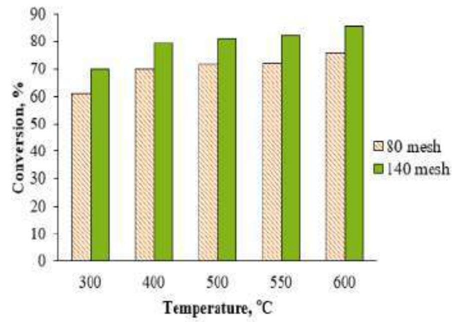

Fig. 6: Effect of temperature on various amounts of particle mesh on conversion

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 [53] 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 [16].

Table 1. Density of bio-oil

Density of Bio-oil (gram/ml)		
Temperature (°C)	Particle mesh (mesh)	
	80	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 [5] are the leading methods on the pH of microalgae bio-oil at a pH of 8-9.9. In addition, previous research from [8] reported similar observations in bio-oil pH from *Chlorellas* of 9.33 and *Nannochoropsis* 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 [50]. [25] 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) [54]. 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 [16], [28]. The color of the bio-oil from [28] was dark-brown, while the bio-oil from [16] 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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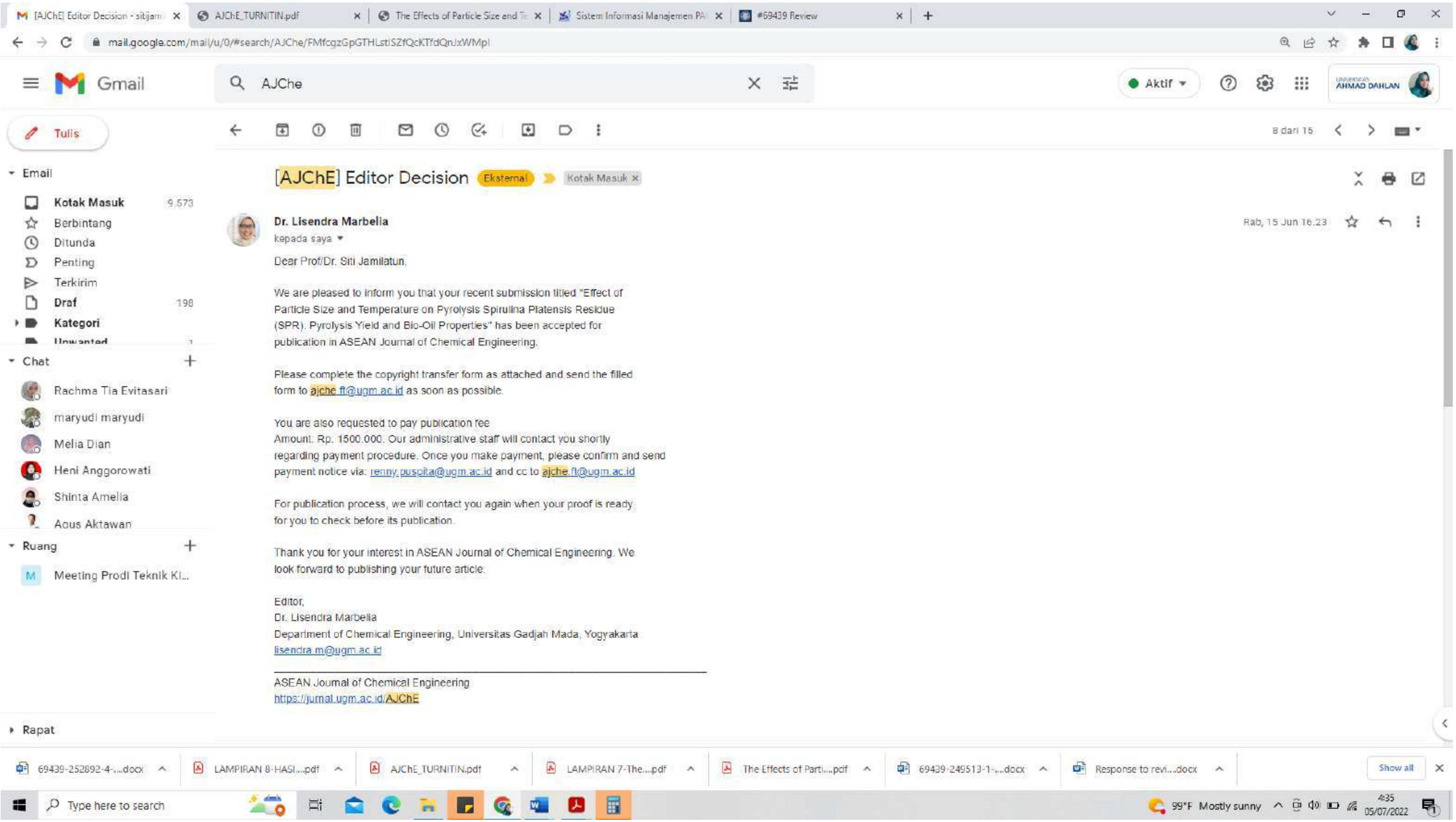

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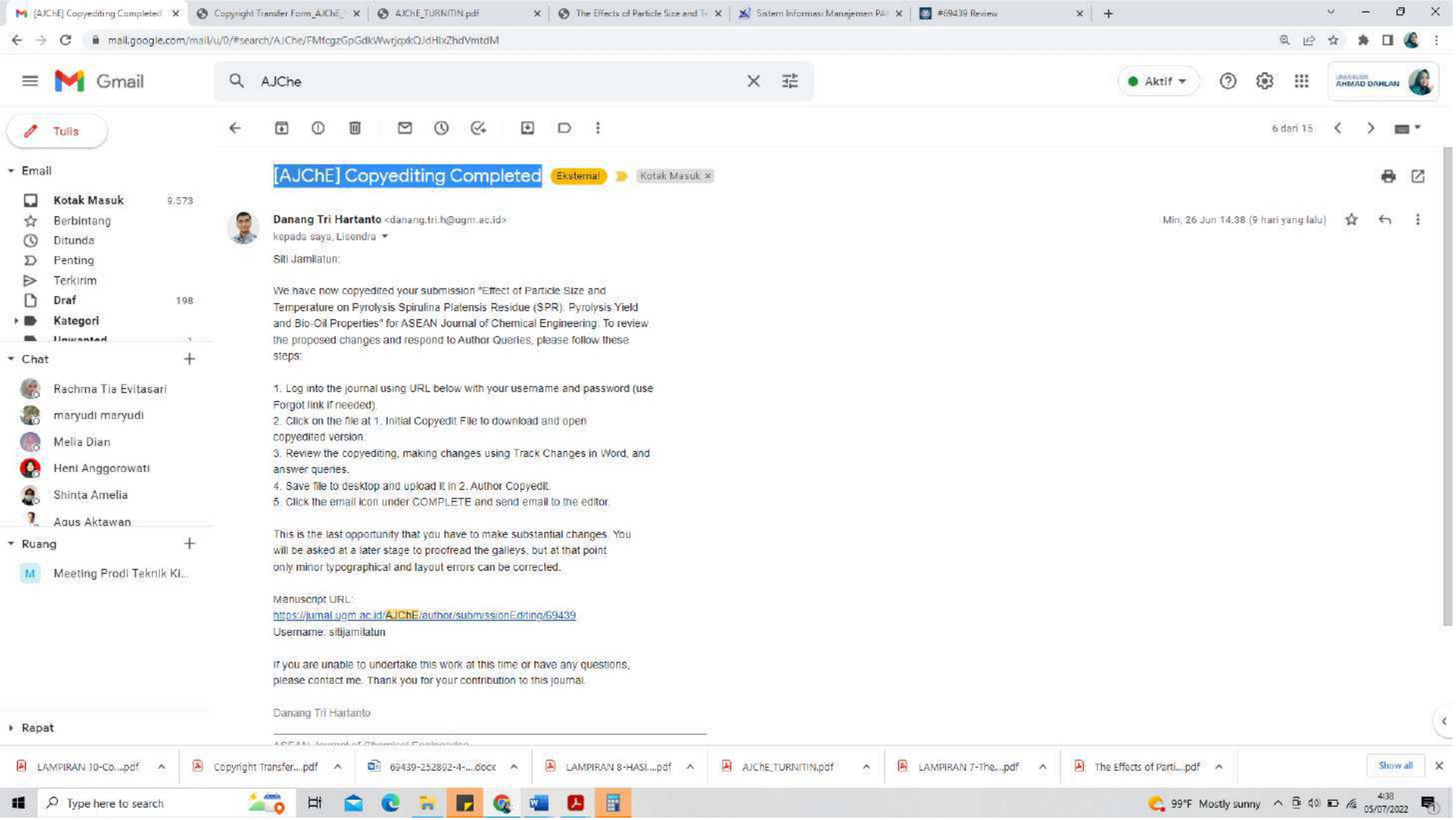

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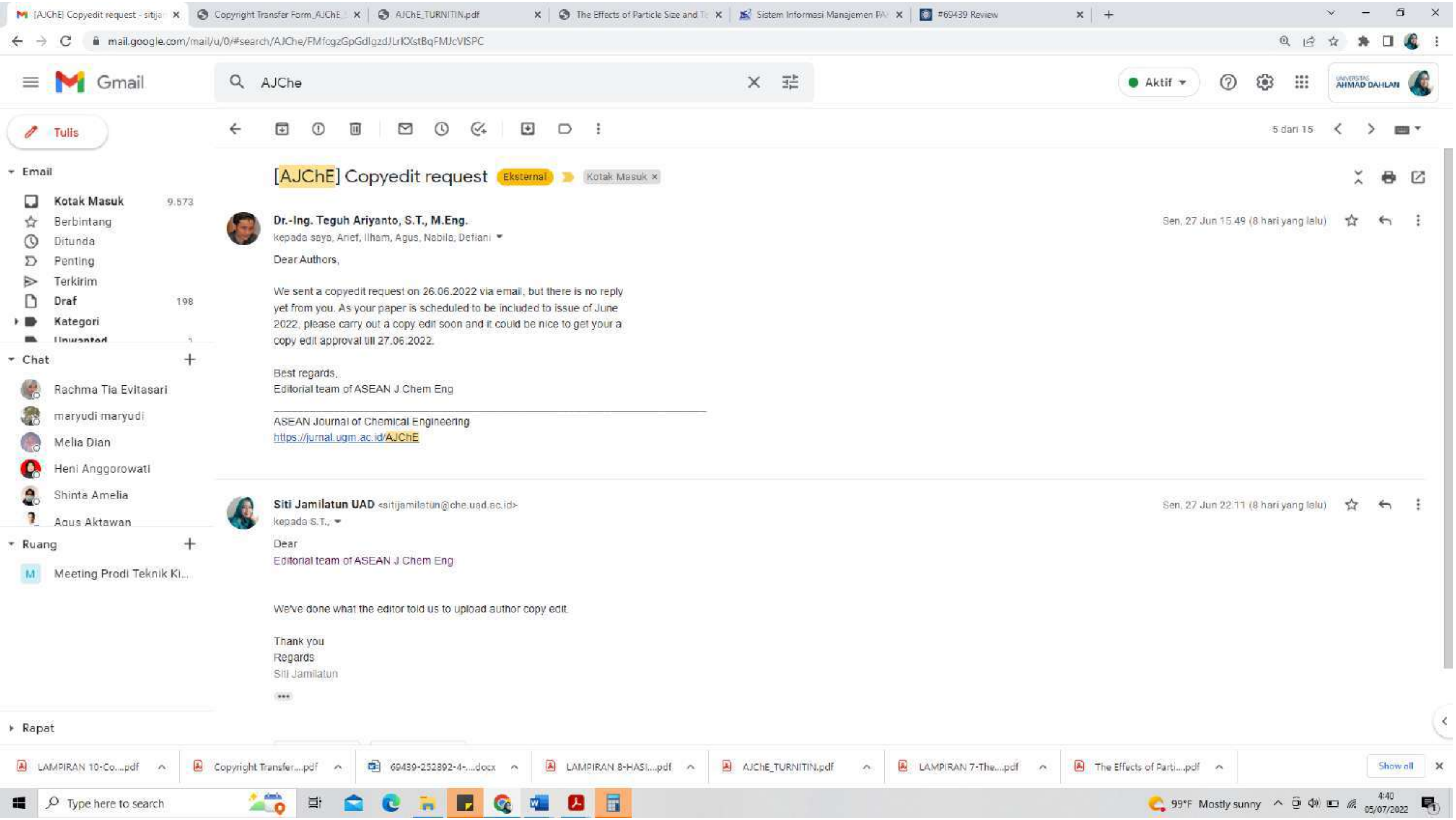

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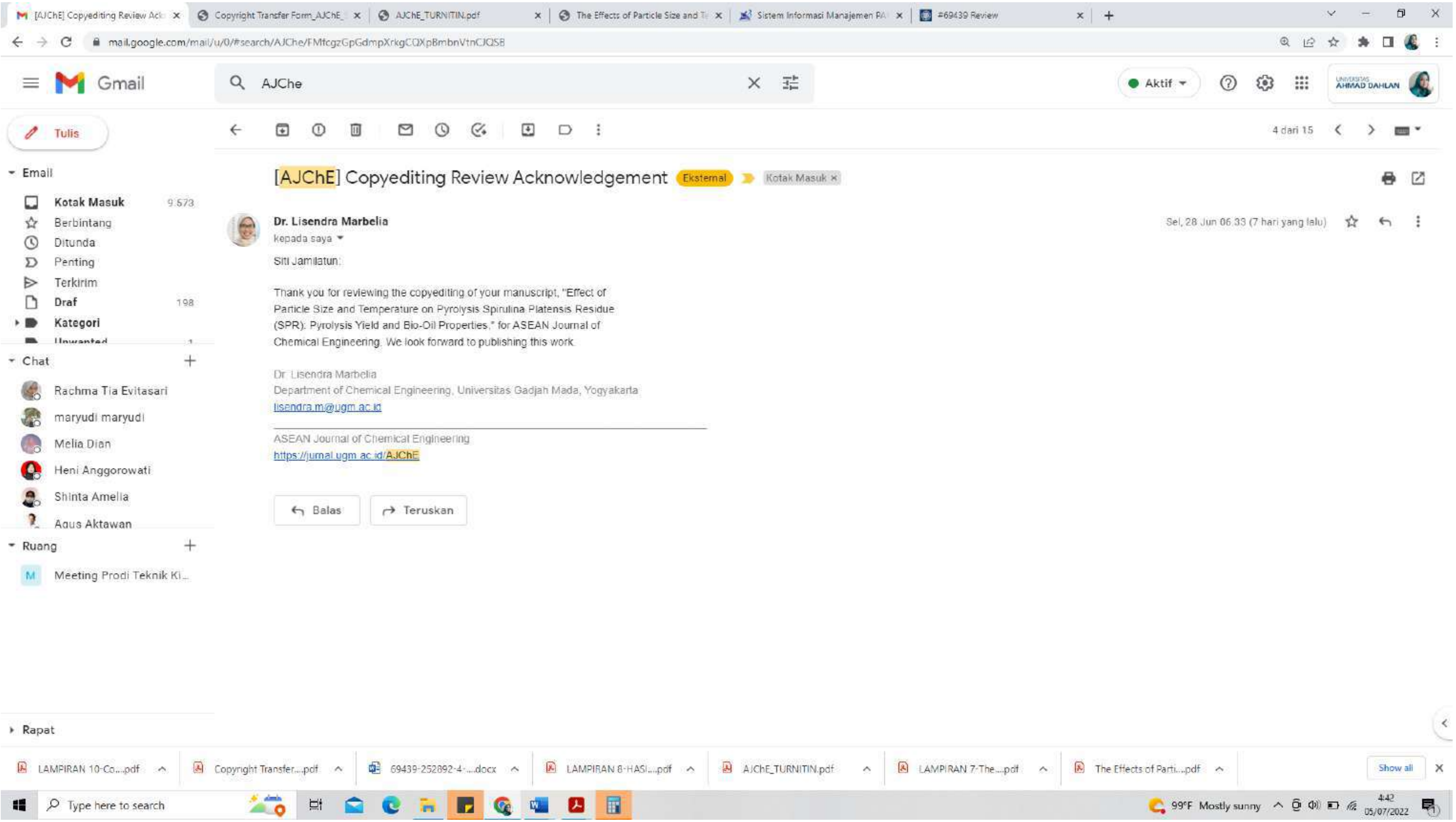

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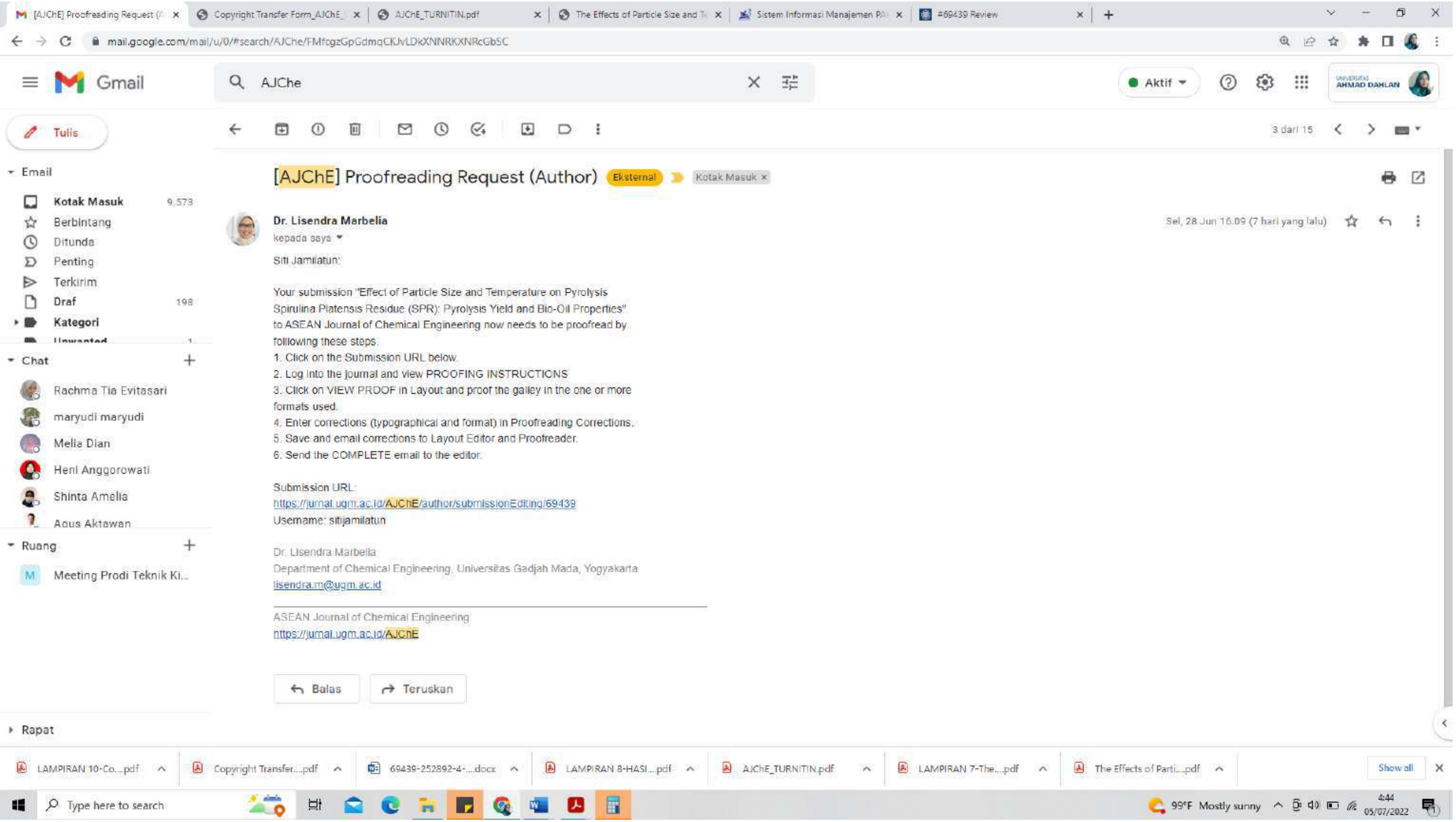

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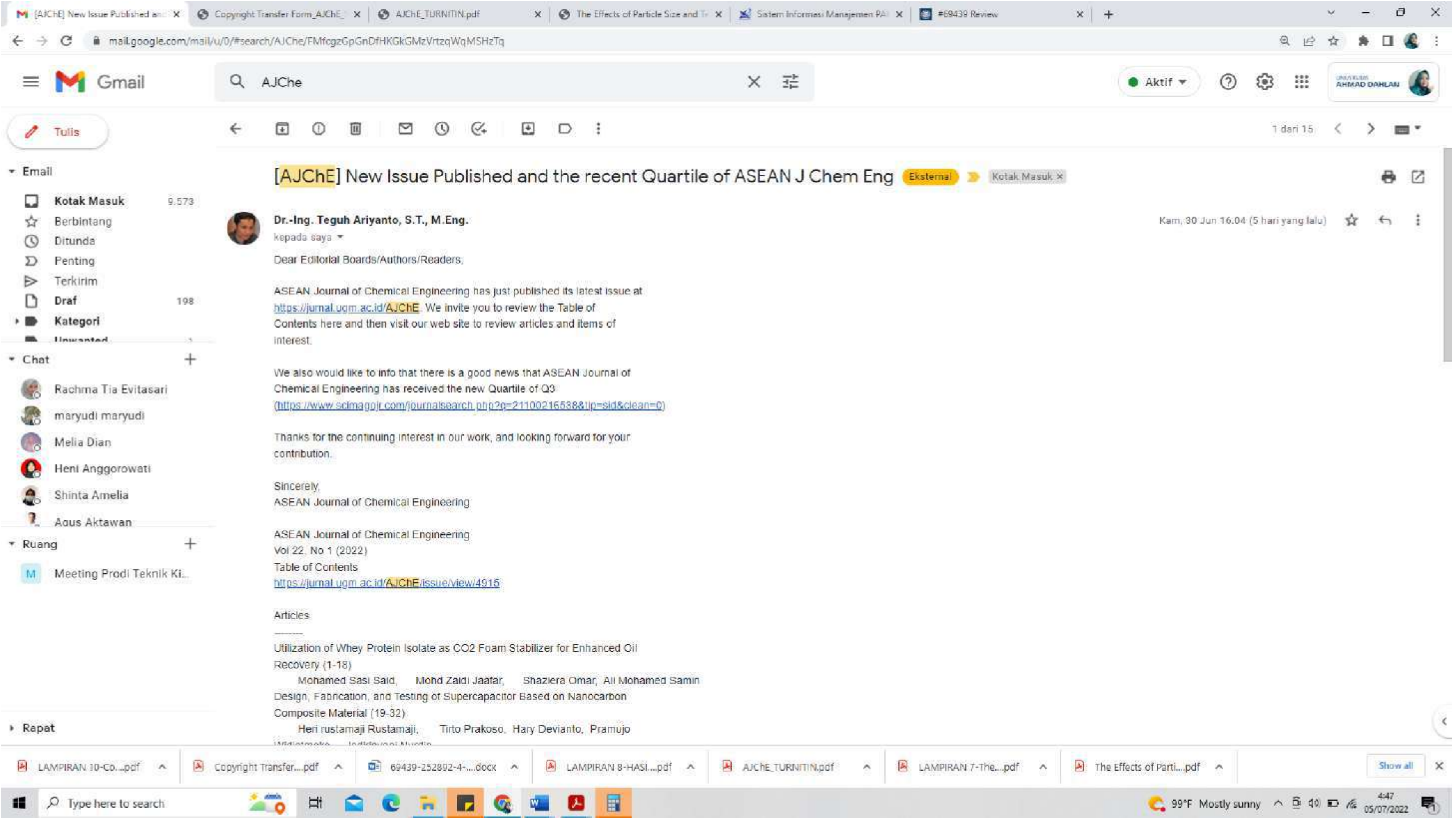

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ASEAN Journal of Chemical Engineering 2022, Vol. 22, No. 1, 141 – 155

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

Siti Jamilatun[1] *
Arief Budiman[2]
Ilham Mufandi[3]
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Submitted 28 September 2021 *Revised* 31 May 2022 *Accepted* 15 June 2022

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 CO_2 level (Ferreira et al. 2021), dairy effluent (Vieira Costa et al.

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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 CO_2 in the air into O_2 with the help of light. The criterion by which the success rate of the process becomes is the amount of O_2 obtained. The greater the amount of O_2 produced, the more CO_2 is converted. This condition indicates that the photosynthesis process in the photobioreactor effectively reduces the concentration of CO_2 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 ^oC/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 ^oC. 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 (CH_3OH). 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.

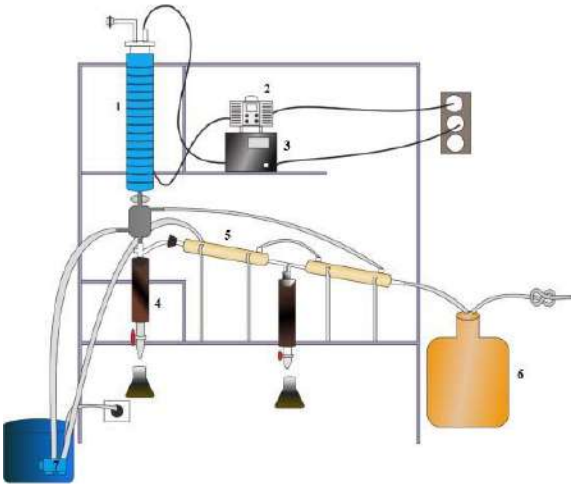

Fig. 1: Pyrolysis equipment

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 ºC. 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:

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

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

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

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

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

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

$$X = \frac{(W_B + W_{WP} + W_G)}{W_M} x100\ \% \quad (7)$$

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.

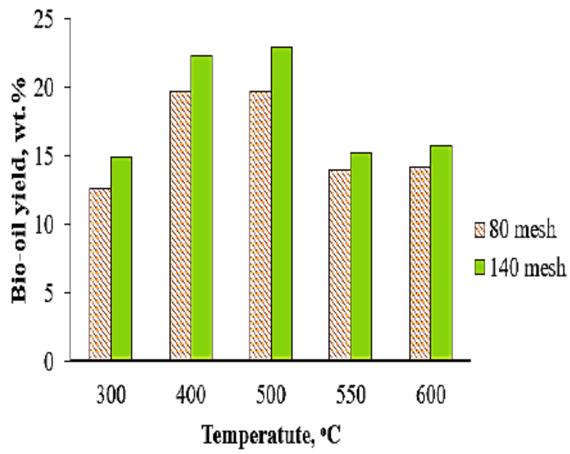

Fig. 2: Effect of temperature and SPR particle mesh on bio-oil yield

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 CH_4 (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. Puny 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 ºC, 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 ºC. 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.

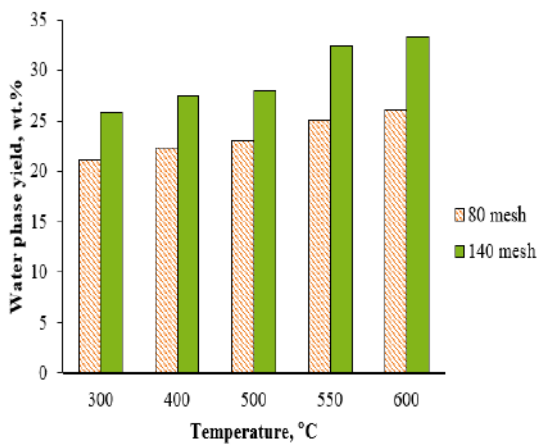

Fig. 3: Effect of temperature and SPR particle mesh on water phase yield

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 ºC. 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 ºC. In contrast, temperatures above 150 ºC 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).

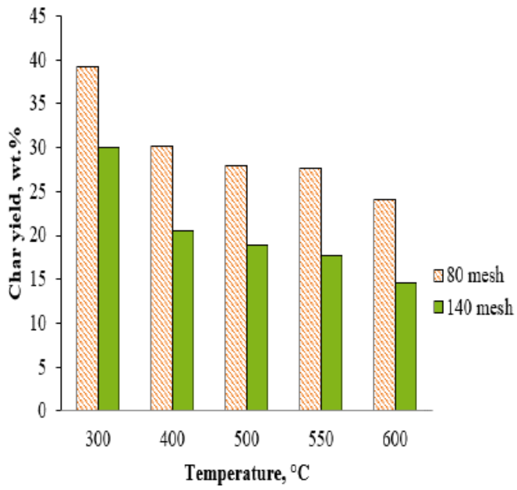

Fig. 4: Effect of temperature and SPR particle mesh on char yield

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 ºC. 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.

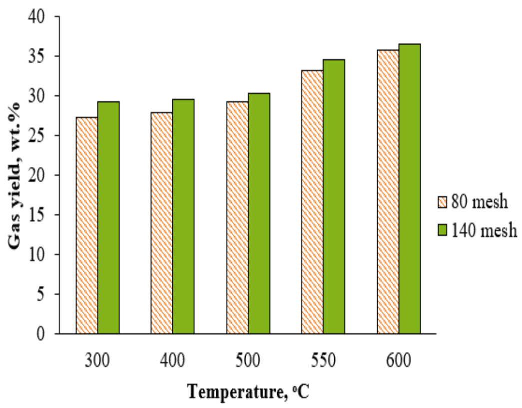

Fig. 5: Effect of temperature and SPR particle mesh on gas yield

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.

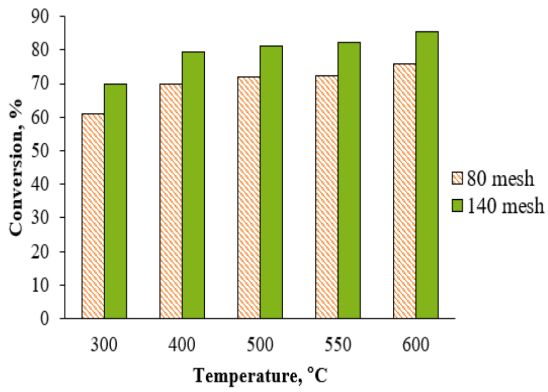

Fig. 6: Effect of temperature on various amounts of particle mesh on conversion

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 (Chukwuneke et al. 2019).

Table 1. Density of bio-oil

Density of Bio-oil (gram/ml)		
Temperature (ºC)	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 ºC. 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 ºC with a pH level of 8.9. In comparison, the particle mesh of 140 at a temperature of 550 ºC 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 *Nannochoropsis* 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 ºC 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 (Chukwuneke 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 (Chukwuneke 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 ºC with a particle mesh of 140 mesh of 22.92%. The optimal temperature for SPR pyrolysis is at 500 ºC. 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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