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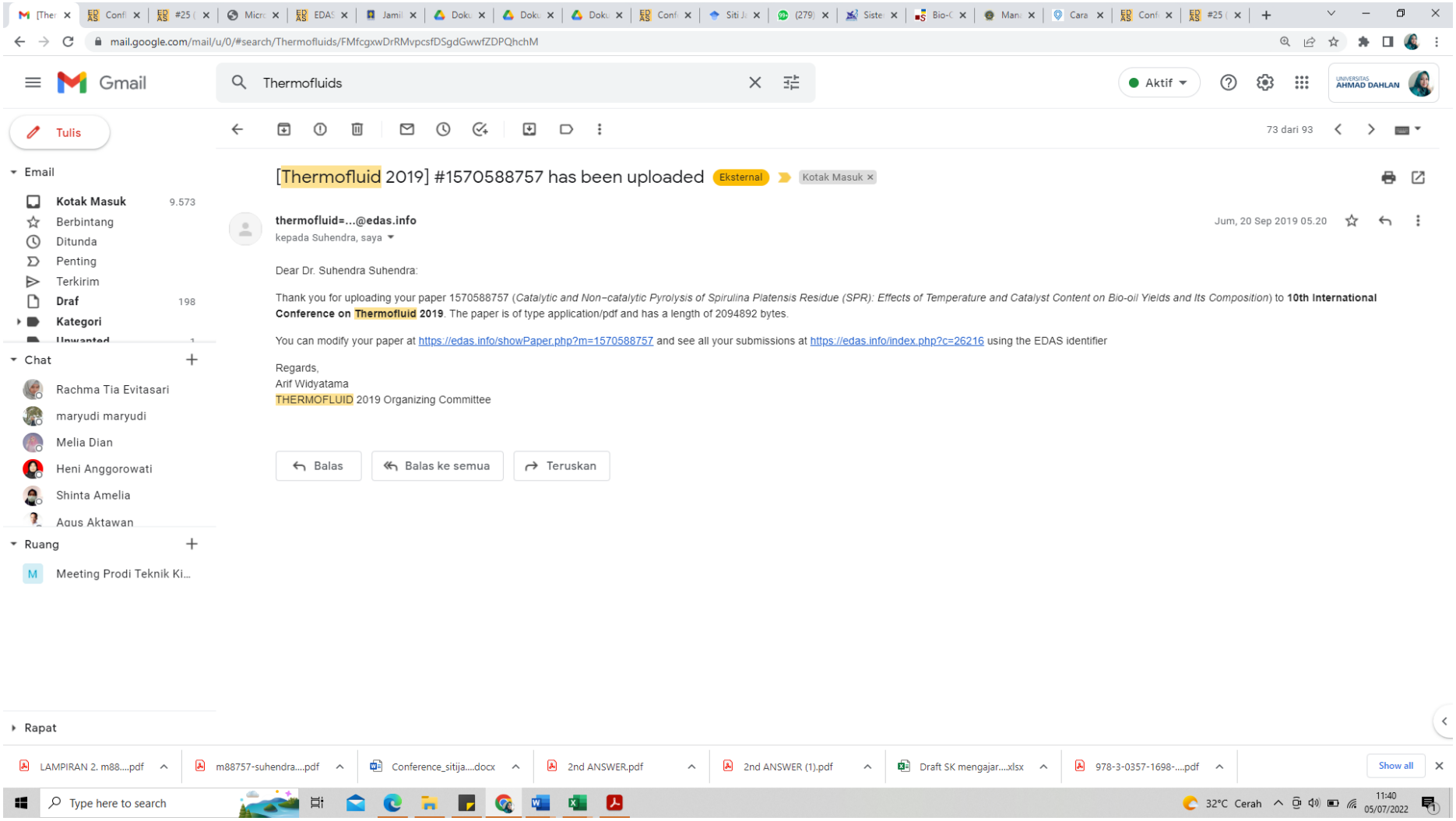

Catalytic and Non−catalytic Pyrolysis of Spirulina Platensis Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition

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Abstract. Problems associated with the development of the first and second generations of biofuel, especially regarding its raw materials and complex processes, has led to the evolution to the third generation one, a microalgae-based bio-oil through pyrolysis. This research explored the effect of temperature ranging between 300−700 °C and the presence of Silica−alumina (Si-Al) catalyst at the variation of 5–20 wt.% on the pyrolysis process of Spirulina Platensis microalgae solid residue, in search of optimum condition for collecting maximum bio-oil yield and its desired composition. The experiments without catalysts reached its optimum yield of 28 wt.% at 550 °C. Whilst the involvement of 5 wt.% of Si-Al catalyst in the reactor resulted in a higher yield than that of without catalyst beyond 550 °C. Furthermore, a high amount of catalyst content at 20 wt.% seems to improve bio-oil yield collection up to 34.10 wt.%. Interestingly, referring to its C atoms number, SPR-based bio-oil by catalytic pyrolysis tended to produce such considerable amount of gasoline than the one without catalyst.

Keywords: *Spirulina platensis* residue (SPR), pyrolysis, thermal decomposition, bio-oil.

1. BACKGROUND

Despite being widely tested, first and second generations biofuel leave some issues regarding their availability and competition against primary use of their raw materials. First generation biofuels (i.e. biodiesel, corn ethanol, sugar alcohol) from vegetable oils and corn sugar sources are constrained by the limited feedstocks and competition with food products. In addition, the second generation biofuels (ie. hydrotreating oil, bio-oil, lignocellulose ethanol, butanol, mixed alcohols) raise problem due to its extensive agricultural land requirement, although they come from non-edible oil, non−food, cheap and abundant plant waste biomass sources. Such situations stimulate researcher for finding an alternative to overcome problems mentioned before. Development of third generation biofuels (biodiesel, bioethanol, biohydrogen, biomethane) based on microalgae biomass offer a simpler production process of pyrolysis and non-competing sources.

Moreover, microalgae may provides many advantages, such as: (i) environmentally friendly, (ii) not competing with food and agricultural land, (iii) very high oil products (iv) simple cellular structure, (v) lipid−rich (40−90% in percent dry weight), (vi) rapidly growing rate, (vii) able to grow in brine and poor conditions, (viii) evolved from CO2 gas produced by power plants, and (ix) containing no sulfur, non toxic and highly biodegradable. On these ground, microalgae seems very potential to be developed as a source of energy of the future.

Basically, microalgae-based biofuel can be produced via biochemistry conversion, thermochemical conversion, chemical reaction, and direct burning. Among them, thermochemical conversion through pyrolysis process seems quite promising due to its fairly simple process and without the need of additional chemicals. Process at an atmospheric pressure and a relatively low temperature between 350 and 600 °C can produce bio-oil, gas and char for later being used as fuel and adsorbent, respectively. Therefore, such research in the area of microalgae-based bio-oil through pyrolysis gain significant priority.

Pyrolisis itself can be performed with or without catalyst. Study on pyrolysis without catalyst using various raw materials (ie. Chlorella vulgaris, Spirulina plantesis residue and Saccharina japonica) reported optimum bio-oil yield ranging between 24.5 and 32.7 wt.% at temperature between 500 – 550 °C. The use of catalyst does not necessarily improve bio-oil yield uptake as demonstrated by Lim et al, presenting fairly similar yield on pyrolysis of Palm oil with Br2O3 catalyst. Neither changing the type of catalyst (ie. ZnO and Al2O3) would result in significant difference of bio-oil uptake from Eastern giant fennel (Ferula orientalis l.). Rather than that, the use of catalyst may be seen as composition changer to the extends of the way hydrocarbon is chemically modified. Silica-alumina (Si-Al), which widely available on the market as cheap commodity, may play such role.

This paper discusses the effect of temperature and catalyst (Si-Al) content on the pyrolysis of Spirulina platensis residue (SPR) on bio-oil yield and its composition. Product yields were observed referred to its mass-basis of each components. Whilst, gas chromatography/mass spectrometry (GC−MS) technique were employed for characterizing bio-oil compositions.

2. METHODS

2.1.Materials

2.1.1. Spirulina Platensis Residue (SPR)

SPR was obtained from solid residue extraction of Spirulina platensis with size of 80−140 mesh (0.177−0.105 mm). The proximate analysis was performed to characterize the moisture content, ash, volatile matter and fixed carbon. Whilst, C, H, O and N content were quantified by ultimate analysis. The calorific value, conducted at the Research and Development Center for Mineral and Coal Technology (TEKMIRA), Bandung, Indonesia, was done using a calorimeter bomb where the results are in Table 1.

TABLE 1. Main characteristic of SPR [14]

Components	SPR
Proximate analysis (wt.%)	
Moisture	9.99
Ash	8.93
Volatiles	68.31
Fixed carbon	12.77
Ultimate analysis (wt.%)	
Carbon	41.36
Hydrogen	6.60
Nitrogen	7.17
Oxygen	35.33
Sulfur	0.55
Lipid	0.09
Carbohydrate	25.59

2.1.2. Silica−alumina catalyst

Silica−alumina catalyst was kindly supplied by PT. Pertamina, Balongan, Indonesia. Silica-alumina properties, including the ratio, specific surface area, pore volume, pore diameter and SEM−EDX, were analyzed at LPPT−UGM. The results are shown in Table 2.

TABLE 2.Silica−alumina catalyst specifications

Si-Al specification	Value
SiO_2/Al_2O_3	1.67
Pore surface area	240.553 m^2/g surface area
Pore diameter	3.3 nm
Average pore volume	0.199 cm^3/g total pore volume
Compositions (wt.%)	
C	12.33
O	55.73
Al	15.42
Si	16.51

2.1.3. Experimental methods

SPR pyrolysis was performed in a 40 cm ID and 60 cm length of a fixed−bed reactor system, depicted in Figure 1.

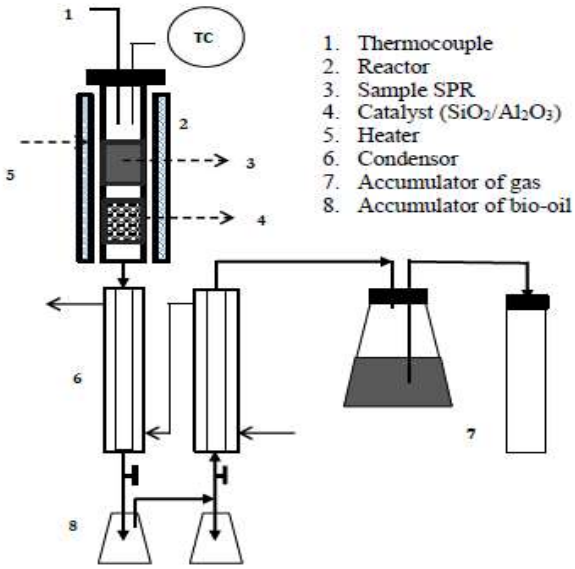


FIGURE 1.: The schematic diagram of the fixed bed tubular reactor

The reactor consists of upper and bottom part for placing SPR and Si-Al catalyst, respectively. A nickel wire heater, coiled outside the reactor tube, are provided for maintaining operating temperature between 300 and 700 °C. In addition, condenser was used for bio-oil and gas products for cooling. Fifty (50) g of SPR was inserted into reactor chamber. Heat was then introduced at a constant rate of 20°C/min from ambient to processing temperature. Then, pyrolisis process was always maintained for 1 hour. After the reaction finished, bio-oil and gas could be collected in their corresponding accumulator, following their path through condenser for temperature cooling. It is noted that bio-

oil would first require to be separated by decantation from its water in liquid phase. Lastly, the amount of solid (bio−char) left behind in reactor chamber was picked up and weighed.

3.1. Tests and Measurements

3.3.1. Weight percentage of yield procentage of yield ercentage of yield product analysis

Weight of liquid products (refer to bio−oil and water), gas and bio-char were calculated by the following equation:

$$Y_{bo} = (W_{bo}/W_M)x\ 100\ \% \quad \text{.....(1)}$$
$$Y_{wtr} = (W_{wtr}/W_M)\ x\ 100\ \% \quad \text{.....(2)}$$
$$Y_{Cha} = (W_{Cha}\ /W_M)\ x\ 100\ \% \quad \text{.....(3)}$$
$$Y_{Gas} = 100 - \left(Y_{liq} + Y_{Char}\right) \quad \text{.....(4)}$$

Y and *W* represent yield percentage and weight, respectively. Its corresponding subscripts, of M, bo, wtr, gas and char, are for initial SPR, bio-oil, water, gas and bio-char.

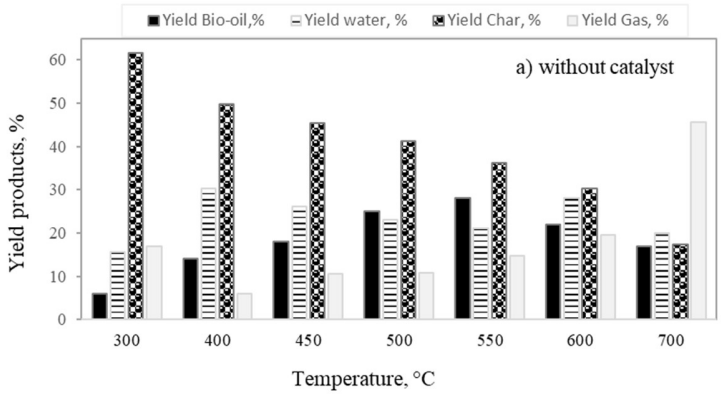
2.3.2. Characterization of bio-oil compositions

Bio-oil obtained from pyrolysis liquid product (light phase) was tested for its composition using Gas chromatography-mass spectrometry (GC-MS). Then, based on the number of C constituent atoms, bio-oil compositions were grouped into fractions of LPG (C4), gasoline (C5-C11), diesel (C12-C18) and heavy naphtha (C> 19).

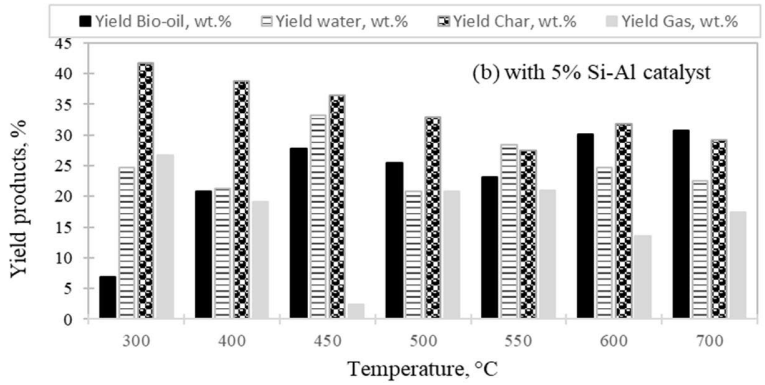
3. RESULTS

3.1. Effects of Temperature and Catalyst Content on Pyrolysis Yield Product

Figure 2a showed the yield products of bio−oil, gas, char and water phase, obtained from pyrolysis process without using catalyst for various temperature between 300 and 700 °C. It might be expected that the more heat introduced into system resulted in more hydrocarbon decomposition. Such phenomenon was perhaps seen by the reduction of solid product (and bio-char) yield from 61.55 to 17.3 wt.% with increasing temperature, prior to stabilize at 600 °C, and above. Along with that, solid texture analysis exhibiting gradual changes from soft to hard texture was reported. This would means decomposition initially occurred by releasing some hydrocarbon compounds, followed by gradual compositional changes and finally reached bio-char stable composition. Through another perspective, this would means that the remaining hydrocarbon were converted to gas and liquid products.



(a)



(b)

FIGURE 2. Effect of temperature on yield products, obtained from SPR pyrolysis process a) without and b) with the presence of 5% Si-Al catalyst

The liquid products consisted of water and bio-oil was initially collected at 300 °C. At this point, bio-oil presented the lowest yield of 6 wt.%. Trend of increasing bio-oil uptake was then observed, following their 50 °C incremental increase in processing temperature up to 550 °C. Interestingly, discontinuation on that trend, seen by sudden yield decrease, resulted to consider 550 °C as peak maximum temperature for SPR pyrolysis without catalyst. Such phenomenon was quite typical, as similarly reported elsewhere. Above 600 ºC, bio-oil yields tended to decrease due to the secondary cracking reaction that resulted in further decomposition into gas and char. Response to bio-oil secondary cracking might be confirmed by the inverted gas yield plot presenting a peak minimum at temperature between 300 and 400 °C, followed by sharp yield increase to 45.7 wt.% at 700 °C.

For a comparative analysis, the yield result of pyrolysis at various temperature in the presence of 5 % Si-Al catalyst is presented at Figure 2b. As observed, trend on bio-char and water yield remain fairly similar. However, no such peak maximum seen on bio-oil yield since it would rather shows an increase of bio-oil with increasing temperature. Conversely, gas yield fluctuated, followed by a tendency to decrease at the later stage around 550 °C and above, in the presence of catalyst.

Hence to further understand the influence of 5% Si-Al catalyst, each yield on Figure 2b were then compared against results shown on Figure 2a for their corresponding temperature. It was evident that catalyst was able to boost bio-oil uptake at temperature between 300 and 450 °C, considered to be primary phase decomposition. Moreover, the more significant bio-oil yield were perceived for pyrolysis process conducted at temperature higher than 550 °C, taking into account that the relative data on the curve “without catalyst” lean toward decreasing trend as oppose to increasing trend on the one with catalyst.

As the focus were paid on maximizing bio-oil uptake, there might be a possibility that the amount of bio−oil would further increase with temperature. For that reason, the SPR pyrolysis with Si-Al catalyst was further experimented to study the effect of the amount % catalyst between 5 and 20 % at the highest temperature previously tested of 700 °C, where the results are shown in Figure 3.

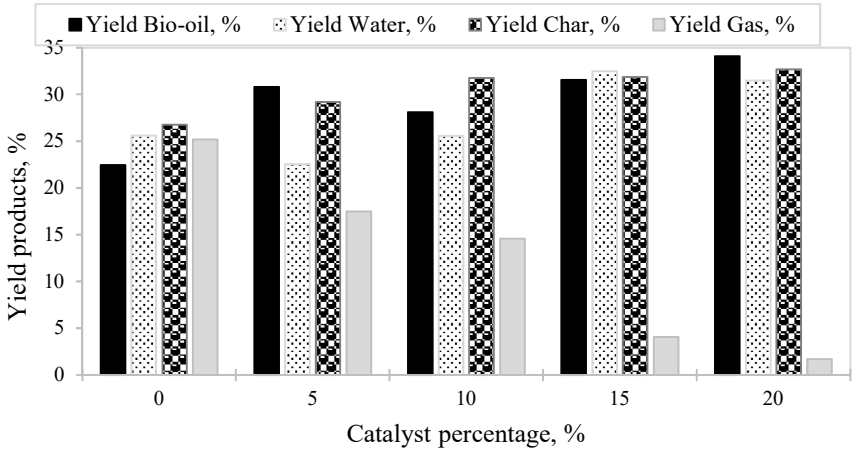


FIGURE 3. The evolution of yield product percentage as function of Si-Al catalyst percentage between 0 and 20% on SPR pyrolysis products at 700 °C

As seen, the result presented that yield gas decrease quite significantly from 17.47 to 1.7 wt.% with the increase of catalyst involved from 0 to 20%. Consequently, liquid product and bio-char seemed to increase. Liquid products, a total amount of Bio−oil and water, were increased by ±17% from 48.04 to 65.10 wt.% in which 12% increase were contributed from the raise of bio-oil uptake. In fact, the involvement of catalyst by only 5% has led to increase of about 8%. A higher Si-Al catalyst percentage tested between 5 and 20% did not cause significant rise despite its increasing trend. Regardless, its concurrent decrease in gas and bio-oil increase might suggest the occurrence of vapor condensation.

3.2. Fractional Composition of bio−oil products as a function of temperature and catalyst content

Table 3 presented the fractional composition, referring to their number of C atoms (LPG, gasoline, diesel and heavy naphta) of bio-oil obtained by pyrolysis without catalyst at temperature of 300, 400, 500 and 600 °C.

TABLE 3.Bio-oil composition referring to its C-atom category, obtained at various pyrolysis temperature between 300 and 600 °C, without catalyst

Pyrolysis temperature, °C	Amount of C			
	C<4 (LPG)	C5−C11 (Gasoline)	C12−C18 (Diesel)	C>19 (*Heavy nafta*)
300	16.60	64.50	9.73	0.09
400	0.66	52.14	25.97	21.26
500	−	15.32	49.53	35.12
600	−	15.66	49.92	36.94

At 300 °C, bio−oil consisted of LPG (C<4), gasoline (C5−C11), diesel (C12−C18) and heavy naphta (C>19) in which their corresponding content were 16.60, 64.50, 9.73, and 0.03 wt.%, respectivelly. At 400 °C, LPG fraction started to disappear showing only 0.66 wt.% collected. As expected, this fraction was not found at temperature 500−600°C. In addition, gasoline fraction also exhibited sharp decrease in weight percentage from 64.50 to 15.66 with the raise of tested temperature. Consequently, other fractions would rise, given the fact that diesel increased from 9.73 at 300 °C to 49.92 wt.% at 600 °C, besides the presence of 36.94% of heavy naphta at 600 °C from almost none at 300 °C. By combining information summarized at Figure 2a and Table 3, bio-oil uptake could be obtained maximum at 550 °C where its composition likely consisted of about 15, 50 and 35, respectively for LPG, gasoline and diesel. Interestingly, such composition seemed to present its potential for being used as future fuel due to its similarity on hydrocarbon content as compared to fuel derived from crude oil.

Table 4 presented the result of LPG, Diesel, and Heavy naphta fraction on bio-oil yield, obtained from pyrolysis process with Si-Al catalyst having content from 5 to 20 wt.%. In the presence of catalyst, none of heavy naphta was

produced regardless the catalyst content to suggest the suppression of longer chain hydrocarbon production. In addition, diesel, having chain number between 12 and 18, exhibited a decrease as well. Thereof, gasoline were the major fraction produced, around 80 wt.%.

Table 4: Component of bio-oil SPR based on the amount of C on various % of the amount of Silica−alumina catalyst

Catalyst (%)	Amount of C		
	C<4 (LPG)	C5−C11 (Gasoline)	C12−C18 (Diesel)
5	-	80.06	19.34
10	-	41.48	57.06
15	-	86.56	13.13
20	-	81.89	17.73

The influence of catalyst content on bio-oil fraction was rather inconclusive at this point. Statistically, gasoline and diesel fraction composition were around 80 and 20 % regardless the catalyst content, except for the result when using 10% of Si-Al catalyst. This great amount of gasoline fraction agreed with study reported by Sunarno 2017, that the rise in pyrolysis temperature and the use of silica-alumina catalysts by impregnation of Ni metal will increase the gasoline, kerosene and phenol fractions, whilst, the oxygenate compound will decrease. In any case, the introduction of only 5% Si-Al catalyst was able to drive the pyrolysis process to lean toward the production of gasoline, which would be prospective to replace crude oil-based gasoline fuel.

3.3 Acknowledgments and Legal Responsibility

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3.4. CONCLUSIONS

This research aimed to study the influence temperature and catalyst content on the pyrolysis process of Spirulina Plantesis Residue (SPR) with and without Silica-Alumina (Si-Al) catalyst. Pyrolysis process of SPR, for both without and with the catalyst, produced liquid product (bio−oil and water phase), gas and char. On the basis of weight percentage, the optimum bio-oil uptake of 28 % were obtained from pyrolysis at temperature 550 °C, considered as a peak maximum within the range of tested temperature (300-700 °C). At this temperature, the resulting bio−oil compositions was likely to be consisted of gasoline (C5−C11), diesel (C12−C18) and heavy naphtha (C>19), considering similarity on such fraction obtained at 500 and 600 °C

On the other hand, the pyrolysis of SPR with catalyst presented the absence of such peak maximum, since bio-oil yield seemed to keep increasing with temperature. At 700 °C, the use of 20% Si-Al catalyst was able to produce 34.10 wt.% of bio-oil where its fraction were mainly composed by gasoline at around 80%, apart from the remaining 20% of diesel .

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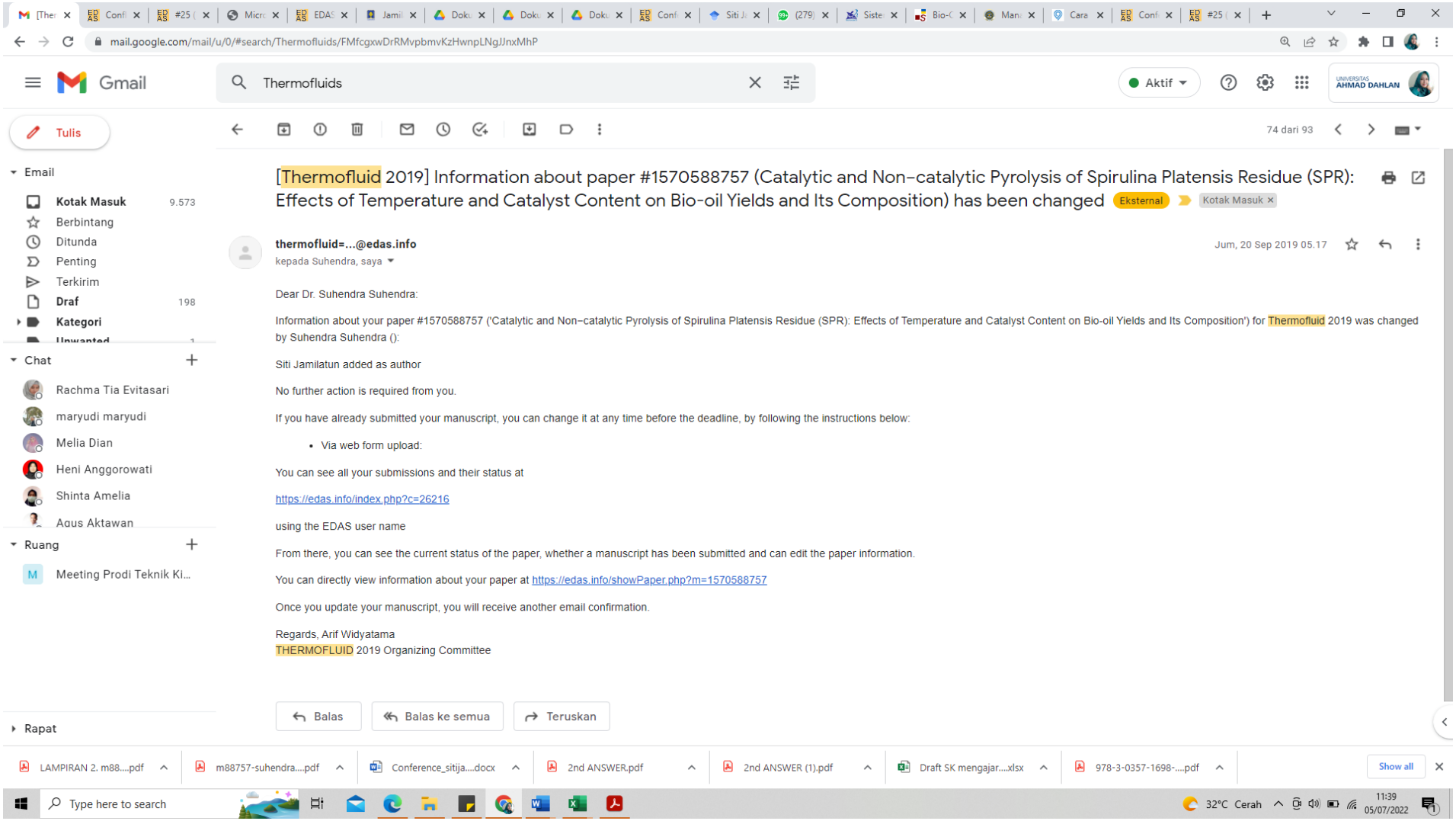
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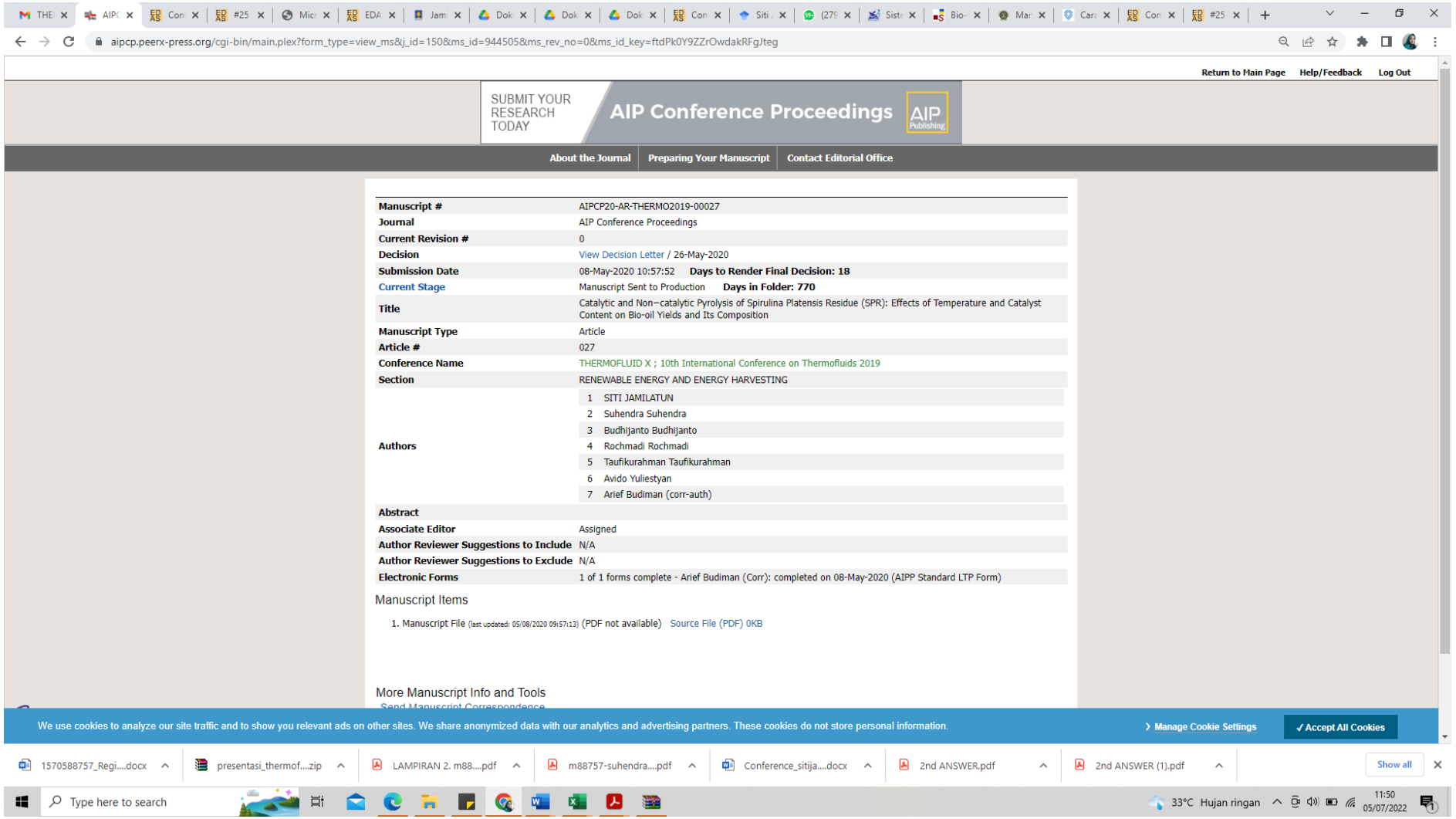

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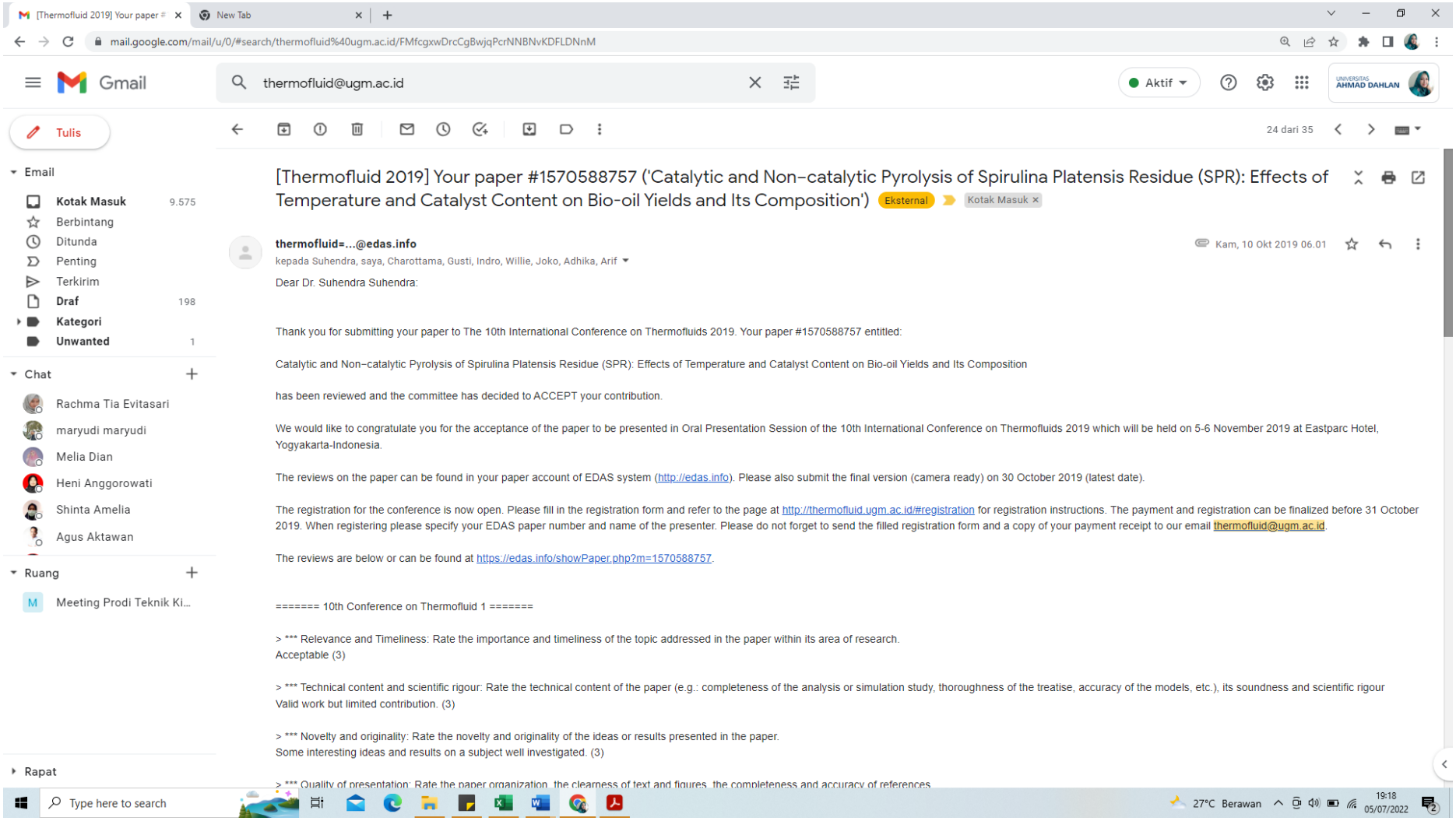
LAMPIRAN 5. PAPER REVIEW



[Thermofluid 2019] Your paper #1570588757 ('Catalytic and Non-catalytic Pyrolysis of Spirulina Platensis Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition')

Eksternal Kotak Masuk ×

thermofluid=...@edas.info
kepada Suhendra, saya, Charottama, Gusti, Indro, Willie, Joko, Adhika, Arif

Kam, 10 Okt 2019 06.01

Dear Dr. Suhendra Suhendra:

Thank you for submitting your paper to The 10th International Conference on Thermofluids 2019. Your paper #1570588757 entitled:

Catalytic and Non-catalytic Pyrolysis of Spirulina Platensis Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition

has been reviewed and the committee has decided to ACCEPT your contribution.

We would like to congratulate you for the acceptance of the paper to be presented in Oral Presentation Session of the 10th International Conference on Thermofluids 2019 which will be held on 5-6 November 2019 at Eastparc Hotel, Yogyakarta-Indonesia.

The reviews on the paper can be found in your paper account of EDAS system (http://edas.info). Please also submit the final version (camera ready) on 30 October 2019 (latest date).

The registration for the conference is now open. Please fill in the registration form and refer to the page at http://thermofluid.ugm.ac.id/#registration for registration instructions. The payment and registration can be finalized before 31 October 2019. When registering please specify your EDAS paper number and name of the presenter. Please do not forget to send the filled registration form and a copy of your payment receipt to our email thermofluid@ugm.ac.id.

The reviews are below or can be found at https://edas.info/showPaper.php?m=1570588757.

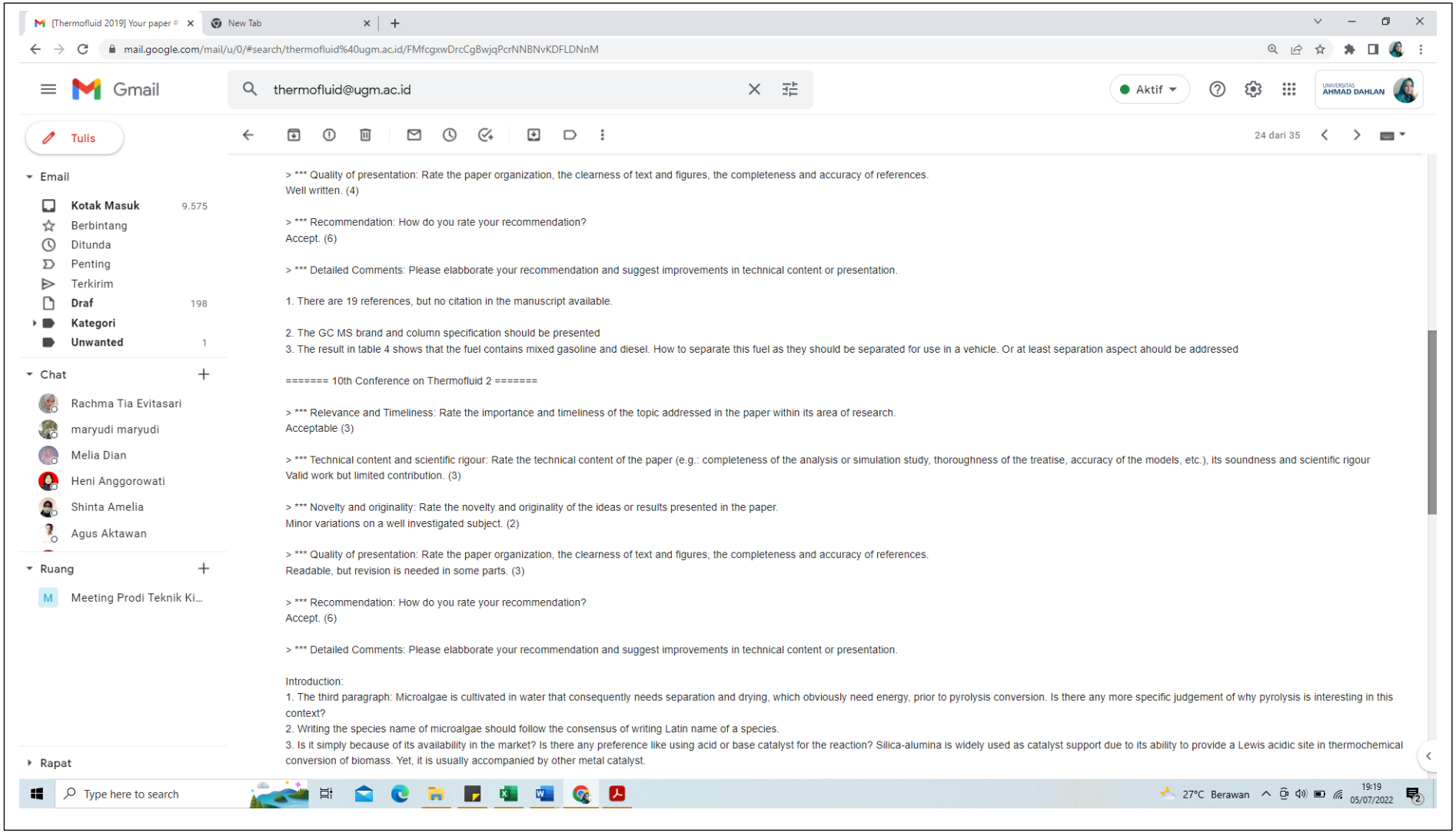
======= 10th Conference on Thermofluid 1 =======

> *** Relevance and Timeliness: Rate the importance and timeliness of the topic addressed in the paper within its area of research.
Acceptable (3)

> *** Technical content and scientific rigour: Rate the technical content of the paper (e.g.: completeness of the analysis or simulation study, thoroughness of the treatise, accuracy of the models, etc.), its soundness and scientific rigour
Valid work but limited contribution. (3)

> *** Novelty and originality: Rate the novelty and originality of the ideas or results presented in the paper.
Some interesting ideas and results on a subject well investigated. (3)

> *** Quality of presentation: Rate the paper organization, the clearness of text and figures, the completeness and accuracy of references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1. There are 19 references, but no citation in the manuscript available.
2. The GC MS brand and column specification should be presented
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Acceptable (3)
> *** Technical content and scientific rigour: Rate the technical content of the paper (e.g.: completeness of the analysis or simulation study, thoroughness of the treatise, accuracy of the models, etc.), its soundness and scientific rigour
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> *** Quality of presentation: Rate the paper organization, the clearness of text and figures, the completeness and accuracy of references.
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It is also correlated to the discussion in sub-section 3.2. The authors can focus on the GCMS results by grouping all the chemicals based on the groups (alkanes, carboxylic acids, aldehydes, ketones, etc.). This is very important to revise if the authors expect a publication in a journal.

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Additionally, we would like to inform you that there will be a city tour on the second day of the conference on Wednesday, 6 November 2019 The main destination is Ullen Sentalu Museum and Sentra batik, Yogayakarta

There will be no additional charge (facility for participants) to join this tour. However, we need your early confirmation (on join or do not join in this tour) to list all possibilites regarding this city tour.

Should you have any questions or difficulties in registering, please do not hesitate to contact us.

With kindest regards,
Arif Widyatama
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Thermofluid 2019

#25 (1570588757): Catalytic and Non−catalytic Pyrolysis of Spirulina Platensis Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition

#25 (1570588757): *Catalytic and Non−catalytic Pyrolysis of Spirulina Platensis Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition*

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Title	*Catalytic and Non−catalytic Pyrolysis of Spirulina Platensis Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition*
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Keywords		Spirulina platensis residue (SPR); Catalytic pyrolysis; Thermal decomposition; Bio-oil
Topics	Only the chairs (thermofluid2019-chairs@edas.info) can edit	Renewable Energy; Biofuels and biomass; Energy and Environment
Presenter(s)		Suhendra Suhendra
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Reviews

2 10th Conference on Thermofluids

Review 1

Relevance and Timeliness	Technical content and scientific rigour	Novelty and originality	Quality of presentation	Recommendation
Acceptable (3)	Valid work but limited contribution. (3)	Some interesting ideas and results on a subject well investigated. (3)	Well written. (4)	Accept. (6)

Detailed Comments (Please elabborate your recommendation and suggest improvements in technical content or presentation.)

1. There are 19 references, but no citation in the manuscript available.

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Review 2

Relevance and Timeliness	Technical content and scientific rigour	Novelty and originality	Quality of presentation	Recommendation
Acceptable (3)	Valid work but limited contribution. (3)	Minor variations on a well investigated subject. (2)	Readable, but revision is needed in some parts. (3)	Accept. (6)

Detailed Comments (Please elabborate your recommendation and suggest improvements in technical content or presentation.)

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Catalytic and Non−catalytic Pyrolysis of *Spirulina platensis* Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition

Siti Jamilatun[1], Suhendra[1], Budhijanto[2], Rochmadi[2], Taufikurahman[3], Avido Yuliestyan[4] and Arief Budiman[2,5*)]

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[4]*Department of Chemical Engineering, Faculty of Industrial Technology, Universitas Pembangunan Nasional "Veteran" Yogyakarta, Jalan SWK 104, Yogyakarta 55283, Indonesia*

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Abstract. Problems associated with the development of the first and second generations of biofuel, especially regarding its raw materials and complex processes, have led to the evolution of the third generation one, a microalgae-based bio-oil through pyrolysis. This research explored the effect of temperature ranging between 300−700 °C and the presence of Silica−alumina (Si-Al) catalyst at the variation of 5–20 wt.% on the pyrolysis process of *Spirulina platensis* microalgae solid residue, in search of optimum condition for collecting maximum bio-oil yield and its desired composition. The experiments without catalysts reached their optimum yield of 28 wt.% at 550 °C. While the involvement of 5 wt.% of Si-Al catalyst in the reactor resulted in a higher yield than that of without catalyst beyond 550 °C. Furthermore, a high amount of catalyst content at 20 wt.% seems to improve bio-oil yield collection up to 34.10 wt.%. Interestingly, referring to its C atoms number, SPR-based bio-oil by catalytic pyrolysis tended to produce such a considerable amount of gasoline leading to *Pertamax*, and the one without catalyst produced gasoline leading to *Pertalite*.

Keywords: *Spirulina platensis* residue (SPR), catalytic pyrolysis, thermal decomposition, bio-oil.

1. BACKGROUND

Despite being widely tested, first and second generations, biofuel leaves some issues regarding its availability and competition against the primary use of their raw materials (1,2). First-generation biofuels (i.e., biodiesel, corn ethanol, sugar alcohol) from vegetable oils and corn sugar sources are constrained by the limited feedstocks and competition with food products (3). In

addition, the second-generation biofuels (i.e., hydrotreating oil, bio-oil, lignocellulose ethanol, butanol, mixed alcohols) raise a problem for its extensive agricultural land requirement, although it comes from non-edible oil, non−food, cheap and abundant plant waste biomass sources (4,5). Such situations stimulate the researcher to find an alternative to overcome the problems mentioned before. Development of third-generation biofuels (biodiesel, bioethanol, biohydrogen, biomethane) based on microalgae biomass offers a simpler production process of pyrolysis and non-competing sources (6).

Moreover, microalgae may provides many advantages, such as: (i) environmentally friendly, (ii) not competing with food and agricultural land, (iii) very high oil products (iv) simple cellular structure, (v) lipid−rich (40−90% in percent dry weight), (vi) rapidly growing rate, (vii) able to grow in brine and poor conditions, (viii) evolved from CO_2 gas produced by power plants, and (ix) containing no sulfur, non toxic and highly biodegradable (7-9). On these ground, microalgae seem very potential to be developed as a source of energy for the future.

Basically, microalgae-based biofuel can be produced via biochemistry conversion, thermochemical conversion, chemical reaction, and direct burning. Among them, thermochemical conversion through the pyrolysis process seems quite promising due to its fairly simple process and without the need for additional chemicals (10-13,14). Pyrolysis is the thermochemical decomposition of organic matter through the process of heating without oxygen or with little oxygen or without other chemical reagent addition, where the raw material will undergo chemical structure breakdown into the gas phase (14).

The process at atmospheric pressure and a relatively low temperature between 350 and 600 °C can produce bio-oil, gas, and char for later being used as fuel and adsorbent, respectively (4). Therefore, such research in the area of microalgae-based bio-oil through pyrolysis gain significant priority.

Pyrolisis itself can be performed with or without catalyst (6,15-18). Study on pyrolysis without catalyst using various raw materials (i.e., *Chlorella Vulgaris*, *Spirulina plantesis* residue, and *Saccharina japonica*) reported optimum bio-oil yield ranging between 24.5 and 32.7 wt.% at a temperature between 500 – 550 °C. The use of catalysts does not necessarily improve bio-oil yield uptake, as demonstrated by Lim et al. (17), presenting a fairly similar yield on pyrolysis of Palm oil with Br_2O_3 catalyst. Neither changing the type of catalyst (i.e., ZnO and Al_2O_3) would result in a significant difference of bio-oil uptake from Eastern giant fennel (*Ferula Orientalis*) (18).

Pyrolysis study was conducted with a solid catalyst with HZSM-5 (19), hybrid composites (hexagonal mesoporous silicate (HMS) and ZSM-5 with impregnation of Ni, Fe, or Ce (20), metal oxides supported by TiO_2 (21), silica-alumina (19), and silica-alumina reuse (22). Silica - amorphous alumina is the most widely used solid acid catalyst in supporting the production of petrochemicals, chemicals, and renewable energy. High acidity (low Si/Al) can be used in the process of cracking petroleum to increase oxidation of CO (23). Rather than that, the use of catalysts may be seen as a composition changer to the extends of the way hydrocarbon is chemically modified. Silica-alumina (Si-Al), which widely available on the market as a cheap commodity, may play such a role.

This paper discusses the effect of temperature and catalyst (Si-Al) content on the pyrolysis of *Spirulina platensis* residue (SPR) on bio-oil yield and its composition. Product yields were observed referred to its the mass-basis of each component. Whilst gas chromatography/mass spectrometry (GC−MS) technique was employed for characterizing bio-oil compositions.

2. METHODS

2.1 Materials

2.1.1. *Spirulina platensis* Residue (SPR)

SPR was obtained from solid residue extraction of *Spirulina platensis* with a size of 80−140 mesh (0.177−0.105 mm). The proximate analysis was performed to characterize the moisture content, ash, volatile matter, and fixed carbon. Whilst

C, H, O, and N content were quantified by ultimate analysis. The calorific value, conducted at the Research and Development Center for Mineral and Coal Technology (TEKMIRA), Bandung, Indonesia, was done using a calorimeter bomb where the results are in Table 1.

Table 1: Main characteristic of SPR (15)

Components	(SPR)
Proximate analysis (wt.%)	
Moisture	9.99
Ash	8.93
Volatiles	68.31
Fixed carbon	12.77
Ultimate analysis (wt.%)	
Carbon	41.36
Hydrogen	6.60
Nitrogen	7.17
Oxygen	35.33
Sulfur	0.55
Lipid	0.09
Carbohydrate	25.59
Protein	49.60
Higher heating value (MJ/kg)	18.21

2.1.2. silica-alumina catalyst

The silica-alumina catalyst was kindly supplied by PT. Pertamina, Balongan, Indonesia. Silica-alumina properties, including the ratio, specific surface area, pore-volume, pore diameter, and SEM−EDX, were analyzed at LPPT−UGM (14,18). The results are shown in Table 2.

Table 2: Silica−alumina catalyst specifications (19)

Si-Al specification	Value
SiO_2/Al_2O_3	1.67
Pore surface area	240.553 m^2/g surface area
Pore diameter	3.3 nm
Average pore volume	0.199 cm^3/g total pore volume
Compositions (wt.%)	
C	12.33
O	55.73
Al	15.42
Si	16.51

2.2 Experimental methods

SPR pyrolysis was performed in a 40 cm ID and 60 cm length of a fixed−bed reactor system, depicted in Figure 1 (17).

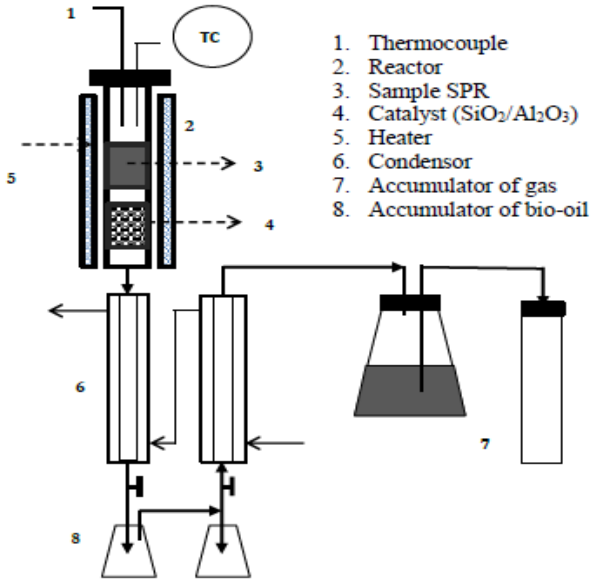

Fig. 1: The schematic diagram of the fixed bed tubular reactor (15)

The reactor consists of an upper and bottom part for placing SPR and Si-Al catalysts, respectively. A nickel wire heater, coiled outside the reactor tube, is provided for maintaining an operating temperature between 300 and 700 °C. In addition, the condenser was used for bio-oil and gas products for cooling. Fifty (50) g of SPR was inserted into the reactor chamber. The heat was then introduced at a constant rate of 10-20 °C/min from ambient to the processing temperature. Then, the pyrolysis process was always maintained for 1 hour. After the reaction finished, bio-oil and gas could be collected in their corresponding accumulator, following their path through the condenser for temperature cooling. It is noted that bio-oil would first require to be separated by decantation from its water in the liquid phase. Lastly, the amount of solid (biochar) left behind in the reactor chamber was picked up and weighed. Si-Al catalyst was used for pyrolysis of 5, 10, 15 and 20 wt.% (2.5; 5; 7.5 and 10 grams). Product data collection was

carried out at a temperature of 30 to the desired temperature, and it was then held for 1 hour to ensure that pyrolysis was running perfectly.

2.3. Tests and measurements

2.3.1. The weight percentage of yield product analysis

Weight of liquid products (refer to bio−oil and water), gas, and bio-char were calculated by the following equation (15):

$$Y_{bo} = (W_{bo}/W_M)x\ 100\ \% \quad \text{.....(1)}$$

$$Y_{wtr} = (W_{wtr}/W_M)\ x\ 100\ \% \quad \text{.....(2)}$$

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

$$Y_{Gas} = 100 - (Y_{liq} + Y_{Char}) \quad \text{.....(4)}$$

Y and *W* represent yield percentage and weight, respectively. Its corresponding subscripts, of M, bo, wtr, gas, and char, are for initial SPR, bio-oil, water, gas, and bio-char.

2.3.2. Characterization of bio-oil compositions

Bio-oil obtained from pyrolysis liquid product (light phase) was tested for its composition using Gas chromatography-mass spectrometry (GC-MS) with the Shimadzu brand. GC-MS was run at an injection temperature of 150 ºC, pressure 41.4 kPa with a total flow of 88.8 mL/min, column flow 0.85 mL/min, linear velocity 33.4 cm/sec, and purge flow 3.0 mL/min. The temperature in the oven column was initially 50 ºC (hold time 5 minutes), increasing to 290 ºC at a speed of 20 ºC/min. (hold time 8 minutes), and the maximum temperature reached 320 ºC with a speed of 15 ºC/min. (hold time 3 minutes). Then, based on the number of C constituent atoms, bio-oil compositions were grouped into fractions of LPG (C≤4), gasoline (C5-C11), diesel (C12-C18) and heavy naphtha (C≥19).

To compare the bio-oil fraction of pyrolysis results with and without catalyst with the fraction, fuel from fossils in the form of *pertalite*, *Pertamax* and diesel was tested with the GC-MS, and the results were divided into fractions C≤4, C5-C11, C12-C18 and C≥19 . Results of GC-MS of *pertalite*, *pertamax*, and diesel at the Integrated Research Laboratory of the Faculty of Pharmacy, Ahmad Dahlan University, Yogyakarta, are presented in Table 3.

Table 3. GC-MS results on *pertalite*, *pertamax* and diesel.

Type of fuel oil	Amount of C, wt.%			
	C≤4	C5-C11	C12-C18	C≥19
Pertalite	7.81	1.48	47.81	42.9
Pertamax	0	84.81	14.39	0.76
Diesel	0	3.35	73.68	22.96

3. RESULTS AND DISCUSSION

3.1. Effects of temperature and catalyst content on pyrolysis yield product

Figure 2a showed the yield products of bio−oil, gas, char, and water phase obtained from the pyrolysis process without using a catalyst for various temperatures between 300 and 700 °C. It might be expected that the more heat introduced into the system resulted in more hydrocarbon decomposition. Such a phenomenon was perhaps seen by the reduction of solid product (and bio-char) yield from 61.55 to 17.3 wt.% with increasing temperature, prior to stabilizing at 600 °C and above. Along with that, solid texture analysis exhibiting gradual changes from soft to hard texture was reported. This would mean decomposition initially occurred by releasing some hydrocarbon compounds, followed by gradual compositional changes, and finally reached bio-char stable composition (16-18). From another perspective, this would mean that the remaining hydrocarbon was converted to gas and liquid products.

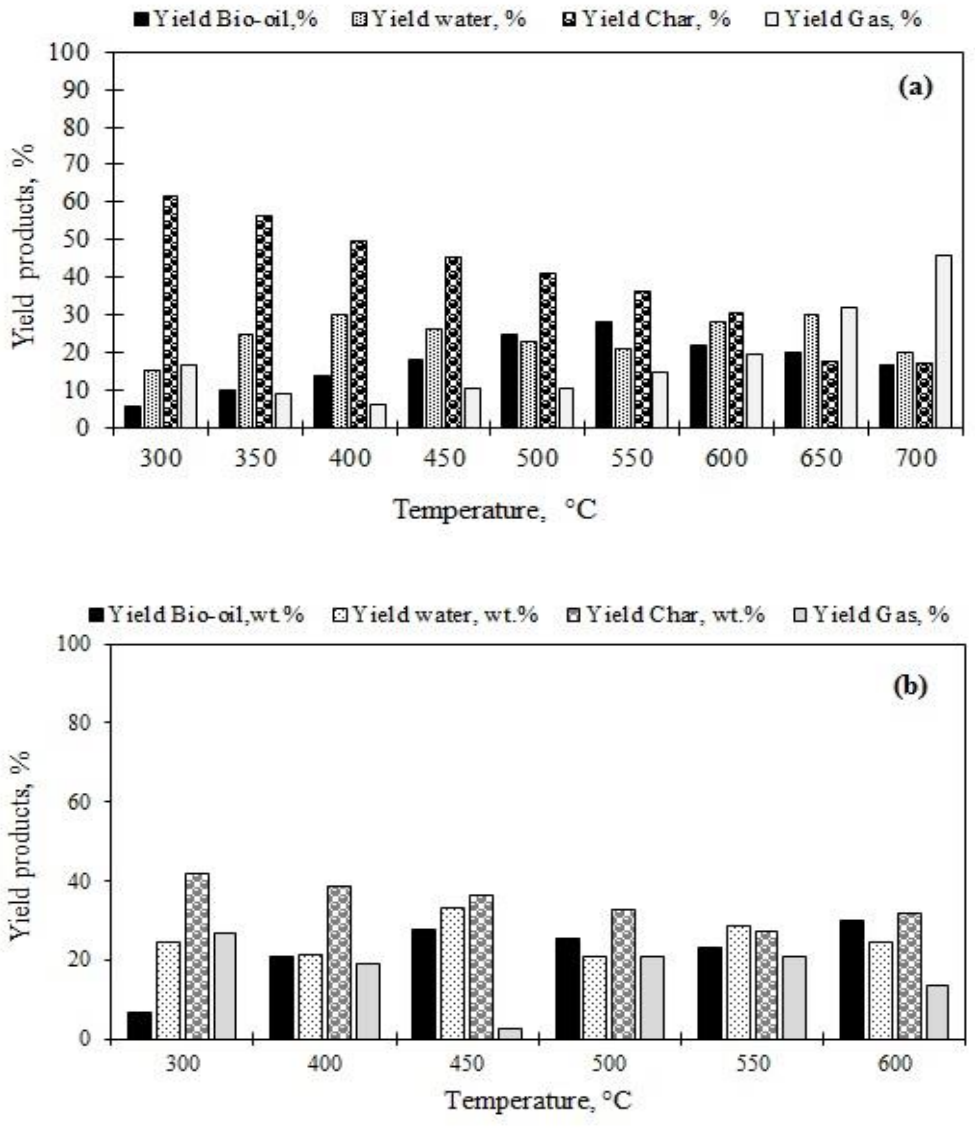

Fig. 2: Effect of temperature on yield products, obtained from SPR pyrolysis process a) without and b) with the presence of 5 wt.% Si-Al catalyst

The liquid products consisted of water, and bio-oil was initially collected at 300 °C. At this point, bio-oil presented the lowest yield of 6 wt.%. The trend of increasing bio-oil uptake was then observed, following their 50 °C incremental increase in processing

temperature up to 550 °C. Interestingly, discontinuation on that trend, seen by sudden yield decrease, resulted in considering 550 °C as peak maximum temperature for SPR pyrolysis without a catalyst. Such a phenomenon was quite typical, as similarly reported elsewhere (24). Above 600 ºC, bio-oil yields tended to decrease due to the secondary cracking reaction that resulted in further decomposition into gas and char (24). Response to bio-oil secondary cracking might be confirmed by the inverted gas yield plot presenting a peak minimum at a temperature between 300 and 400 °C, followed by sharp yield increase to 45.7 wt.% at 700 °C.

For a comparative analysis, the yield result of pyrolysis at various temperatures in the presence of 5 wt.% Si-Al catalyst is presented in Figure 2b. As observed, the trend of biochar and water yield remain fairly similar. However, no such peak maximum saw on bio-oil yield since it would rather show an increase of bio-oil with increasing temperature. Conversely, gas yield fluctuated, followed by a tendency to decrease at the later stage around 550 °C and above, in the presence of a catalyst.

Hence to further understand the influence of 5 % Si-Al catalyst, each yield in Figure 2b was then compared against results shown in Figure 2a for their corresponding temperature. It was evident that the catalyst was able to boost bio-oil uptake at a temperature between 300 and 450 °C, considered to be primary phase decomposition. Moreover, the more significant bio-oil yield was perceived for the pyrolysis process conducted at a temperature higher than 550 °C, taking into account that the relative data on the curve "without catalyst" lean toward decreasing trend as opposed to increasing trend on the one with catalyst.

As the focus was paid on maximizing bio-oil uptake, there might be a possibility that the amount of bio−oil would further increase with temperature. For that reason, the SPR pyrolysis with Si-Al catalyst was further experimented to study the effect of the amount % catalyst between 5 and 20 % at the highest temperature previously tested of 700 °C, where the results are shown in Figure 3.

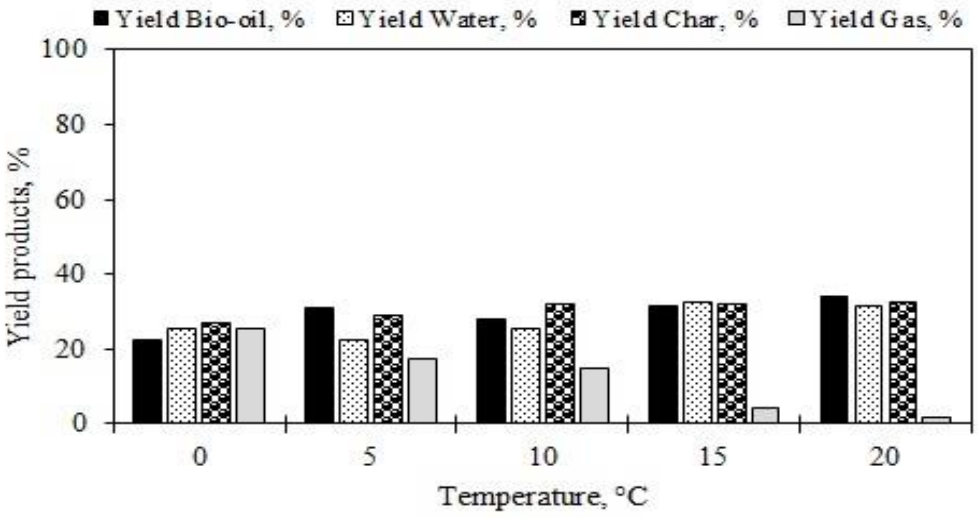

Fig. 3: The evolution of yield product percentage as a function of Si-Al catalyst percentage between 0 and 20 wt.% on SPR pyrolysis products at 700 °C

As seen, the result presented that yield gas decrease quite significantly from 17.47 to 1.70 wt.% with the increase of catalyst involved from 0 to 20 wt.%. Consequently, liquid products and bio-char seemed to increase. Liquid products, a total amount of Bio−oil and water, were increased by ±17.00 wt.% from 48.04 to 65.10 wt.% in which 12 wt.% increase was contributed from the raise of bio-oil uptake. In fact, the involvement of catalysts by only 5 wt.% has led to an increase of about 8%. A higher Si-Al

catalyst percentage tested between 5 and 20 wt.% did not cause a significant rise despite its increasing trend. Regardless, its concurrent decrease in gas and bio-oil increase might suggest the occurrence of vapor condensation.

3.2. Fractional composition (based on C-number category range) of bio−oil products as a function of temperature and catalyst content

Table 4 presented the fractional composition, referring to their number of C atoms (LPG, gasoline, diesel, and heavy naphtha) of bio-oil obtained by pyrolysis without catalyst at a temperature of 300, 400, 500 and 600 °C.

Table 4: Bio-oil composition referring to its C-atom category, obtained at various pyrolysis temperature between 300 and 600 °C, without catalyst

Pyrolysis temperature, °C	Amount of C, wt.%			
	C≤4 (LPG)	C5−C11 (Gasoline)	C12−C18 (Diesel)	C≥19 (*Heavy Naptha*)
300	16.60	64.50	9.73	0.09
400	0.66	52.14	25.97	21.26
500	−	15.32	49.53	35.12
600	−	15.66	49.92	36.94

At 300 °C, bio−oil consisted of LPG (C≤4), gasoline (C5−C11), diesel (C12−C18), and heavy naphtha (C≥19) in which their corresponding content was 16.60, 64.50, 9.73, and 0.09 wt.%, respectively. At 400 °C, LPG fraction started to disappear, showing only 0.66 wt.% collected. As expected, this fraction was not found at temperature 500−600 °C. In addition, gasoline fraction also exhibited a sharp decrease in weight percentage from 64.50 to 15.66 with the raise of tested temperature. Consequently, other fractions would rise, given the fact that diesel increased from 9.73 at 300 °C to 49.92 wt.% at 600 °C, besides the presence of 36.94 wt.% of heavy naphtha at 600 °C from almost none at 300 °C. By combining information summarized in Figure 2a and Table 4, bio-oil uptake could be obtained maximum at 550 °C, where its composition likely consisted of about 15, 50, and 35, respectively, for LPG, gasoline, and diesel. Interestingly, such composition seemed to present its potential for being used as future fuel due to its similarity on hydrocarbon content as compared to fuel derived from crude oil (17). Table 3 shows that the GC-MS test results for *pertalite* petroleum fuels consist of C≤4, C5-C11, C12-C18, and C≥19, respectively, with the values of 7.81, 1.48, 47.81 and 42.90 wt.%. It can be concluded that the bio-oil produced by pyrolysis of the SPR at a temperature of 500-600 ºC is equivalent to *pertalite*.

Table 5 presented the result of LPG, Diesel, and Heavy naphtha fraction on bio-oil yield obtained from the pyrolysis process with Si-Al catalyst having content from 5 to 20 wt.%. In the presence of a catalyst, none of the heavy naphthas was produced regardless of the catalyst content to suggest the suppression of longer chain hydrocarbon production. In addition, diesel, having chain numbers between 12 and 18, exhibited a decrease as well. Thereof, gasoline was the major fraction produced, around 80 wt.%.

The influence of catalyst content on bio-oil fraction was rather inconclusive at this point. Statistically, gasoline and diesel fraction composition were around 80 and 20 wt.% Regardless of the catalyst content, except for the result when using 10 wt.% of Si-Al catalyst. This great amount of gasoline fraction agreed with the study reported by Sunarno 2018 (10) that the rise in pyrolysis temperature and the use of silica-alumina catalysts by impregnation of Ni metal will increase the gasoline, kerosene and phenol

fractions, while, the oxygenate compound will decrease. In any case, the introduction of only 5 wt.% Si-Al catalyst was able to drive the pyrolysis process to lean toward the production of gasoline, which would be prospective to replace crude oil-based gasoline fuel. The existence of about 20 wt.% of the C12-C18 (diesel) fraction is still tolerated as a by-product in gasoline. Table 3 shows that the GC-MS test results for fuel from *Pertamax* petroleum consist of C≤4, C5-C11, C12-C18, and C≥19, respectively, with values of 0, 84.81, 14.39, and 0.76 %. It can be concluded that with the use of Si-Al catalysts: 5, 15, and 20 %, the bio-oil fraction consists of C5-C11 with an average of 82.84 % and C12-C18 with an average of 16.73 %, the resulting values lead to *Pertamax*.

Table 5: Component of bio-oil SPR based on the amount of C on various wt.% of the amount of Silica−alumina catalyst

Catalyst (%)	Amount of C, wt.%		
	C≤4 (LPG)	C5−C11 (Gasoline)	C12−C18 (Diesel)
5	-	80.06	19.34
10	-	41.48	57.06
15	-	86.56	13.13
20	-	81.89	17.73

4. CONCLUSION

This research aimed to study the influence temperature and catalyst content on the pyrolysis process of *Spirulina Plantesis* Residue (SPR) with and without the Silica-Alumina (Si-Al) catalyst. Pyrolysis process of SPR, for both without and with the catalyst, produced liquid product (bio−oil and water phase), gas, and char. On the basis of weight percentage, the optimum bio-oil uptake of 28 wt.% was obtained from pyrolysis at temperature 550 °C, considered as a peak maximum within the range of tested temperature (300-700 °C). At this temperature, the resulting bio−oil compositions were likely to consist of gasoline (C5−C11), diesel (C12−C18) and heavy naphtha (C≥19), considering similarity on such fraction obtained at 500 and 600 °C

On the other hand, the pyrolysis of SPR with catalyst presented the absence of such peak maximum, since bio-oil yield seemed to keep increasing with temperature. At 700 °C, the use of 20 wt.% Si-Al catalyst was able to produce 34.10 wt.% of bio-oil, where its fraction was mainly composed of gasoline leading to *pertamax*, whereas without a catalyst, it led to *pertalite* by referring the number of C atoms in bio-oil.

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Parallel Session Schedules (Judul diatas author)

ROOM: Orchid		
Track/area: Automotive and Aerospace Engineering, Fluid Dynamics Time: 13.10-16.15		
Chairperson: Dr. M. Agung Bramantya		
TIME (GMT +7 WIB)	CODE	TITLES AND AUTHORS
13.10 - 13.25	FD1	**Effect of Velocity on The Formation of Rolled-up Vortex on The Delta Wing Using a Water Tunnel Technique** Author: Setyawan Bekti Wibowo, Sutrisno, Tri Agung Rohmat, Soeadgihardo, Siswantoro, David Fernando and Wega Naufal
13.25 - 13.40	FD2	**Effects of Swirl Blade Angle on The Non-Premixed Flame Stability in Radial Fuel Flow Type Burner** Author: Pri Hestuwati and Tri Agung Rohmat
13.40 - 13.55	AA1	**Effects of the Area Rule Theory to the Fuselage for a Surveillance Unmanned Aerial Vehicle Using Computational Fluid Dynamics Methods** Author: Muhammad Agung Bramantya and Joseph Putra Nararya
13.55 - 14.10	AA2	**Study of the Effect of 4-Digit NACA Variation on Airfoil Performance using Computation Fluid Dynamics** Author: Ray Ginting and Muhammad Agung Bramantya
14.10 - 14.25	AA3	**Analysis of Liquid Rocket Attitude and Trajectory Estimation** Author: Idris Eko Putra and Arif Nur Hakim
14.25 - 14.40	AA4	**Effect of Undertray Inlet Angle and Drag Reduction System on Aerodynamics Performance at Student Formula Car Using Numerical Simulation** Author: Fauzun and Gilang Sandy Firdaus
14.40 - 14.55	AA5	**The Influence of Plenum Geometry Toward Engine Performance at BM Student Formula Car** Author: Fauzun and Arinta Budhi Nugraha
14.55-15.10	FD3	**A 2-D CFD Model of Albumin-rich-fluid distribution in Human Subcutaneous Tissue for Early Stage of Lymphedema Prediction** Author: Irfan Aditya Dharma, Marlin Ramadhan Baidillah, Michiko Sugawara and Masahiro Takei
15.10-15.25	FD4	**Experimental Study of Particle Separation using Fluidised Bed Method** Author: Achmad Choirul Anam and Nur Ikhwan
15.25-15.40	FD5	**Effect of Impeller Trailing Edge Shape to the Radial Compressor Performance** Author: Putra Adnan Fadilah, Firman Hartono and Dadang Furqon Erawan
15.40-15.55	Coffee Break and Pray	

ROOM: Magnolia		
Track/area: Multiphase Flow Time: 13.10-16.15		
Chairperson: Dr. Deendarlianto		
TIME (GMT +7 WIB)	CODE	TITLES AND AUTHORS
13.10 - 13.25	MP1	**Mechanistic Multiphase Flow Modeling: A New Approach for Gas Lift Design Using Dimensionless Scaling Curve for Tubing Size Optimization** Author: Ardhi Hakim Lumban Gaol, Wijoyo Niti Daton, Prasandi Abdul Aziz, Steven Chandra and Hanif Farrastama Yoga
13.25 - 13.40	MP2	**The Performance and Flow Characteristics of Swirl Flow Injector Type Airlift Pump System** Author: IGNB Catrawedarma, Deendarlianto and Indarto
13.40 - 13.55	MP3	**Experimental Study on the Effect of Submergence Ratio and Air Flow Rate on the Characteristics of Liquid-Gas-Solid Three-Phase Airlift Pump** Author: Ramdhani, Indarto, Deendarlianto and IGNB Catrawedarma
13.55 - 14.10	MP4	**Experimental Study of the Effect of the Swirl Flow on the Characteristics of Microbubble Generator Orifice Type** Author: Drajat Indah Mawarni, Akmal Abdat, Indarto, Deendarlianto, Wiratni and Wibawa Endra Juwana
14.10 - 14.25	MP5	**The Characteristics of Liquid-Gas-Solid Three Phase Airlift Pump for Particle Diameter Mesh 24-20 and 16-12** Author: Yulistiansah Agustiansah, Indarto, Deendarlianto and IGNB Catrawedarma
14.25 - 14.40	MP6	**Visualization Study on The Flow Pattern of Gas-Solid-Liquid Three-Phase Flow in Upriser Airlift Pump** Author: Nurmala Dyah F, Deendarlianto ,Indarto and IGNB Catrawedarma
14.40 - 14.55	MF7	**The Lattice Boltzmann Meshless Simulation of Multiphase Interfacial-Instability**. Author: Bahrul Jalaali and Pranowo
14.55-15.10	MP8	**Investigation on the effect of particle size in dissolution mass transfer inside porous media with micro-tomography** Author: Anindityo Patmonoaji, Yingxue Hu, Chunwei Zhang, Kento Tsuji and Tetsuya Suekane
15.10-15.25	MP9	**The Effect of Weber Number on the Dynamic Contact Angle During the Impacting of Single Droplet onto a Hot Oblique Surface** Author: Ian Adi Prabowo, Deendarlianto, Indarto and Teguh Wibowo
15.25-15.40	Coffee Break and Pray	

ROOM: Carnation		
Track/area: Heat Transfer and Phase Change Time: 13.10-16.15		
Chairperson: Dr. Adhika Widyaparaga		
TIME (GMT +7 WIB)	CODE	TITLES AND AUTHORS
13.10 - 13.25	HT1	**Analysis of The Effect of Baffle Arrangement on Gas-Glycol Type Shell and Tube Heat Exchanger Using Computational Fluid Dynamics Simulation** Author: Aryo Tejo and Tri Agung Rohmat
13.25 - 13.40	HT2	**Analysis of Surface Wettability on Condensation** Author: Zulfa Khalida Devina Rosa Hendarti and Hadi Rahmad
13.40 - 13.55	HT3	**Heat Transfer Characteristic of Proposed Heat Transfer Configurations of Temperature Chamber Design for Energy Test Refrigerator** Author: Bayu Utomo, Qudsiyyatul Lailiyah, Prayoga Bakti and Intan Paramudita
13.55 - 14.10	HT4	**Development of Portable Grashof Incubator Type A up to H Using Digital Thermostat W1209 to Improve Heat Performance According to SNI IEC 60601-2-19: 2014 Criteria** Author: Ibnu Roihan, Raldi Artono Koestoer and William Jerrel Iskandar
14.10 - 14.25	HT5	**Flow Past a Heated Rotating Horizontal Cylinder in Cross-Flow** Author: Koustubh R Bhagat and Pritanshu Ranjan
14.25 - 14.40	HT6	**Approaches for Modeling Burner Heat Impact During the Fire Resistance Tests** Author: Konstantin Y. Mokhov, Artem V. Pogodin, Andrey Y. Kudryavtsev, Alexander A. Ryabov and Dmitry Y. Strelets
14.40 - 14.55	HT7	**3D Simulation on Convective Drying Process for Cylindrical Tea Particle Using CFD Software to Analyse The Heat and Mass Transfer Phenomenons** Author: E Yohana, N Sinaga, M E Yulianto, V Paramita, H Pachusadewo and M I Nugraha
14.55-15.10	HT8	**An Overview Of Heat Transfer Enhancement on Double Pipe Heat Exchanger** Author: Mustaza Ma'a, Tri Agung Rohmat and Samsul Kamal
15.10-15.25	Coffee Break and Pray	

ROOM: Heliconia		
Track/area: Renewable Energy and Energy Harvesting Time: 13.10-16.15		
Chairperson: Dr. Khasani		
TIME (GMT +7 WIB)	CODE	TITLES AND AUTHORS
13.10 - 13.25	RE1	**The Low-Medium Enthalpy Geothermal Power Plant at Lahendong, Indonesia** Author: Lina Agustina and Suyanto
13.25 - 13.40	RE2	**Preliminary Study on HTL Processes of Biomass for Biofuels: Bio Crude Oil** Author: Joni Prasetyo, Galuh Wirama Murti, Agus Kismanto, SD Sumbogo Murti, Agus Rekso, Arie Rahmadi and Hens Saputra
13.40 - 13.55	RE3	**Catalytic and Non−catalytic Pyrolysis of Spirulina Platensis Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition** Author: Siti Jamilatun, Suhendra, Budhijanto, Rochmadi, Taufikurahman, Avido Yuliestyan and Arief Budiman
13.55 - 14.10	RE4	**Biodiesel Synthesis from Used Cooking Oil Using Calcium Oxide (CaO) Catalyst from Chicken Bones** Author: Siti Miskah, Tine Aprianti and Yadi Utama
14.10 - 14.25	RE5	**Numerical Simulation on the Separation Process of Liquid-Solid Two-Phase Flow in the Hydrocyclone Separator Applicable in Geothermal Power Plant** Author: Khasani and Albertus Whisnu Luky Febiatmoko
14.25 - 14.40	RE6	**Equipment Design for Water Treatment Geothermal Hot Spring Bath in Leilem Village, North Sulawesi** Author: Brenda Claudia Mauren and Andang Widiharto
14.40 - 14.55	RE7	**Prediction of the Performance of the Co-axial Thermoacoustic Engine (CoATE) using DeltaEC** Author: Sugiyanto, Samsul Kamal, Joko Waluyo, and Adhika Widyaparaga
14.55 -15.10	Coffee Break and Pray	

ROOM: Lotus		
Track/area: Conversion Energy and Energy Efficiency Time: 13.10-16.15		
Chairperson: Akmal Irfan Majid, S.T., M.Eng.		
TIME (GMT +7 WIB)	CODE	TITLES AND AUTHORS
13.10 - 13.25	EE1	**Effect of Room Temperature Set Points on Energy Consumption in a Residential Air Conditioning** Author: Cecep Sunardi, Yudi Prana Hikmat, Ade Suryatman Margana, Kasni Sumeru and Mohamad Firdaus Bin Sukri
13.25 - 13.40	EE2	**The Effects of Wind Orientation on the Performance of a Split Air Conditioning Unit** Author: Andriyanto Setyawan
13.40 - 13.55	EE3	**Experimental Study of the Effects of Input Voltage on the Transient Temperature Behaviors of Thermoelectric Mini-Cold Storage** Author: Muhammad Aulia Rahman, Christoforus Yacob Sianipar, Akmal Irfan Majid, Arif Widyatama and Suhanan Suhanan
13.55 - 14.10	EC1	**Energy and Exergy Analysis of Renewable Multi-Generation System** Author: Vontas Alfenny Nahan and Audrius Bagdanavicius
14.10 - 14.25	EC2	**Vapor/Liquid Equilibrium Measurement of Gasoline (Petrosol CA/CB/CC) and Ethanol Mixture**. Author: Rizky Tetrisyanda, Annas Wiguno and Gede Wibawa
14.25 - 14.40	EC3	**Experimental Investigation on Performance of Air Conditioning System Using Dedicated Subcooling** Author: Triaji Pangripto, Markus, A.P. Edi Sukamto, Kasni Sumeru and Mohamad Firdaus Bin Sukri
14.40 - 14.55	EC4	**The Effect of Water Mass Flow Rate and Pressure on Specific Energy Consumption and Specific Aquades Production Using Throttling Process Method** Author: Engkos Achmad Kosasih, Salsabil Dwikusuma Prasetyo, Muhammad Badra Shidqi and Fakhri Rabbani Putranto
14.55-15.10	EC5	**The Effect of Gas Composition, Air Intake Cooling, and Steam Injection on Combined Cycle Power Plant Performance** Author: Annas Wiguno, Rizky Tetrisyanda and Gede Wibawa
15.10-15.25	Coffee Break and Pray	

Catalytic and non–catalytic pyrolysis of Spirulina platensis residue (SPR): Effects of temperature and catalyst content on bio-oil yields and its composition

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Catalytic and Non−Catalytic Pyrolysis of Spirulina Platensis Residue (SPR): Effects of Temperature and Catalyst Content on Bio-oil Yields and Its Composition

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Abstract. Problems associated with the development of the first and second generations of biofuel, especially regarding its raw materials and complex processes, have led to the evolution of the third generation one, a microalgae-based bio-oil through pyrolysis. This research explored the effect of temperature ranging between 300−700 °C and the presence of Silica−alumina (Si-Al) catalyst at the variation of 5–20 wt.% on the pyrolysis process of *Spirulina platensis* microalgae solid residue, in search of optimum condition for collecting maximum bio-oil yield and its desired composition. The experiments without catalysts reached their optimum yield of 28 wt.% at 550 °C. While the involvement of 5 wt.% of Si-Al catalyst in the reactor resulted in a higher yield than that of without catalyst beyond 550 °C. Furthermore, a high amount of catalyst content at 20 wt.% seems to improve bio-oil yield collection up to 34.10 wt.%. Interestingly, referring to its C atoms number, SPR-based bio-oil by catalytic pyrolysis tended to produce such a considerable amount of gasoline leading to *Pertamax*, and the one without catalyst produced gasoline leading to *Pertalite*.

INTRODUCTION

Despite being widely tested, first and second generations, biofuel leaves some issues regarding its availability and competition against the primary use of their raw materials [1,2]. First-generation biofuels (i.e., biodiesel, corn ethanol, sugar alcohol) from vegetable oils and corn sugar sources are constrained by the limited feedstocks and competition with food products [3]. In addition, the second-generation biofuels (i.e., hydrotreating oil, bio-oil, lignocellulose ethanol, butanol, mixed alcohols) raise a problem for its extensive agricultural land requirement, although it comes from non-edible oil, non−food, cheap and abundant plant waste biomass sources [4,5]. Such situations stimulate the researcher to find an alternative to overcome the problems mentioned before. Development of third-generation

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biofuels (biodiesel, bioethanol, biohydrogen, biomethane) based on microalgae biomass offers a simpler production process of pyrolysis and non-competing sources [6].

Moreover, microalgae may provides many advantages, such as: (i) environmentally friendly, (ii) not competing with food and agricultural land, (iii) very high oil products (iv) simple cellular structure, (v) lipid−rich (40−90% in percent dry weight), (vi) rapidly growing rate, (vii) able to grow in brine and poor conditions, (viii) evolved from CO_2 gas produced by power plants, and (ix) containing no sulfur, non toxic and highly biodegradable [7-9]. On these ground, microalgae seem very potential to be developed as a source of energy for the future.

Basically, microalgae-based biofuel can be produced via biochemistry conversion, thermochemical conversion, chemical reaction, and direct burning. Among them, thermochemical conversion through the pyrolysis process seems quite promising due to its fairly simple process and without the need for additional chemicals [10-13,14]. Pyrolysis is the thermochemical decomposition of organic matter through the process of heating without oxygen or with little oxygen or without other chemical reagent addition, where the raw material will undergo chemical structure breakdown into the gas phase [14].

The process at atmospheric pressure and a relatively low temperature between 350 and 600 °C can produce bio-oil, gas, and char for later being used as fuel and adsorbent, respectively [4]. Therefore, such research in the area of microalgae-based bio-oil through pyrolysis gain significant priority.

Pyrolisis itself can be performed with or without catalyst [6,15-18]. Study on pyrolysis without catalyst using various raw materials (i.e., *Chlorella Vulgaris, Spirulina plantesis* residue, and *Saccharina japonica*) reported optimum bio-oil yield ranging between 24.5 and 32.7 wt.% at a temperature between 500 – 550 °C. The use of catalysts does not necessarily improve bio-oil yield uptake, as demonstrated by Lim et al. [17], presenting a fairly similar yield on pyrolysis of Palm oil with Br_2O_3 catalyst. Neither changing the type of catalyst (i.e., ZnO and Al_2O_3) would result in a significant difference of bio-oil uptake from Eastern giant fennel (*Ferula Orientalis*) [18].

Pyrolysis study was conducted with a solid catalyst with HZSM-5 (19), hybrid composites (hexagonal mesoporous silicate (HMS) and ZSM-5 with impregnation of Ni, Fe, or Ce (20), metal oxides supported by TiO_2 (21), silica-alumina]19], and silica-alumina reuse [22]. Silica - amorphous alumina is the most widely used solid acid catalyst in supporting the production of petrochemicals, chemicals, and renewable energy. High acidity (low Si/Al) can be used in the process of cracking petroleum to increase oxidation of CO [23]. Rather than that, the use of catalysts may be seen as a composition changer to the extends of the way hydrocarbon is chemically modified. Silica-alumina [Si-Al], which widely available on the market as a cheap commodity, may play such a role.

This paper discusses the effect of temperature and catalyst (Si-Al) content on the pyrolysis of *Spirulina platensis* residue (SPR) on bio-oil yield and its composition. Product yields were observed referred to its the mass-basis of each component. Whilst gas chromatography/mass spectrometry (GC−MS) technique was employed for characterizing bio-oil compositions.

METHODS

Materials

Spirulina platensis Residue (SPR)

SPR was obtained from solid residue extraction of Spirulina platensis with a size of 80−140 mesh (0.177−0.105 mm). The proximate analysis was performed to characterize the moisture content, ash, volatile matter, and fixed carbon. Whilst

C, H, O, and N content were quantified by ultimate analysis. The calorific value, conducted at the Research and Development Center for Mineral and Coal Technology (TEKMIRA), Bandung, Indonesia, was done using a calorimeter bomb where the results are in Table 1.

TABLE 1: Main characteristic of SPR [15]

Components	(SPR)
Proximate analysis (wt.%)	
Moisture	9.99
Ash	8.93
Volatiles	68.31
Fixed carbon	12.77
Ultimate analysis (wt.%)	
Carbon	41.36
Hydrogen	6.60
Nitrogen	7.17
Oxygen	35.33
Sulfur	0.55
Lipid	0.09
Carbohydrate	25.59
Protein	49.60
Higher heating value (MJ/kg)	18.21

Silica-alumina catalyst

The silica-alumina catalyst was kindly supplied by PT. Pertamina, Balongan, Indonesia. Silica-alumina properties, including the ratio, specific surface area, pore-volume, pore diameter, and SEM−EDX, were analyzed at LPPT−UGM [14,18]. The results are shown in Table 2.

TABLE 2: Silica−alumina catalyst specifications [19]

Si-Al specification	Value
SiO_2/Al_2O_3	1.67
Pore surface area	240.553 m^2/g surface area
Pore diameter	3.3 nm
Average pore volume	0.199 cm^3/g total pore volume
Compositions (wt.%)	
C	12.33
O	55.73
Al	15.42
Si	16.51

Experimental methods

SPR pyrolysis was performed in a 40 cm ID and 60 cm length of a fixed−bed reactor system, depicted in Fig. 1 [17].

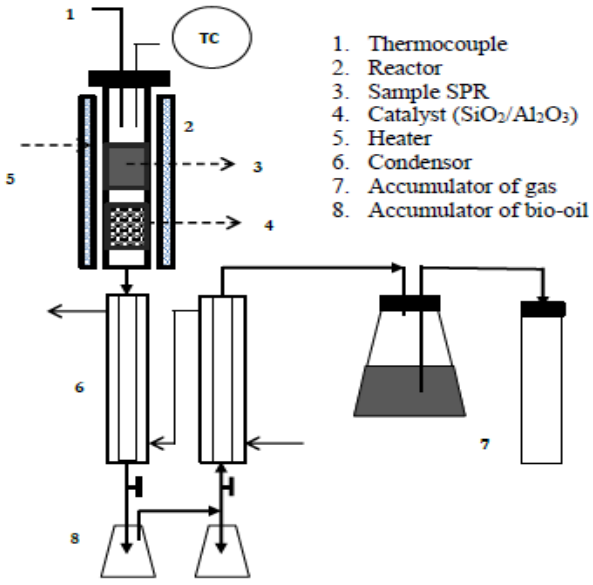

FIGURE. 1: The schematic diagram of the fixed bed tubular reactor [15]

The reactor consists of an upper and bottom part for placing SPR and Si-Al catalysts, respectively. A nickel wire heater, coiled outside the reactor tube, is provided for maintaining an operating temperature between 300 and 700 °C. In addition, the condenser was used for bio-oil and gas products for cooling. Fifty (50) g of SPR was inserted into the reactor chamber. The heat was then introduced at a constant rate of 10-20 °C/min from ambient to the processing temperature. Then, the pyrolysis process was always maintained for 1 hour. After the reaction finished, bio-oil and gas could be collected in their corresponding accumulator, following their path through the condenser for temperature cooling. It is noted that bio-oil would first require to be separated by decantation from its water in the liquid phase. Lastly, the amount of solid (biochar) left behind in the reactor chamber was picked up and weighed. Si-Al catalyst was used for pyrolysis of 5, 10, 15 and 20 wt.% (2.5; 5; 7.5 and 10 grams). Product data collection was carried out at a temperature of 30 to the desired temperature, and it was then held for 1 hour to ensure that pyrolysis was running perfectly.

Tests and measurements

The weight percentage of yield product analysis

Weight of liquid products (refer to bio−oil and water), gas, and bio-char were calculated by the following equation [15]:

$$Y_{bo} = (W_{bo}/W_M)x\ 100\ \% \quad (1)$$

$$Y_{wtr} = (W_{wtr}/W_M)\ x\ 100\ \% \quad (2)$$

$$Y_{Char} = (W_{Char}/W_M)\ x\ 100\ \% \quad (3)$$

$$Y_{Gas} = 100 - \left(Y_{liq} + Y_{Char}\right) \quad (4)$$

Y and W represent yield percentage and weight, respectively. Its corresponding subscripts, of M, bo, wtr, gas, and char, are for initial SPR, bio-oil, water, gas, and bio-char.

Characterization of Bio-oil Compositions

Bio-oil obtained from pyrolysis liquid product (light phase) was tested for its composition using Gas chromatography-mass spectrometry (GC-MS) with the Shimadzu brand. GC-MS was run at an injection temperature of 150 ºC, pressure 41.4 kPa with a total flow of 88.8 mL/min, column flow 0.85 mL/min, linear velocity 33.4 cm/sec, and purge flow 3.0 mL/min. The temperature in the oven column was initially 50 ºC (hold time 5 minutes), increasing to 290 ºC at a speed of 20 ºC/min. (hold time 8 minutes), and the maximum temperature reached 320 ºC with a speed of 15 ºC/min. (hold time 3 minutes). Then, based on the number of C constituent atoms, bio-oil compositions were grouped into fractions of LPG (C≤4), gasoline (C5-C11), diesel (C12-C18) and heavy naphtha (C≥19).

To compare the bio-oil fraction of pyrolysis results with and without catalyst with the fraction, fuel from fossils in the form of pertalite, Pertamax and diesel was tested with the GC-MS, and the results were divided into fractions C≤4, C5-C11, C12-C18 and C≥19 . Results of GC-MS of pertalite, pertamax, and diesel at the Integrated Research Laboratory of the Faculty of Pharmacy, Ahmad Dahlan University, Yogyakarta, are presented in Table 3.

TABLE 3. GC-MS results on *pertalite*, *pertamax* and diesel.

Type of fuel oil	Amount of C, wt.%			
	C≤4	**C5-C11**	**C12-C18**	**C≥19**
Pertalite	7.81	1.48	47.81	42.9
Pertamax	0	84.81	14.39	0.76
Diesel	0	3.35	73.68	22.96

RESULTS AND DISCUSSION

Effects of Temperature and Catalyst Content on Pyrolysis Yield Product

Figure 2a showed the yield products of bio−oil, gas, char, and water phase obtained from the pyrolysis process without using a catalyst for various temperatures between 300 and 700 °C. It might be expected that the more heat introduced into the system resulted in more hydrocarbon decomposition. Such a phenomenon was perhaps seen by the reduction of solid product (and bio-char) yield from 61.55 to 17.3 wt.% with increasing temperature, prior to stabilizing at 600 °C and above. Along with that, solid texture analysis exhibiting gradual changes from soft to hard texture was reported. This would mean decomposition initially occurred by releasing some hydrocarbon compounds, followed by gradual compositional changes, and finally reached bio-char stable composition [16-18]. From another perspective, this would mean that the remaining hydrocarbon was converted to gas and liquid products.

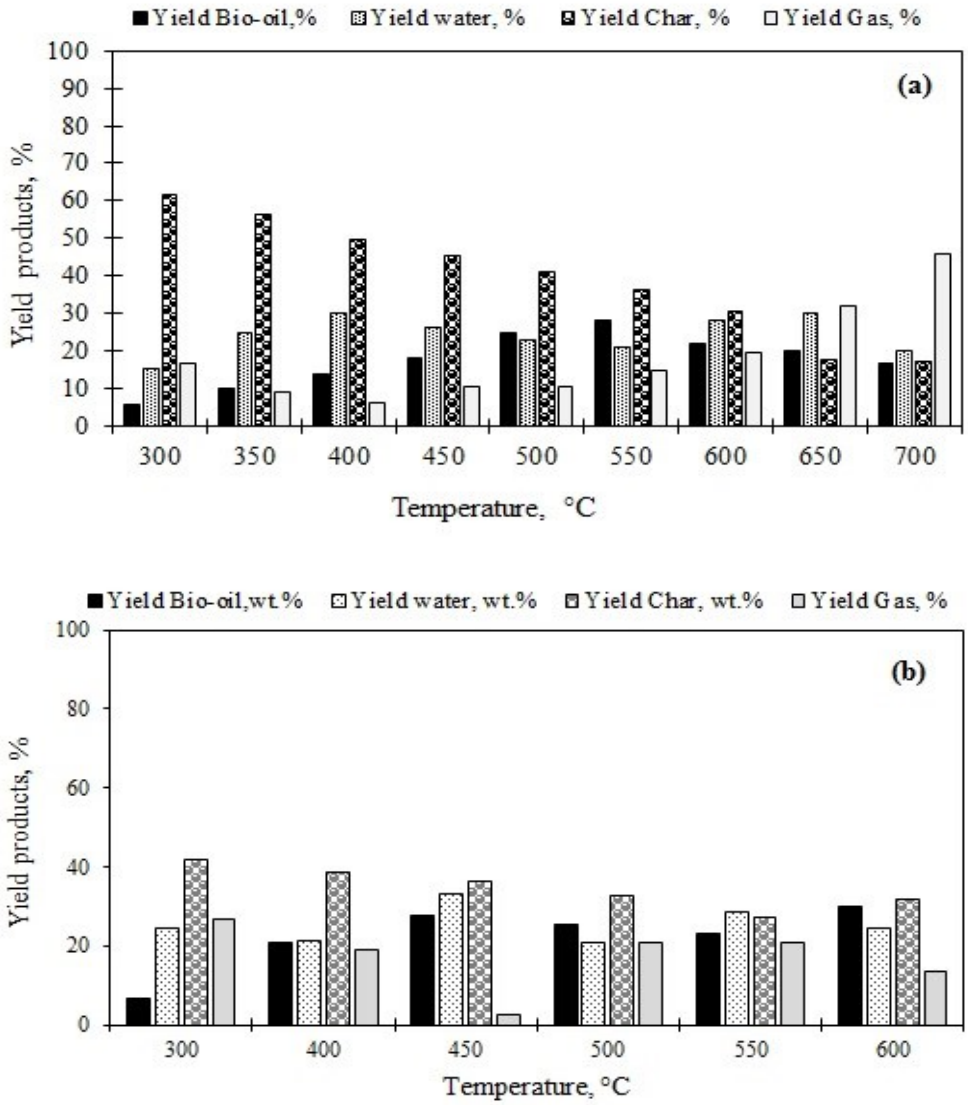

FIGURE. 2: Effect of temperature on yield products, obtained from SPR pyrolysis process a) without and b) with the presence of 5 wt.% Si-Al catalyst

The liquid products consisted of water, and bio-oil was initially collected at 300 °C. At this point, bio-oil presented the lowest yield of 6 wt.%. The trend of increasing bio-oil uptake was then observed, following their 50 °C incremental increase in processing temperature up to 550 °C. Interestingly, discontinuation on that trend, seen by sudden yield decrease, resulted in considering 550 °C as peak maximum temperature for SPR pyrolysis without a catalyst. Such a phenomenon was quite typical, as similarly reported elsewhere [24]. Above 600 oC, bio-oil yields tended to decrease due to the secondary cracking reaction that resulted in further decomposition into gas and char (24). Response to bio-oil secondary cracking might be confirmed by the inverted gas yield plot presenting a peak minimum at a temperature between 300 and 400 °C, followed by sharp yield increase to 45.7 wt.% at 700 °C.

For a comparative analysis, the yield result of pyrolysis at various temperatures in the presence of 5 wt.% Si-Al catalyst is presented in Fig. 2b. As observed, the trend of biochar and water yield remain fairly similar. However, no such peak maximum saw on bio-oil yield since it would rather show an increase of bio-oil with increasing temperature. Conversely, gas yield fluctuated, followed by a tendency to decrease at the later stage around 550 °C and above, in the presence of a catalyst.

Hence to further understand the influence of 5 % Si-Al catalyst, each yield in Fig. 2b was then compared against results shown in Fig. 2a for their corresponding temperature. It was evident that the catalyst was able to boost bio-oil uptake at a temperature between 300 and 450 °C, considered to be primary phase decomposition. Moreover, the more significant bio-oil yield was perceived for the pyrolysis process conducted at a temperature higher than 550 °C, taking into account that the relative data on the curve "without catalyst" lean toward decreasing trend as opposed to increasing trend on the one with catalyst.

As the focus was paid on maximizing bio-oil uptake, there might be a possibility that the amount of bio−oil would further increase with temperature. For that reason, the SPR pyrolysis with Si-Al catalyst was further experimented to study the effect of the amount % catalyst between 5 and 20 % at the highest temperature previously tested of 700 °C, where the results are shown in Fig. 3.

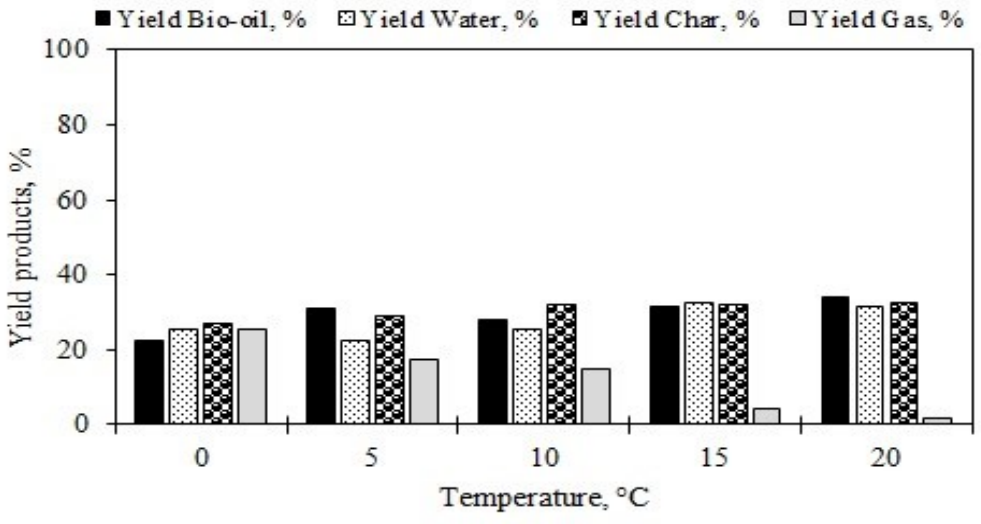

FIGURE. 3: The evolution of yield product percentage as a function of Si-Al catalyst percentage between 0 and 20 wt.% on SPR pyrolysis products at 700 °C

As seen, the result presented that yield gas decrease quite significantly from 17.47 to 1.70 wt.% with the increase of catalyst involved from 0 to 20 wt.%. Consequently, liquid products and bio-char seemed to increase. Liquid products, a total amount of Bio−oil and water, were increased by ±17.00 wt.% from 48.04 to 65.10 wt.% in which 12 wt.% increase was contributed from the raise of bio-oil uptake. In fact, the involvement of catalysts by only 5 wt.% has led to an increase of about 8%. A higher Si-Al catalyst percentage tested between 5 and 20 wt.% did not cause a significant rise despite its increasing trend. Regardless, its concurrent decrease in gas and bio-oil increase might suggest the occurrence of vapor condensation.

Fractional Composition (Based on C-Number Category Range) Of Bio−Oil Products as A Function of Temperature and Catalyst Content

Table 4 presented the fractional composition, referring to their number of C atoms (LPG, gasoline, diesel, and heavy naphtha) of bio-oil obtained by pyrolysis without catalyst at a temperature of 300, 400, 500 and 600 °C.

TABLE 4. Bio-oil composition referring to its C-atom category, obtained at various pyrolysis temperature between 300 and 600 °C, without catalyst

Pyrolysis temperature, °C	Amount of C, wt.%			
	C≤4 (LPG)	C5−C11 (Gasoline)	C12−C18 (Diesel)	C≥19 (*Heavy Naptha*)
300	16.60	64.50	9.73	0.09
400	0.66	52.14	25.97	21.26
500	−	15.32	49.53	35.12
600	−	15.66	49.92	36.94

At 300 °C, bio−oil consisted of LPG (C≤4), gasoline (C5−C11), diesel (C12−C18), and heavy naphtha (C≥19) in which their corresponding content was 16.60, 64.50, 9.73, and 0.09 wt.%, respectively. At 400 °C, LPG fraction started to disappear, showing only 0.66 wt.% collected. As expected, this fraction was not found at temperature 500−600 °C. In addition, gasoline fraction also exhibited a sharp decrease in weight percentage from 64.50 to 15.66 with the raise of tested temperature. Consequently, other fractions would rise, given the fact that diesel increased from 9.73 at 300 °C to 49.92 wt.% at 600 °C, besides the presence of 36.94 wt.% of heavy naphtha at 600 °C from almost none at 300 °C. By combining information summarized in Fig.F 2a and Table 4, bio-oil uptake could be obtained maximum at 550 °C, where its composition likely consisted of about 15, 50, and 35, respectively, for LPG, gasoline, and diesel. Interestingly, such composition seemed to present its potential for being used as future fuel due to its similarity on hydrocarbon content as compared to fuel derived from crude oil [17]. Table 3 shows that the GC-MS test results for pertalite petroleum fuels consist of C≤4, C5-C11, C12-C18, and C≥19, respectively, with the values of 7.81, 1.48, 47.81 and 42.90 wt.%. It can be concluded that the bio-oil produced by pyrolysis of the SPR at a temperature of 500-600 ºC is equivalent to pertalite.

Table 5 presented the result of LPG, Diesel, and Heavy naphtha fraction on bio-oil yield obtained from the pyrolysis process with Si-Al catalyst having content from 5 to 20 wt.%. In the presence of a catalyst, none of the heavy naphthas was produced regardless of the catalyst content to suggest the suppression of longer chain hydrocarbon production. In addition, diesel, having chain numbers between 12 and 18, exhibited a decrease as well. Thereof, gasoline was the major fraction produced, around 80 wt.%.

The influence of catalyst content on bio-oil fraction was rather inconclusive at this point. Statistically, gasoline and diesel fraction composition were around 80 and 20 wt.% Regardless of the catalyst content, except for the result when using 10 wt.% of Si-Al catalyst. This great amount of gasoline fraction agreed with the study reported by Sunarno [10] that the rise in pyrolysis temperature and the use of silica-alumina catalysts by impregnation of Ni metal will increase the gasoline, kerosene and phenol fractions, while, the oxygenate compound will decrease. In any case, the introduction of only 5 wt.% Si-Al catalyst was able to drive the pyrolysis process to lean toward the production of gasoline, which would be prospective to replace crude oil-based gasoline fuel. The existence of about 20 wt.% of the C12-C18 (diesel) fraction is still tolerated as a by-product in gasoline. Table 3 shows that the GC-MS test results for fuel from Pertamax petroleum consist of C≤4, C5-C11, C12-C18, and C≥19, respectively, with values of 0, 84.81, 14.39, and 0.76 %. It can be concluded that with the use of Si-Al catalysts: 5, 15, and 20 %, the bio-oil fraction consists of C5-C11 with an average of 82.84 % and C12-C18 with an average of 16.73 %, the resulting values lead to Pertamax.

TABLE 5. Component of bio-oil SPR based on the amount of C on various wt.% of the amount of Silica−alumina catalyst

Catalyst (%)	Amount of C, wt.%		
	C≤4 (LPG)	C5−C11 (Gasoline)	C12−C18 (Diesel)
5	-	80.06	19.34
10	-	41.48	57.06
15	-	86.56	13.13
20	-	81.89	17.73

CONCLUSION

This research aimed to study the influence temperature and catalyst content on the pyrolysis process of Spirulina Plantesis Residue (SPR) with and without the Silica-Alumina (Si-Al) catalyst. Pyrolysis process of SPR, for both without and with the catalyst, produced liquid product (bio−oil and water phase), gas, and char. On the basis of weight percentage, the optimum bio-oil uptake of 28 wt.% was obtained from pyrolysis at temperature 550 °C, considered as a peak maximum within the range of tested temperature (300-700 °C). At this temperature, the resulting bio−oil compositions were likely to consist of gasoline (C5−C11), diesel (C12−C18) and heavy naphtha (C≥19), considering similarity on such fraction obtained at 500 and 600 °C

On the other hand, the pyrolysis of SPR with catalyst presented the absence of such peak maximum, since bio-oil yield seemed to keep increasing with temperature. At 700 °C, the use of 20 wt.% Si-Al catalyst was able to produce 34.10 wt.% of bio-oil, where its fraction was mainly composed of gasoline leading to pertamax, whereas without a catalyst, it led to pertalite by referring the number of C atoms in bio-oil.

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