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Siti Jamilatun, Yeni Elisthatiana, Siti Nurhalizatul A³⁴ Ilham Mufandi, & Arif Budiman :
Effect of Temperature on Yield Product and Characteristics of Bio-oil From
Pyrolysis of *Spirulina platensis* Residue

EFFECT OF TEMPERATURE ON YIELD PRODUCT AND CHARACTERISTICS OF BIO-OIL FROM PYROLYSIS OF *Spirulina platensis* RESIDUE

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Abstract : Dependence on the use of fossil fuels in Indonesia is still quite high, especially crude oil; if no new energy reserves found, it will disrupt long-term energy availability. Biofuel is a renewable energy source derived from biomass, such as the type of microalgae *spirulina platensis* (SP). Solid residues from SP extraction still contained high levels of protein and carbohydrates. This solid residue can be processed by pyrolysis to produce bio-oil, water phase, charcoal, and gas. Bio-oil and gas products can use as fuel, charcoal can use for pharmaceutical needs, and the water phase as a chemical can use in food and health. The pyrolysis process carried out in a fixed-bed reactor with temperature ranging from 300-600°C. Heating was carried out by electricity through a nickel wire wrapped outside the reactor. Pyrolysis product in the form of gas condensed in the condenser, the condensate formed measured by weight. Char weight measured after the pyrolysis process completed. At the same time, non-condensable gas calculated by gravity from the initial weight difference of SPR minus liquid weight (bio-oil and water phase) and char. SPR samples were analyzed proximate and ultimate, while bio-oil products examined by the GC-MS method. The experimental results showed that the optimum pyrolysis temperature at 500°C produced by 18.45% of bio-oil, 20% of the water phase, 32.02 of charcoal, and 29.54% of gas by weight. GC-MS results from bio-oil consisted of ketones, aliphatics, nitrogen, alcohol, acids, while PAHs, phenols, and aromatics not found.

Keywords : Bio-oil, Pyrolysis, *Spirulina platensis*

Abstrak : Ketergantungan penggunaan bahan bakar fosil di Indonesia masih cukup tinggi terutama minyak mentah, jika tidak ditemukan cadangan energi baru maka akan mengganggu ketersediaan energi jangka panjang. Biofuel adalah salah satu sumber energi terbarukan yang berasal dari biomassa seperti jenis mikroalga *spirulina platensis* (SP). Residu padat dari ekstraksi SP masih mengandung protein dan karbohidrat yang cukup tinggi. Residu padat ini dapat diproses dengan pirolisis untuk menghasilkan bio-minyak, fase air, arang, dan gas. Produk bio-minyak dan gas dapat digunakan untuk

bahan bakar, arang dapat digunakan untuk kebutuhan farmasi, dan ⁷e air sebagai bahan kimia dapat digunakan di bidang makanan dan kesehatan. Proses pirolisis dilakukan dalam reaktor fixed-bed dengan suhu 300-600°C. Pemanasan dilakukan dengan listrik melalui kawat nikel yang dibungkus di luar reaktor. Produk pirolisis berupa gas dikondensasi dalam kondensor, kondensat yang terbentuk diukur beratnya. Berat char diukur setelah proses pirolisis selesai, sementara gas yang tidak dapat dikondensasi dihitung beratnya dari perbedaan bobot awal SPR dikurangi bobot cair (bio-oil dan fase air) dan char. Sampel SPR dianalisis proksimat dan ultimat, sedangkan produk bio-minyak dianalisis dengan metode GC-MS. Hasil percobaan menunjukkan bahwa suhu optimum pirolisis adalah 500°C yang menghasilkan bio-oil, water phase, arang, dan gas berturut-turut adalah 18,45; 20; 32,02 dan 29,54 % berat. Hasil GC-MS dari bio-oil terdiri dari keton, alifatik, nitrogen, alkohol dan asam, sedangkan PAH, fenol dan tidak ditemukan.

Kata kunci : Bio-oil, Pirolisis, *Spirulina platensis*

Introduction

Indonesia is the most significant energy consumption in Southeast Asia ¹⁸ ranks fifth in Pacific Asia after China, India, Japan, and South Korea. The final energy consumption in Indonesia in 2016 was dominated by fuel oil (BBM) around 47%. Based on the sector area, the transportation sector has the most significant consumption of around 42%. It was more than that the industrial sector around 36%. The energy consumption of the transportation sector almost entirely fulfilled by fossil fuel (BPPT-OEI, 2018).

The ¹⁴ increasing fossil energy needs will disrupt energy source, so it is necessary to get renewable energy in the form of biofuel from biomass (BPPT-OEI, 2018). Biomass is a renewable energy source abundant throughout the world that can produce heat and power as planned and can neutralize CO₂ (Bridgwater *et al.*, 1999). The types of biomass consist of wood, herbal plant, agricultural waste, aquatic, animal waste, and household waste (Jamilatun and Salamah, 2014; Vassilev *et al.*, 2010).

The advantages of biofuel from biomass are (i) available abundant and stables raw material from biomass, low sulfur, potential green energy source (Choi *et al.*, 2020; Jamilatun *et al.*, 2017). Biomass processing can be done in two ways: Biochemical (anaerobic digestion and fermentation) and Thermochemical (direct combustion, gasification, pyrolysis, and liquefaction). In general, the thermochemical conversion is faster than biological methods, and the process is relatively more straightforward (Basu, 2010). Pyrolysis is the Thermochemical conversion to convert biomass into non-condensable gas (CO₂, C²⁴H₂, and CH₄), condensable-gas (water phase dan bio-oil), and char product at a temperature around 300-600°C in the absence of oxygen. (Jamilatun *et al.*, 2010; Jamilatun *et al.*, 2014). According (Gronli, 1996) bio-oil has dark brown and has a heating value about half of the conventional fuel oil.

Currently, the conversion of renewable energy from biomass with the pyrolysis method is highly unusual to develop into bio-fuel. (Suttibak, 2012) has

reported that the biomass from cassava waster into bio-fuel. The development of renewable energy from sugarcane has investigated by (Treedet and Suntivarakorn, 2011; Mufandi *et al.*, 2020). Biomass from Napier grass also has experimented with (Treedet and Suntivarakorn, 2018; Mufandi *et al.*, 2019). However, there is one type of biomass that needs to be developed by researchers, namely biomass from microalgae as one of the alternative fuel oil sources in the future. The development of microalgae as a renewable energy source is urgent to research because microalgae have many advantages compared to other energy sources such as high growth rate, not competing with a food source, and without requiring large areas to grow (Gultom *et al.*, 2014).

One of the benefits of microalgae is to produce more oil about 136.900 L/hectare for microalgae of 70% in biomass. Its value is equivalent to 74 times more than *Jatropha* oil and 23 times more than palm oil. Besides, microalgae planting land not require extensive area. In America, around 50% of all transportation fuel required 2 Ha of microalgae land, 140 Ha of *jatropha* land, and 45 Ha of palm oil land (Scheck *et al.*, 2008). The comparison of oil yield and area needed in each type of biomass that can see in table 1.

Table 1. The comparison of oil yield and area for each kind of biomass (Scheck *et al.*, 2008).

Plant	Oil Yield (L/Ha)	Area needed (Ha) ^a	% Area of land harvest in AS
Corn	172	1540	846
Soybean	446	594	326
Canola	1190	223	122
<i>Jatropha</i>	1892	140	77
Coconut	2689	99	54
Plam	5950	45	24
Microalgae	136.900	2	1.1
Microalgae	58.700	4.5	2.5

¹ fulfill 50% for all transportation fuel in AS.

^b70% oil (by wt) in biomass.

^c30% oil (by wt) in biomass.

According to (Chen *et al.*, 2018; Miao *et al.*, 2004), the bio-oil from microalgae has high heat about 1.4 times of biomass from wood, low viscosity, and low density. The biomass from microalgae is more satisfying as fuel than lignocellulosic material.

The composition of the bio-oil varies according to process conditions and raw material. The bio-oil content consists of water of 15-35 wt.% and hundreds of compounds. The bio-oil compounds as follows nitrogen (amides, amines, pyrroles, indole, pyridine, and its derivatives), oxygenated compounds (acid, alcohol, ketone, aldehyde, phenol, ether, ester, sugar, furan) and hydrocarbons (alkenes, benzene, toluene dan xylene) and solid particles (Hu and Gholizadeh, 2019; Yang

et al., 2019). Water content in bio-oil depends on the water content in the raw material of biomass.

Bio-oil from the pyrolysis of microalgae and biomass with high protein content is generally more stable, higher High Heating Value (HHV), and lower Oxygen content (O) than bio-oil from lignocellulose. Hydrocarbon linear in the bio-oil content has produced from pyrolysis lipid, aromatic hydrocarbon obtained from protein pyrolysis, and glucose obtained from carbohydrate pyrolysis (Hu *et al.*, 2013; Kan *et al.*, 2016). The composition of bio-oil included carbohydrate derivatives, protein, and lipids. Every type of microalgae has different structures, two kinds of compounds, and weight percent. The application of the use of bio-oil consists of fuel, electric generation for the factory, chemical material for various chemical industry needs (Jamilatun and Salamah, 2015; Jamilatun *et al.*, 2016; Jamilatun and Salamah, 2017; Jamilatun *et al.*, 2019).

From the above explanation, the biomass from microalgae is highly potential to convert into renewable fuels. In this experiment, the biomass from microalgae was conducted in the pyrolysis process in a fixed-bed reactor with temperature ranging from 300 to 600°C. The pyrolysis process in experiment has produced gas, liquid (water phase and bio-oil), and char. The bio-oil product was tested by GC-MS to identify the bio-oil compound such as oxygenated, nitrogenated, aliphatic, aromatic, monocyclic aromatic hydrocarbon (MAH), and polycyclic aromatic hydrocarbon (PAH).

Experimental Materials, Devices, and Methods

Materials

Dry *Spirulina platensis* residue (SPR) used in this experiment, it was obtained from *Spirulina platensis* (SP) solid residue extraction. SPR was dried under the sun to reduce the water content. SPR samples were analyzed to obtain ultimate, proximate, and High Heating Value (HHV) that can see in table 2. The analysis of SPR samples conducted at the Food and Agriculture Product Laboratory, Department of Agriculture Technology, UGM.

Devices

Dry *Spirulina platensis* residue (SPR) was pyrolyzed in the fixed bed reactor with an inner diameter of 40 mm, an outer diameter of 44 mm, and a height of 600 mm. The reactor made from stainless steel equipped with heater in the around of reactor (Jamilatun *et al.*, 2017; Jamilatun *et al.*, 2019). The detail of the pyrolysis system shown in figure 1.

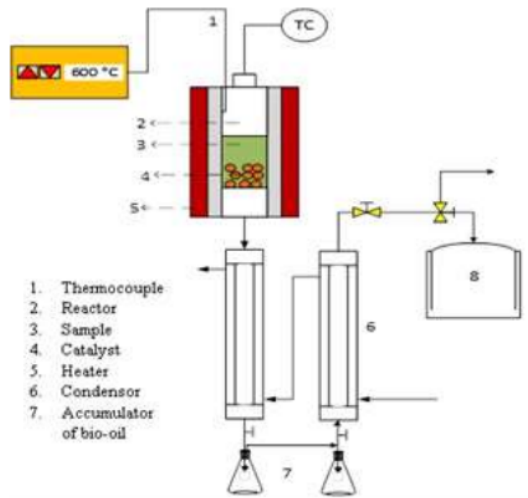


Figure 1. The SPR pyrolysis equipment (Jamilatun *et al.*, 2019)

Methods

Fifty grams of SPR was fed, tightly closed, and heated. SPR samples heated in the fixed bed reactor with a constant heating rate from room temperature to the desired temperature (300, 429 500, 550, 600 °C). Then the temperature was held constant. The temperature-controlled by the thermocouple placed inside of the reactor. The hot gas was condensed. The liquid product comes out and collected in the accumulator. The gas product obtained in the storage gas. After the experiment finished, the solid product/char was taken and weighed. The yield of bio-oil, gas, and char calculated by equation (Jamilatun *et al.*, 2017):

$$Y_L = (W_L/W_M) \times 100 \% ; Y_{Ai} = (W_{Ai}/W_M) \times 100 \% \dots (1)$$

$$Y_A = (W_A/W_M) \times 100 \% = Y_L - Y_{Ai} \dots \dots \dots (2)$$

$$Y_C = (W_C/W_M) \times 100 \% ; Y_G = 1 - (Y_L + Y_C) \dots \dots \dots (3)$$

In this case, Y_L is liquid product yield; W_L is the weight of the liquid product, W_M is sample weight, Y_A is water phase yield, W_A is the weight of the water phase, Y_{Ai} is bio-oil yield, W_{Ai} is the weight of bio-oil, Y_C is char yield, Y_G is gas product yield. GC-MS analyzed Bio-oil properties.

Results and Discussions

Spirulina Platensis Residue (SPR) Characteristic

Proximate analysis, ultimate analysis, HHV were used to know the SPR characteristic. It can display in table 2.

Table 2. *Spirulina platensis* residue (SPR) characteristic (Jamilatun *et al.*, 2017)

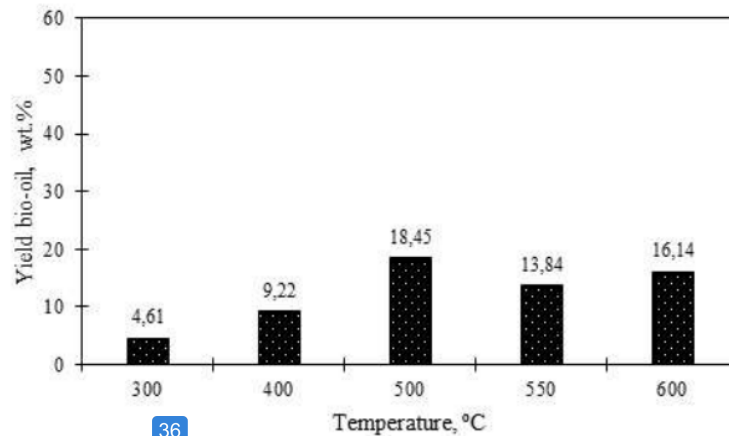
Component	SPR
<i>Composition analysis (wt.%)</i>	
Lipid	0,09
Carbohydrate	38,51
Protein	49,60
<i>Ultimate analysis (wt.%)</i>	
Sulfur	0,55
Carbon	41,36
Hydrogen	6,60
Nitrogen	7,17
Oxygen	35,33
Higher heating value (MJ/kg)	18,21

From table 2 it shows the proximate and ultimate analysis, the results have shown that the SPR properties were 49.6 wt.% of protein, 38.51 wt.% of carbohydrate, and 0.09 wt.% of lipid. In the utilization of microalgae residue, this analysis conducted to measure that the SPR samples have shallow lipid content. (Jamilatun *et al.*, 2017; Jamilatun *et al.*, 2019).

The Influence of Temperature on Bio-oil Yield

The impact of temperature on bio-oil yield shown in figure 2. From these figures, and it shows that the maximum of bio-oil yield at an optimum temperature of 500°C was 18.45 wt.%. This experiment found that the bio-oil yield was increased from 4.61 wt.% to 18.45 wt.% when the temperature is ranging from 300 to 500°C. While the temperature was decreased from 18.45 wt.% to 16.14 wt.% at the temperature under 500°C. The decrease of bio-oil yield was affected by secondary cracking occurs in tar (bio-oil and water phase). Secondary cracking is tar decomposition into gas and char. So it can be said that based on the highest amount of bio-oil, the temperature of 500°C is the optimum temperature for pyrolysis without a catalyst. The influence of temperature on SPR decomposition was very significant. Other forces are biomass type, heating rate, the residence time in the reactor, and reactor type (Yang *et al.*, 2019).

On the pyrolysis process occurs the cracking reaction, namely the breaking of the C-C bonding from a long carbon chain (polymer) and massive molecular weight into a short carbon chain (monomer) with a small molecular weight. This case can affect increasing pyrolysis temperature; the more bonds (hydrocarbon chains) broken, so yield increased. High temperature also affects the reduction of liquid products and consistent with its top gas products. It occurs the secondary cracking process, which breaks long chains of organic compounds and hydrocarbons into shorter chains so that they cannot be condensed again (Jamilatun *et al.*, 2019).

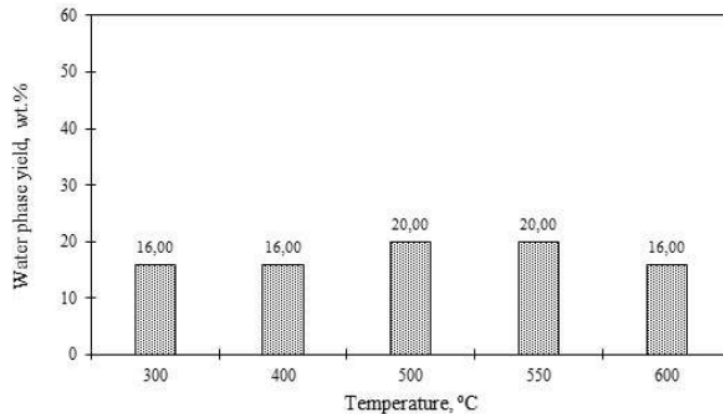


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Figure 2. The effect temperature on yield bio-oil

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The Influence of Temperature on Water Phase Yield

Figure 3 shows the influence of temperature on the water phase. From these experiments, the water phase yield has increased, which is affected by the temperature increase from 300-400 °C. Then the water phase yield was relatively constant at 400-550°C and dropped slightly at a temperature of 550°C. At a temperature of 600, the water phase yield has decreased. The water phase yield influenced by the water content of SPR (9.99 wt.% of dry based) and the reactions of water formation during pyrolysis proses (dehydration). Based on (Basu, 2010), the average water content of tar in biomass is above 20 wt.%.



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Figure 3. The influence of pyrolysis temperature on water phase yield

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The Influence of Temperature on Char Yield

Figure 4 shows the influence of temperature on char yield from SPR pyrolysis. Form these experiments; the char yield obtained from the temperature of 300°C, 400 °C, 500 °C, 550 °C and 600 °C with the percentage of 49.02 wt.%,

39.96 wt.%, 32,02 wt.%, 30.11 wt.%, and 28.39 wt.%, respectively. These results indicated that the char yield has significantly decreased. Based on the literature review (Dickerson T, 2013) shown that the pyrolysis at low temperatures less than 400 ° C or relatively low heating will produce relatively high char products. A lower heating rate and a longer the residence time causing a secondary cracking reaction will affect the bio-oil properties. The higher of the pyrolysis temperature used so that the char content will be lower because the SPR complier content will decompose, and the volatile matter content will decrease with the increase of the pyrolysis temperature.

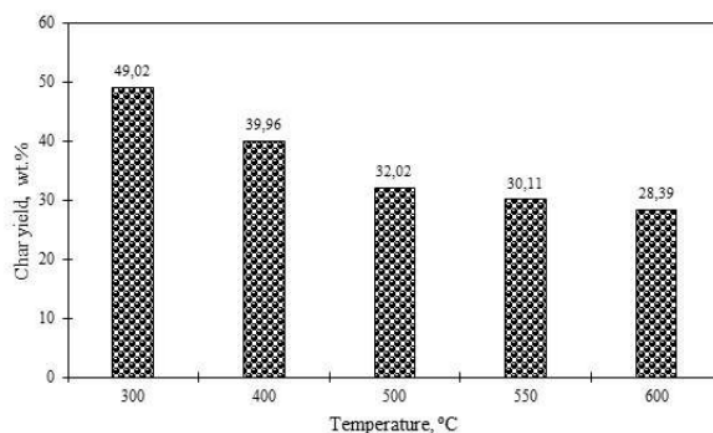
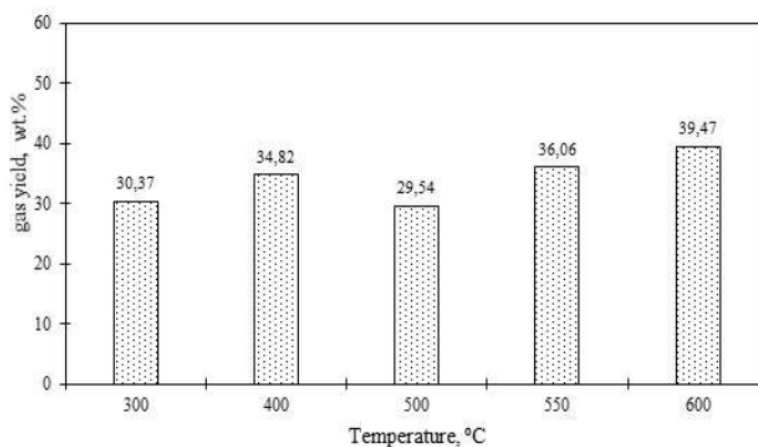


Figure 4. The influence of temperature on char yield

The Influence of Temperature on Gas Yield

Figure 5 shows the influence of temperature on gas yield. From these experiments, the gas yield obtained from the temperature of 300 °C, 400 °C, 500 °C, 550 °C, 600 °C with the percentage of 30.37 wt.%, 34.8 wt.%, 29.54 wt.%, 36.06 wt.%, and 39.47 wt.%, respectively. The experiment found that the temperature of 500 °C obtained the lowest yield among others because the SPR conversion at 500 °C of temperature was more bio-oil than converted to gas. The gas yield at a temperature of 550 °C and 600 °C has increased with the percentage of 36.06 wt.%, and 39.47 wt.%, respectively. So it can be seen that gas products are growing with increasing pyrolysis temperature.



2 **Figure 5.** The influence of temperature on gas yield

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The Influence of Temperature on Bio-oil Composition

The bio-oil tested by GCMS that can see in figure 6. From the testing, it found that the forms of bio-oil consist of nitrogen, alcohol, ketone, aldehyde, carbonyl, poly-aromatic, aliphatic, acid, phenol, aromatic—the bio-oil compositions displayed in figure 7.

In this experiment, the hydrocarbon compounds in bio-oil are aromatic and aliphatic hydrocarbon. The aromatic hydrocarbon can increase the octane value of the fuel, while high aliphatic hydrocarbons can apply as transportation fuels (Li G *et al.*, 2013). The aliphatic hydrocarbon includes alkane (single bond), alkene (double bond), cycloalkane, and cycloalkene that resulted from lipid pyrolysis at SPR. Aromatic hydrocarbon consists of monocyclic aromatic hydrocarbon (MAH) and polycyclic aromatic hydrocarbon (PAH). PAH compound in the bio-oil is quite high at 500°C (Zhou H *et al.*, 2015). PAH can form when products such as coal, oil, gas, and organic matter not burned out completely. Aromatic compounds not identified in this study.

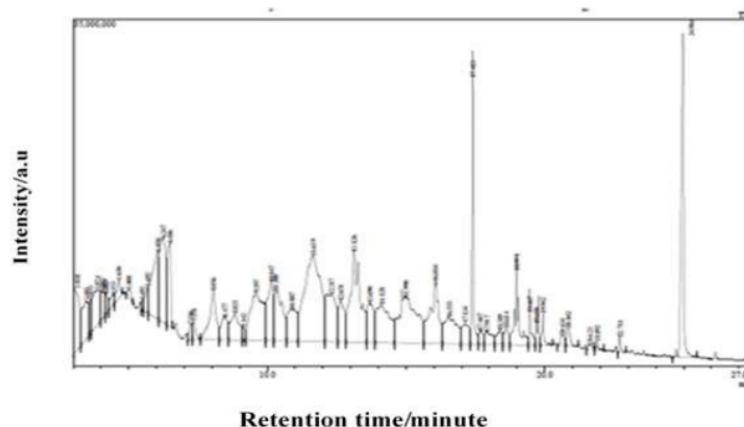


Figure 6. Spectra GC-MS bio-oil from SPR pyrolysis

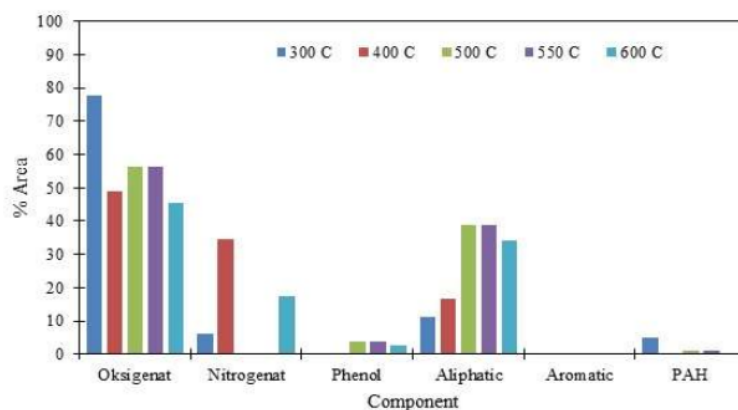


Figure 7. The influence of temperature on bio-oil compositions

Conclusions

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 6 Biofuel is one of the renewable energy sources that obtained from biomass. *Spirulina platensis* residue (SPR) obtained from *Spirulina platensis* (SP) solid residue extraction. It heated in the fixed-bed reactor with temperature ranging from 300-600°C to produce three products, such 10 gas, char, and liquid (water phase and bio-oil). This experiment found that the maximum bio-oil yield was 18.45 wt.% at a pyro 25 is temperature of 500°C. The maximum water phase yield obtained about 20 wt.% at the pyrolysis temperature from 500-550°C. The maximum char yield obtained at temperature 300°C about 49.02 wt.%. The gas yield was obtained 35.47 wt.% at a temperature of 600°C. However, 35 pyrolysis temperature had effected the pyrolysis product. GC-MS tested the bio-oil yield. From the analysis result, the compositions of the bio-oil consist of nitrogen, alcohol, ketone, aldehyde, carbonyl, poly-aromatic, aliphatic, acid, phenol, and aromatic.

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