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Catalytic Hydrothermal Liquefaction of Botryococcus braunii: Enhancing Bio-Crude Oil Yield and Quality

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Abstract (Verdana 8 font)

Abstract. Kemajuan teknologi konversi biomassa kini telah mencapai generasi ketiga, yang menawarkan keuntungan karena tidak bersaing dengan pasokan pangan. Salah satu sumber biomassa generasi ketiga yang paling menjanjikan adalah mikroalga. Salah satu metode efektif untuk mengubah mikroalga menjadi bio-oil adalah Katalitik Hydrothermal Liquefaction (katalitik HTL). Teknik ini meningkatkan rendemen dan kualitas bio-oil, sehingga berpotensi membuat sifat-sifatnya sebanding dengan minyak mentah. Penelitian ini menyelidiki pengaruh penambahan katalis dalam proses Katalitik HTL terhadap karakteristik bio-crude oil yang dihasilkan. Percobaan dilakukan menggunakan reaktor batch dengan katalis silika-alumina, pada berbagai suhu (200, 225, dan 250)°C dan beban katalis (4%, 5%, dan 6%). Proses catalic HTL menghasilkan empat fase produk yang berbeda: bio-oil, fase air, gas, dan residu padat. Analisis menggunakan Kromatografi Gas-Spektrometri Massa (GC-MS) menunjukkan bahwa kandungan hidrokarbon tertinggi, hampir 40%, diperoleh dengan penambahan katalis 6%. Selain itu, peningkatan penambahan katalis terbukti meningkatkan kualitas bio-oil.

Keywords: Biomassa; Bio-crude Oil; Hydrothermal Liquefaction

Abstrak (Verdana 8 font)

Abstract. Advances in biomass conversion technology have now reached the third generation, which offers the benefit of not competing with food supply. Among the most promising third-generation biomass sources is microalgae. One effective method for converting microalgae into bio-oil is catalytic hydrothermal liquefaction (HTL). This technique enhances both the yield and quality of bio-oil, potentially making its properties comparable to those of crude oil. The present study investigates the influence of catalyst addition in the HTL process on the characteristics of the resulting bio-crude oil. Experiments were conducted using a batch reactor with a silica-alumina catalyst, at with varying temperatures (200, 225, and 250)°C and catalyst loading (4%, 5%, and 6%). The catalytic HTL process produced four distinct product phases: bio-oil, aqueous phase, gas, and solid residue. Analysis using Gas Chromatography-Mass Spectrometry (GC-MS) revealed that the highest hydrocarbon content nearly 40% was obtained with the addition of 6% catalyst. Additionally, increasing catalyst loading was found to improve bio-oil quality.

Keywords: Bio-oil; Hydrothermal; Catalytic; Microalgae;

1. Introduction

Indonesia is One of the countries with the most islands, with a population of more than 268 million people in 2019, and it is projected to reach 318.9 million by 2045 (BPS, 2018). This increase is due to the high population growth in Indonesia. Consequently, this growth also leads to an increase in the country's energy demand, which is projected to reach around 1.146 billion Standard Barrels of Oil Equivalent (SBOE) by 2025. Therefore, to ensure national energy security, it is essential to develop alternative renewable fuels that can be produced domestically—one of

which is biofuel. Solids, liquids and gases can also be phases of biofuel which derived from or composed of alternative biomass resources. Biofuels are generally classified into three types: bio-crude oil, bio-gas, and bio-char. First-generation biofuels are derived from food-based biomass sources such as corn, peanuts, etc., to produce liquid fuels like ethanol and biodiesel. However, these compete directly with food supply. 2nd-generation biofuels use non-food biomass feedstocks such as lignocellulosic biomass, yielding the same products ethanol and biodiesel without competing with food resources.

The third generation of biofuels still uses non-food feedstocks such as fungi, microalgae, and other materials that have the potential to replace fossil fuels (Garcia Alba et al., 2013). 3rd-generation biofuels especially those derived from microalgae are considered the most efficient alternative. According to Clarens et al., (2010) Biomass that has high potential in producing biofuel and has the ability to grow rapidly is microalgae, and microalgae has ability faster growth rate, and relatively high biomass productivity, even on relatively small land areas. Additionally, Microalgae can grow in saline or brackish media and artificial media, avoiding competition with conventional crop cultivation.

There are many thermochemical processes available for converting biomass to fuel, one of which is hydrothermal liquefaction (HTL). This process can be conducted either with or without a catalyst. A review by (D. Xu et al., 2018) concluded that the addition of a catalyst in HTL processes can decrease the N₂, S₂, and O₂ percentage in the resulting bio-oil, while also enabling lower reaction temperatures and shorter reaction times. Typically, HTL of microalgae yields around 40% bio-crude oil on a dry basis, but this can increase to 50–60% with the addition of a catalyst (Y. Xu et al., 2015).

(Basu, 2013) explained that there are several types of thermochemical processes, such as liquefaction, pyrolysis, gasification, and combustion. Each of these thermochemical processes operates under specific conditions, as outlined in Table 1. From Table 1, it is shown that the use of a catalyst in the liquefaction process is essential. The impact of catalyst usage in liquefaction also varies depending on the type of catalyst applied. Therefore, the following section presents the influence of catalysts as reported by previous researchers.

Table 1. Comparison of Various Thermochemical Conversion Methods

Process	Temperature, °C	Pressure, MPa	catalyst	Drying
Liquefaction	250 - 330	5 - 20	Necessary	No needed
Pyrolysis	300 - 600	0,1 - 0,5	Not Necessary	Needed
Combustion	700 - 1400	≥ 0,1	Not Necessary	No needed
gasification	500 - 1300	≥ 0,1	Not Necessary	Needed

There are three types of catalysts used in the HTL process: homogeneous catalysts, heterogeneous catalysts, and gas phase catalysts. Homogeneous catalysts commonly used in the HTL of microalgae are those that are water-soluble at room temperature, such as alkaline salts (e.g., Na₂CO₃ and KOH) and organic acids. Catalysts have a positive impact on the HTL process by increasing the yield and reducing the amount of solid residue (Y. Xu et al., 2015).

One of the thermochemical processes that can be used to produce bio-crude oil from biomass is hydrothermal liquefaction (HTL). In hydrothermal liquefaction, water in subcritical and supercritical states comes into contact with the biomass. As shown in Figure 1, the use of water in the HTL process is carried out within a temperature range of 200–370°C and a pressure range of 40–200 bar. It is also observed that the operational conditions for HTL are divided into three regions: liquefaction, catalytic gasification, and high-temperature gasification (Peterson et al., 2008). Within this operational range, the HTL process is significantly influenced by the altered physicochemical properties of water.

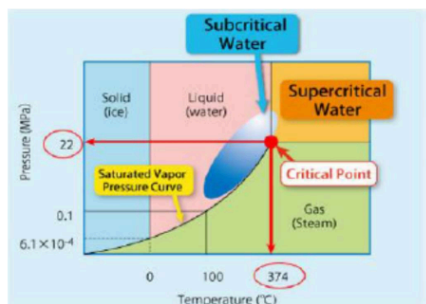


Figure 1. Range of operating conditions of the hydrothermal liquefaction process (Thiruvankadam et al., 2015)

Considering the catalyst impacts and the relatively lower cost, silica-alumina was selected as the catalyst in this study. The aim of this research is to investigate the impact of silica-alumina catalyst on the yield and quality of bio-crude oil produced through the hydrothermal liquefaction process.

2. Methodology

2.1. Materials

Microalgae flour with *Bortryococcus brauni* species which obtained from BPBAP (Balai Perikanan dan Budidaya Air Payau) Situbondo, East Java. Nitrogen Gas (N_2) which obtained from PT. Samator Gas Industri, Yogyakarta. Distilled Water, N-Hexane with 99,8% rate which produced by Merck. Silica-Alumina Catalyst which obtained from PT. Pertamina Balongan, Indonesia.

2.2. Instrumentation

In this study, the hydrothermal liquefaction (HTL) process will be carried out using a series of equipment made of stainless steel, designed to accommodate the high-pressure requirements of HTL process. The main component of this setup is a cylindrical hydrothermal liquefaction reactor with a volume of 1 L. The reactor is encased in a stainless steel layer approximately 2 cm thick and is equipped with an electric heater that operates at heating rate of approximately $10.5^\circ\text{C}/\text{min}$. It is also fitted with a magnetic stirrer for mixing. The detailed specifications of the reactor are presented in.

Table 2. Hydrothermal Liquefaction Reactor Specification

Parameter	Specification
Material	Stainless steel
Wall Thickness	4 cm
Maximum Pressure	6000 psi (± 400 atm)
Maximum Temperature	600°F ($\pm 315^\circ\text{C}$)

2.3. Procedure

In this study, the manipulated or independent variables were as follows:

1. Catalyst loading of 4%, 5%, and 6% (w/w)
2. Process temperature: 200°C , 225°C , and 250°C
3. Residence time: 10, 20, 30, 40, and 50 minutes

2.3.1 Preparation of Silica-Alumina Catalyst

Pertamina Balongan, Indonesia. Combine the Silica-Alumina flour with 5% of gypsum and water. Then form it into cylinder with 0,5 mm diameter using press tool. Dried the catalyst in the

oven with 110°C within 2 hours and calcinated in 500°C inside N₂ air within 2 hours for activation. Restored the catalyst in the desiccator before use.

2.3.2 Liquefaction Hydrothermal Process

15 gram of dry *Botryococcus braunii* Microalgae combined with 150 mL of Distiled Water. Fed the mixture inside HTL reactor (Number 5). Flow N₂ Gas to the reactor tube until the pressure gauge indicated 40 bar pressure. Then, heating of the reaction begins until the reaction time is reached.

2.3.3 The extraction of bio-crude oil with n-hexane

All the products which have been taken (solid residue, aqueous phase and bio-crude oil) were combined with n-hexane. In this research n-hexane with 1:1 ratio used inside the breaker glass. The process was assisted with stirring using magnetic stirrer and 40°C temperature, within 1 hour. From this process, bio-crude oil will be extracted inside the n-hexane.

2.3.4 Separation and distillation of bio-crude oil

After the separation process conducted, n-hexane which on the top of the mixture, separated from the mixture using volume pipette. Organic phase mixture (bio-crude oil and n-hexane) inserted in the distillation circuit. In this process, the 3neck flask will be heated until the n-hexane evaporated and the dew was accommodated to be re-use. The left bio-crude oil (brownish black and thick) moved into stored bottle.

2.3.5 Separation of aqueous phase and solid residue

After the organic phase separated, the next step was the separation of aqueous phase and solid residue. Both products separated through filtration process using filter paper assisted with vacuum pump. The left solid residue in the paper then inserted into the oven with 105°C for drying to constant weigh.

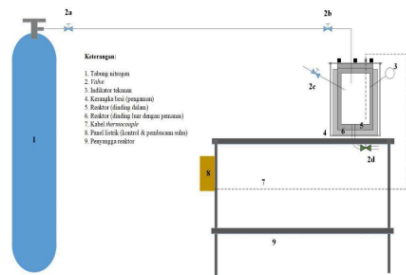


Figure 2. Schematic Circuit of Hydrothermal Process

3. Results and Discussion

3.1. The Product of Catalytic Hydrothermal Liquefaction

The products of HTL catalytic which use *Botryococcus braunii* Microalgae and Autoclave sized 1L tool can be seen in Figure 2. The using of small size HTL reactor is common, while the using of large size still relatively rare. Some research use reactor with 300 L: (Cheng et al., 2014); and (Yan et al., 2018); also (Zhang et al., 2017) are able to produced bio-crude oil in the amount of 30-70%. The amount of bio-crude oil in this research is only 1,278%. The composition of *Botryococcus braunii* Microalgae which has the low lipid content and the dust content which reached 50% are the cause of low amount of bio-crude oil. Later on as shown in Table 3.

Table 3. Functional Group Analysis Results of *Botryococcus braunii*

Parameter	Content (%)
Protein	6,73

Lipid	1,82
Water	10,60
Ash	51,18
carbohydrate	28,84

The high ash content, which reached up to 51%, may also be attributed to a lack of nutrients provided during the microalgae growth phase. Extreme environmental conditions can lead to a decrease in the lipid content of microalgae. Several products are generated during the catalytic Hydrothermal Liquefaction (HTL) process using *Botryococcus braunii* as the feedstock, under operating conditions of approximately 200–250°C and pressures ranging from 40 to 50 bar. The resulting products, as shown in Figure 3, include biocrude oil (bio-crude), an aqueous phase, and a solid phase. Each product has its own distinct characteristics. The high ash content in the microalgae biomass also contributes to the low yield of bio-oil. In addition, with 15 grams of microalgae fed into the reactor and an ash content of about 50%, only around 7 grams of carbohydrates, proteins, and lipids remain, with the lipid percentage being just 1.82%, equivalent to 0.27 grams. Yet, the largest contribution to bio-oil yield comes from the lipid content. This can be seen in Figure 5, which shows three production pathways from carbohydrates, lipids, and proteins with the production of esters or methyl esters coming from the lipid pathway.

The biocrude oil contains a high concentration of long-chain hydrocarbons as well as other long-chain compounds ranging from C₁₂ to C₃₀. The aqueous phase consists of polar compounds such as carboxylic acids, esters, ketones, and other water-soluble substances under standard conditions. Lastly, the solid phase mainly comprises unreacted microalgae and other impurities. The product of bio-crude oil from Hydrothermal Liquefaction (HTL) will be GC-MS analysis to see the comparison in various catalyst loading in order to see the impact of the catalyst loading amount on the quality of resulted bio-crude oil. The result of the analysis can be seen in Figure 4.



Figure 3. Catalytic HTL Products, (a) Bio-Crude Oil (b) Aqueous Phase (c) Solid

Solid product or residue of microalgae which didn't react will be tested further, it is because the result of bio-crude oil yield product can be related to the morphology from microalgae cell. The product of bio-crude oil has few barrier factors, thus allowing the low product of the yield. First, the barrier of the cell has been destroyed. Second, the compound from the destroyed barrier will be degraded to become oil (Garcia Alba et al., 2012). Therefore, morphology analysis of microalgae using SEM analysis was needed. Figure 4 shown the result microalgae solid analysis before reaction, also at 200°C & 250°C. Based on the figure 4, it can be seen the surface of the cell before the reaction of barrier of the cell was already destroyed and when the temperature was further increased, the surface result is smoother. It means, the first probability mentioned above can be discard. Therefore leaving the second probability which is the degradation of cell becoming oil. As explained before that microalgae solid will be degraded or hydrolysis to become monomers depend on reaction temperature.

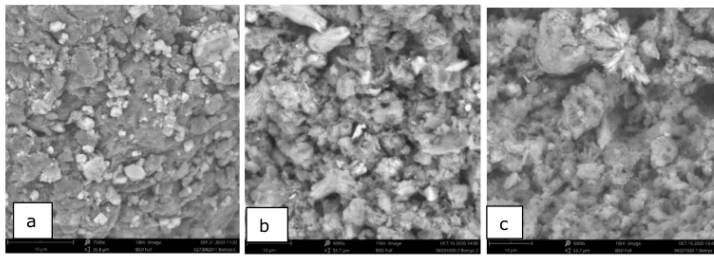


Figure 4. The result of SEM test Microalgae *Botryococcus braunii* (a) cell barrier before Hydrothermal Liquefaction Process (Dayananda et al., 2007) (b) when reaction temperature 200°C (c) when reaction temperature 250°C

Hydrothermal liquefaction is a continuous process involving extraction, hydrolysis, and a series of other reactions that occur with the aid of water alone. As shown in Figures 3b and 4c. The exterior of *Botryococcus braunii* microalgae experiences degradation, resulting in the release of organic macromolecules like lipids, proteins, and carbohydrates into the surrounding water. In this process, water must function as both a polar and non-polar solvent.

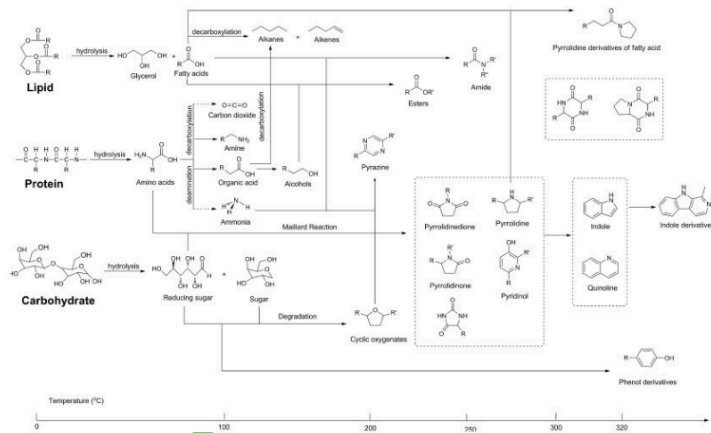


Figure 5. The reaction pathway of the hydrothermal liquefaction process (Gai et al., 2015)

According to (Yang et al., 2020) such conditions can occur under subcritical water conditions, in which water exhibits higher concentrations of H^+ and OH^- ions than under normal conditions. This enhances its hydrolytic capability and its ability to dissolve certain non-polar compounds.

3.2. The Impact of Adding Catalyst on Bio-Crude Oil Product

Bio-crude oil product which taken from Hydrothermal Liquefaction process was analysis using GC-MS to know the components contained inside. After that, it will be grouped based on functional group from each component to see the most functional group. It can be seen in Figure 5. In the research about catalytic HTL and Microalgae *D. tetiolecta* explain that the first reaction from zeolite catalyst is reaction of ketones, decarboxylation, dehydration, and ammonolysis. Based on the mechanism served by (Gai et al., 2015) decarboxylation reaction is able to produce hydrocarbon compound. In this case, it is proven that the selectivity of zeolite catalyst direct to decarboxylation reaction which caused higher catalyst loading, higher the hydrocarbon rate of bio-crude oil.

Figure 6 also illustrates the influence of catalyst loading on the composition of bio-oil. As the catalyst loading increases, it reduces the presence of other compounds such as amides, oxygenates, and N&O heterocyclic components. This indicates that the quality of the bio-oil improves, making its composition more similar to that of crude oil. These findings also support the hypothesis that the addition of silica-alumina catalyst can enhance the hydrocarbon content of the bio-oil.

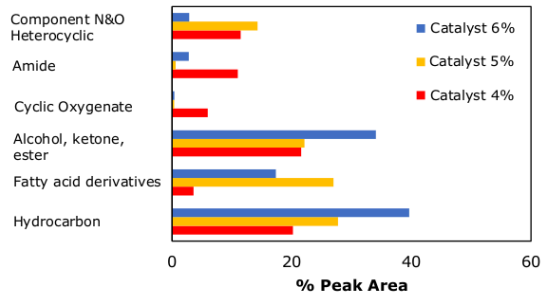


Figure 6. The result of GC-MS analysis of bio-crude oil composition on various catalyst loading at 250°C and 30 minutes residence time

3.3. The Impact of Adding Catalyst on Aqueous phase

Aqueous phase product can be seen in Figure (b) has dark reddish brown. This product formed because of added water change to more non-polar then become solvent to non-polar components inside microalgae. After that, aqueous phase product in this research also analysis using GC-MS for each various catalyst, but the result of GC-MS from aqueous phase shown constant noise without dominant peak on the graphic result. Therefore, it classified on functional group for the ease identification. GC-MS result on aqueous phase shown in Figure 7. only at optimum condition at 250°C of 5% catalyst loading. It probably caused by the component which contained in aqueous phase has bigger molecule weigh so that difficult to evaporate and resulted the GC-MS tool unable to read the component accurately.

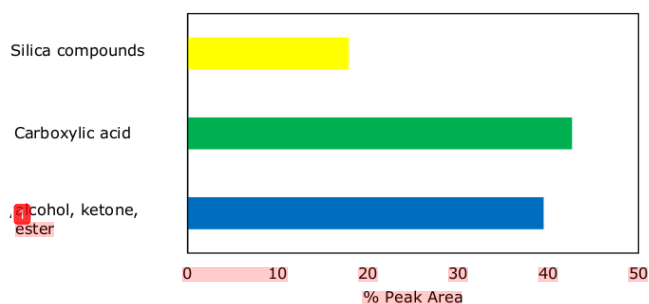


Figure 7. The result of GC-MS analysis of Aqueous phase composition on various catalyst loading at 250°C and 30 minutes residence time

4. CONCLUSION

The optimum condition that produced the highest quality bio-oil was achieved with the addition of 4% catalyst. This is attributed to the hydrocarbon content, which reached nearly 40% based on GC-MS analysis. This indicates that increasing the amount of catalyst enhances the quality of the resulting bio-oil. In addition, under this condition, the aqueous phase product also contained the highest concentration of carboxylic acids.

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