

## Characteristics of Liquid Products from *Spirulina platensis* Pyrolysis under Microwave Irradiation with Activated Carbon Additive

Mohammad Mirza Yuniar Romaz<sup>1</sup>, Sukarni Sukarni<sup>1,2,\*</sup>, Retno Wulandari<sup>1</sup>

<sup>1</sup>Center for Renewable Fuels Research (CRFR), Department of Mechanical and Industrial Engineering, Universitas Negeri Malang, Indonesia

<sup>2</sup>Center of Advanced Materials for Renewable Energy (CAMRY), Universitas Negeri Malang, Indonesia.

\*Corresponding author: sukarni.ft@um.ac.id

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### ABSTRACT

Global energy consumption has surged due to the population's rapid growth, leading to a switch to using renewable and clean energy. Among various strategies for developing renewable and clean energy, biomass pyrolysis emerged as attractive, and microwave irradiation pyrolysis is a prominent technique. This research aims to determine the ideal parameters for microwave-assisted pyrolysis of *Spirulina platensis* (SP) microalgae in order to produce high-value liquid products with the addition of activated carbon (AC) additives. Liquid bio-oil products were characterized concerning their calorific value, chemical bonds, and thermal stability. This research also investigates product distribution as a function of AC loading. Using 900W output power of microwave and 550 °C pyrolysis temperature, the pyrolysis process was shortened by 36.5% at 15% of AC loading. At 20% AC loading, the pyrolysis duration was shortened by 33%. This study demonstrated that the highest liquid and minimum solid residue were obtained at 20% AC loading. The heating value of liquid bio-oil with no AC additives was 35.419 MJ/kg, while at a 10% AC additive was 37.464 MJ/kg. The highest heating value was found at a 15% AC additive, which was 39.345 MJ/kg; meanwhile, at a 20% AC additive, it was 36.097 MJ/kg. These findings conclusively showed that the liquid bio-oil's quality was significantly improved by the addition of activated carbon (AC), establishing it as an attractive option for the microwave-assisted pyrolysis (MAP) process that produces advanced renewable fuels.

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**Keywords:** Activated carbon, biofuel, microalgae, microwave pyrolysis, *Spirulina platensis*.

## I. Introduction

Global energy consumption has surged due to the fast growth of the population in the world, driving an increased reliance on non-renewable sources like coal, oil, and gas [1]. This situation has prompted an urgent search for alternative fuels to sustain the industrial and transportation sectors globally. There is a continual rise in global natural resource demand, with projected oil demand increasing by 35% by 2025 [2]. These conditions cause global warming, which in turn threatens global climate change. This issue prompted a shift toward the use of clean energy, making renewable energy utilization a key focus of research and driving its rapid development [3]. This provides opportunities to develop renewable energy sources, including biomass [4].

Various methods exist to convert biomass into fuels and biochemical products, categorized into thermochemical, chemical, and biochemical processes [5]. Among these, thermochemical methods offer distinct advantages, such as versatility in handling different



feedstocks, shorter reaction times, and higher energy efficiency [6]. Pyrolysis, for instance, involves breaking down complex chemical compounds at moderate to high temperatures without oxygen, yielding solid, gas, and liquid products [7].

The pyrolysis method enables the conversion of biomass into solid, liquid, and gas products [8]. Conventional pyrolysis and microwave-assisted pyrolysis are the two well-known types of pyrolysis [9]. In the conventional pyrolysis process, heat is produced by a variety of sources, including heated surfaces, sands, and electrical [10]. Recently, microwave-assisted pyrolysis has emerged as a prominent thermochemical process due to its distinct advantages over conventional methods. This technique is renowned for its unique heating capabilities, superior product upgrading, and enhanced conversion efficiency. Microwave heating offers several advantages, including fast and environmentally friendly processing, higher product yields, the capability to process large raw materials directly, flexibility to handle diverse feedstock, no requirement for agitation or fluidization, and reduced particle (ash) content in resulting bio-oil [11]. Studies of microwave-assisted pyrolysis (MAP) using biomass such as corn stover [12], wood [13], rice straw [14], and pine sawdust [15] have been reported, and it is suggested that MAP is well-suited for the conversion of biomass.

Microalgae have attracted growing interest as a potential source of third-generation biofuels in recent years [16]. Microalgae biomass offers rapid growth rates and requires minimal land space compared to terrestrial biomass like switchgrass, with productivity rates up to 50 times faster [17]. Microalgae biomass production is higher than 30 times that of traditional oil crops per surface area, which are rich in oil [18], [19]. Microalgae do not significantly compete with traditional agricultural resources, as they can be grown on non-arable land or in wastewater, thus preserving valuable agricultural land [20]. Due to their numerous advantages, microalgae are considered one of the most promising biomass resources for replacing fossil fuels [21]. Consequently, the study of microalgae for energy production is of critical importance. *Spirulina platensis* (SP) stands out as a leading candidate due to its abundant cultivation [22]. It boasts a large-scale cultivation capacity of 3000 tons annually and has lower ash content than other microalgae species such as *Chlorella sp* and *Nannochloropsis* [12], [13], [16].

Microwave heating primarily relies on dipole rotation and ionic conduction, converting energy directly into heat instead of heat transfer [23]. However, dry biomass has a poor ability to convert microwave power into heat [24]. Since microwave pyrolysis involves selective heating, where the materials exposed to microwave energy exhibit varying dielectric responses due to interactions between their particles or molecules and internal magnetic fields [25]. It is necessary to use an additive in the microwave cavity or inside the reactor to get optimum microwave absorption ability and high efficiency of biomass pyrolysis to improve the pyrolysis conditions, product yields, and properties of the products [26]. Combining microalgae with an additive could enhance the material's ability to effectively absorb microwave power [27]. Activated carbon could be an excellent microwave absorber because of its behavior that is easily heated by microwave.

Anis et al. succeeded in producing liquid biofuel from pyrolysis of used cooking oil using the MAP method using a microwave oven, which has an easier, cheaper process and lower energy consumption. Because it speeds up chemical processes and reduces residence time, this technique has become one of the finest in the pyrolysis process, saving energy [28]. Hu et al. [29] converted *Chlorella vulgaris* using a microwave-assisted pyrolysis process on various catalysts, including CaO, SiC, activated carbon, and the solid residue of

the product (biochar). The highest biofuel yield was obtained at 87.47% when using a 5% activated carbon catalyst. Borges et al. [30] investigated the microwave-assisted pyrolysis (MAP) of *Chlorella sp.* and *Nannochloropsis sp.* strains, utilizing HZSM-5 as the catalyst and silicon carbide (SiC) as the absorber. The findings revealed that incorporating both SiC and HZSM-5 into the process led to substantial improvements in several key aspects. Specifically, the presence of these additives significantly enhanced the yield and quality of the bio-oil produced. In addition, the overall properties of the final bio-oil, such as its chemical composition and energy content, were notably improved, demonstrating the effectiveness of these materials in optimizing the pyrolysis process. The results highlight the potential of combining HZSM-5 and SiC to improve the efficiency and performance of MAP in producing high-quality bio-oil from microalgae. Eshun et al. [31] reported that the conversion of *Chlorella sp.* using microwave pyrolysis for 20 minutes at 750 watts power achieved the highest yield of 28.6%. Sardi et al. examined microwave-assisted pyrolysis (MAP) in low-rank coal (LRC) using two catalyst and receptor combinations: HZSM-5 with  $\text{Fe}_2(\text{SO}_4)_3$ , and activated carbon (AC) with  $\text{Fe}_2(\text{SO}_4)_3$ . The study revealed that the combination of AC and  $\text{Fe}_2(\text{SO}_4)_3$  produced a higher bio-oil yield compared to HZSM-5 with  $\text{Fe}_2(\text{SO}_4)_3$ . This result is attributed to the sufficient acidity of carbon-based materials like AC and LRC, which also serve as effective microwave absorbers [32], [33]. Hu et al. conducted pyrolysis of *Chlorella vulgaris* under varying microwave power levels, catalyst, amount of activated carbon, and solid residue content. The results indicated that increasing the microwave power levels and using a catalyst enhanced gas production. The catalyst facilitated the pyrolysis process, with activated carbon proving to be the most effective catalyst, followed by solid residue. The highest bio-oil yield, reaching 87.47%, was achieved with 5% activated carbon [34].

Based on the background above, activated carbon could promote the pyrolysis process of biomass, but pyrolysis of SP and activated carbon was uncommonly discussed. This study investigated the production and characterization of pyrolysis products from microwave-assisted pyrolysis of *Spirulina plantensis* (SP) and activated carbon as an additive. The research aims to determine the optimal amount of additive for microwave-assisted pyrolysis to obtain high-value-added chemical products. This research also investigates pyrolysis product yields and characterizes the liquid bio-oil concerning their calorific value, chemical bonds and functional groups, and fuel thermal behavior.

## II. Material and Methods

### A. Raw Material

The SP microalgae (Spiruganik+TM) used during the present study was purchased as a powder from the Jepara Brackish Water Cultivation Center. Mesh 60 (0.25mm) was used to sift the particle size.

### B. Additive

The additive used in this study was Haycarb activated carbon powder (AC). Powder-activated carbon was crushed and sieved into 20 mesh sizes. It was then dried in the oven at 100 °C for 2 hours. The dried activated carbon was then collected into the vial and ready to use. SP was mixed with AC at various amounts of 10, 15, and 20 (wt,%) by using mechanical mixing (magnetic stirrer) at 1200 rpm and 15 minutes. After the process, the SP+AC samples were ready to be used. The absorbent used in this study is granule-char. The granule char was 300g in each pyrolysis process. The absorbent was placed in a different place from the

sample and placed under the sample reactor as a source of heat to maintain temperature stability while the temperature-holding process was being carried out.

### C. Experimental Method

The pyrolysis of SP and variations of additives were carried out with a 2450 MHz frequency of microwave oven, using 900 W output power and 12.23 cm wavelength. The microwave was manufactured for kitchen use and has a capacity of 23L. In order to ensure that each pyrolysis experiment was carried out properly, the microwave oven was modified. Figure 1 shows a schematic of the microwave-assisted pyrolysis system.

The prepared sample was placed in a 500 mL clay pottery reactor, which was positioned vertically inside the microwave compartment. To purge the air from the reactor, N<sub>2</sub> gas was used as a carrier, flowing at a rate of 0.5 L/min for 15 minutes before the experiment began. K-type thermocouples were installed to monitor the temperatures of both the sample and the microwave cavity. One thermocouple was placed inside the reactor, ensuring it did not touch the bottom, in order to accurately measure the temperature of the sample. The temperature of the pyrolysis reaction was regulated using an Omron E5CWL-type temperature controller.

A double glass-tube condenser was used to condense the pyrolysis vapor product, with a pump circulating cooling fluid from a water bath containing a water-ice mixture. Two 500 mL glass bottles were used to collect the crude oil product. Non-condensable gases were vented through the exhaust and safely burned. The pyrolysis process was carried out until a temperature of 550°C was reached. The resulting bio-oil and biochar from the pyrolysis were then stored for further characterization.

### D. Pyrolysis Yield

Crude oil has compound physical and chemical properties of hydrocarbons that are influenced by the arrangement of the carbon chain. Besides crude oil, another by-product is obtained from pyrolysis. Solid and syngas are the products besides crude oil. Every product yield can be calculated by using equations as follows:

$$\%Y_{Liq} = \frac{M_{Liq}}{M_{SP}} \times 100\% \quad \dots\dots\dots (1)$$

$$\%Y_{Aqs} = \frac{M_{Aqueous}}{M_{SP}} \times 100\% \quad \dots\dots\dots (2)$$

$$\%Y_{Bo} = \frac{M_{Bo}}{M_{SP}} \times 100\% = Y_{liq} - Y_{aqs} \quad \dots\dots\dots (3)$$

$$\%Y_{Char} = \frac{M_{char}}{M_{SP}} \times 100\% \quad \dots\dots\dots (4)$$

$$\%Y_{gas} = 100\% - (\%Y_{Liq} - \%Y_{Char}) \quad \dots\dots\dots (5)$$

where % Y<sub>liq</sub>, % Y<sub>gas</sub>, and % Y<sub>char</sub> are the yields of liquid, gas, and char produced in wt.%; M<sub>liq</sub>, and M<sub>char</sub> are the weight of the liquid and the charcoal mass collected, respectively, while M<sub>SP</sub> is the initial mass of raw material during the pyrolysis process.

## E. Characterization

### 1. HHV

The purpose of Bomb Calorimeter testing is to accurately measure the calorific value of the sample being analyzed, which indicates the amount of energy released during combustion. This testing was conducted at the ITS Energy and Environment Laboratory, utilizing the Envilife OBC-1A+ model of the Bomb Calorimeter. During the process, the sample was carefully placed in the calorimeter, and the test was performed under controlled conditions to ensure precise measurements. The results obtained from the testing provided the calorific values of the samples, which are crucial for evaluating the energy content and potential efficiency of the materials being studied.

### 2. FTIR

The investigated samples' functional groups were examined using Fourier Transform Infrared Spectroscopy (FTIR). The measurement was carried out in the  $4000\text{--}600\text{ cm}^{-1}$  wave number range. The solid sample was combined with 5–10% KBr powder to get the samples ready for FTIR analysis. A Mini Hand Press tool was then used to press the homogenous mixture into KBr pellets. The KBr pill was prepared for analysis once it had formed. Graphs of transmittance or absorbance were used to display the results. A Shimadzu IR Prestige-21 instrument was used to conduct this analysis at the Laboratory of Advanced Materials and Minerals, Universitas Negeri Malang.

### 3. TGA

Thermogravimetric analysis (TGA) was used to examine fuel stability at temperatures between 30 and  $900\text{ }^{\circ}\text{C}$  and a heating rate of  $10^{\circ}\text{C min}^{-1}$ . A Mettler—TGA 851 thermobalance was using an  $\text{N}_2$  atmosphere with a flow rate of  $25\text{ mL min}^{-1}$ . For each experiment, approximately 100 mg of the oil sample was used. An aluminum crucible with a 100 mg capacity was employed to ensure a more uniform distribution of the sample's temperature gradient. A computer connected to the thermoanalytical apparatus collected all of the thermal conversion data, which were then assessed using the Mettler STAR program.

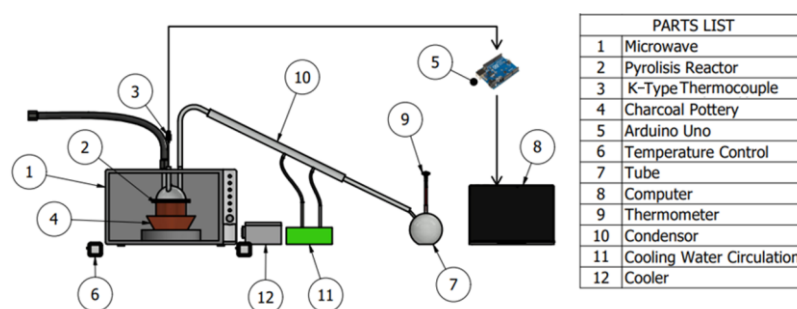


Fig. 1. Microwave-assisted pyrolysis apparatus.

## III. Results and Discussions

The discussion will include the temperature profile, pyrolysis product yield, bio-oil heating value, chemical bond characteristics, and thermal stability characteristics. These groupings are then discussed and explained as follows. Proximate analysis reveals that SP has a high volatile matter content (77.96 wt.%), indicating its suitability for energy recovery through thermochemical conversion technologies [35].

### Temperature Profile

Figure 2 illustrates the real-time temperature profiles of SP pyrolysis, both with and without the addition of AC, subjected to a microwave power 900 W. The temperature stage during microwave pyrolysis can be categorized into four distinct stages: (i) an initial rapid temperature increase from ambient conditions to 100°C; (ii) a drying phase occurring between 100°C and 150°C; (iii) the evaporation of hydrocarbons within the range of 150–450°C; (iv) the pyrolysis process spanning from 450°C to 600°C.

The presence of moisture can indicate a rise in temperature at the initial stage (Figure 2). Due to the dielectric properties and polar nature, moisture (water) could generate heat under a microwave environment, resulting in a good microwave absorber for the first stage of SP pyrolysis. When microwaves interacted with the SP, the moisture absorbed the microwaves and caused water molecules to align themselves to the radiation. This created molecular friction, generating energy as heat and causing a rapid temperature increase [36]. After the moisture evaporated from the SP, essential elements such as hydrocarbons did not absorb the microwaves. This is why the temperature was observed not to rise significantly and tended to remain stable after reaching 100 °C where the SP gradually dried [37]. Around 150°C, the temperature started to increase until it reached 550 °C. At this stage, primary components were started to pyrolyze and volatilize. During the pyrolysis, condensable and non-condensable gas were produced, and the condensable gas would be condensed by a condenser and trapped in a product collector; the non-condensable gas would be removed by an exhaust fan and ready to be burned for safety reasons.

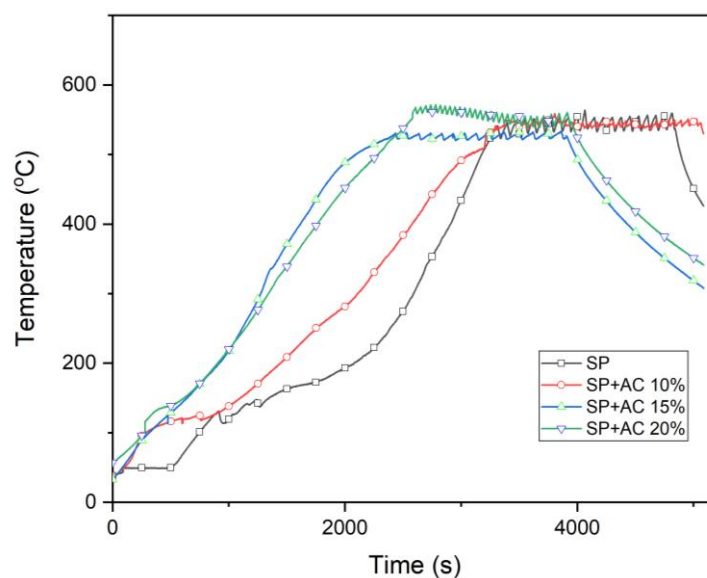


Fig. 2. Effect of AC addition on the real-time temperature of SP

Figure 2 shows that the addition of AC on SP increased the heating rate, and the reaction temperature was faster than SP with no AC addition. AC played an important role in heat transfer and interaction between microwave and SP during the pyrolysis process. SP with no AC additive needs 66 min to reach the set temperature. SP with 10% AC addition reaches the set temperature almost the same, around 63 min, which means 4.5% faster than pure SP. The 10% AC addition promotes drying faster than pure SP. SP with 10%, 15%, and 20% AC loading has the same drying process behavior, which is faster than the SP with no AC addition, this phenomenon has also been reported in [36]–[38].

In an earlier study conducted by Zhifeng et al. [34] a similar phenomenon was observed when *C. vulgaris* was mixed with activated carbon (AC) as an absorber. The combination of *C. vulgaris* and activated carbon resulted in a 17.88% higher maximum pyrolysis temperature compared to pure *C. vulgaris*, indicating a significant enhancement in thermal performance. Furthermore, the rate of temperature increase was considerably higher in the mixture, with a remarkable 174.30% rise compared to the pure *C. vulgaris* sample. This demonstrates that the presence of activated carbon not only elevates the maximum temperature achieved during pyrolysis but also accelerates the overall heating process, suggesting that activated carbon plays a key role in improving the efficiency and effectiveness of the pyrolysis reaction.

The prominent differences were observed in the second stage, in which 15% and 20% AC loading promoted a very steep temperature rise, indicating the starting decomposition and pyrolyzing of the SP. The addition of 15% AC took 40 min to get the set temperature, while the 20% AC took 42 min. The entire pyrolysis process was shortened by 36.5% at 15% of AC loading. At 20% AC loading, the pyrolysis duration was shortened by 33%. It is caused by the absorption ability of activated carbon, which is admirable [39]. The polar molecules of SP will be distributed averagely on the surface of the activated carbon molecules. On the other hand, after the activation process, activated carbon often has polar functional groups on its surface, which increases its ability to interact with various molecules [34]. Then, under the microwave field, activated carbon, which absorbed and distributed many polar molecules of SP, moved and collided a million times within a second and generated so much heat, which made the pyrolysis reaction better. This indicates that adding AC could significantly accelerate the microwave pyrolysis process, where increasing the AC concentration will result in more polar molecules being distributed. The addition was necessary for achieving time efficiency and energy saving in microwave-assisted pyrolysis of SP.

#### *Product Yield of Pyrolysis*

Figure 3 shows the product distributions of SP pyrolysis at various amounts of AC additives and a pyrolysis temperature of 550°C. The product distribution contained gas, char, and liquid phases (water and bio-oil). In the pyrolysis of SP without additives, the yields were 27.2% gas, 30% char, and 42.8% liquid phase. Adding 10% AC yielded results similar to pure SP, indicating minimal impact on product yield. However, with a 15% AC addition, the liquid phase yield increased to 46.6%, while char and gas yields decreased to 27% and 26%, respectively. This indicates that 15% AC significantly enhanced liquid yield compared to pure SP and the 10% AC addition. Adding 20% AC resulted in the highest liquid phase yield among all tested conditions, suggesting this concentration is optimal for maximizing liquid product yield.

It can be expected that increasing the amount of AC in the system will lead to greater hydrocarbon decomposition. This effect was observed as a decrease in the yield of the solid product (biochar). As the AC concentration increases, the char yield obtained decreases. As seen in Figure 3, char yield was 30% without additives. Char yield decreased to 28%, 26.2%, and 22% as the AC loading increased from 10%, 15%, and 20%, respectively. The more AC concentration increases in SP, the faster the pyrolysis heating process in SP will be. The larger amount of activated carbon (AC) used the greater the contact area of the additives involved in the microalgae degradation process. This reduces the activation energy, leading to a higher conversion of reactants into products. Increased contact between the reacting substances results in higher liquid yield obtained [40]. Additionally, according to Lili et al.

[41], activated carbon has a high surface area, which significantly enhances reaction performance. This is because activated carbon can spread the polar molecules in the SP more evenly and cause faster heating because, under the microwave field, the polar molecules collide with each other and produce heat. As the temperature rapidly increases from ambient to its maximum value, endothermic reactions occur faster, and they absorb much heat [19], [42]. Furthermore, there was a significant enough temperature differential between the sample and the surrounding air to permit heat escape. The pyrolysis reaction was so strong under these circumstances that it briefly produced a significant amount of bio-oil and gas.

The operating temperature used was 550°C, which is the optimal temperature for producing bio-oil according to several previous studies [40], [43]. Whereas earlier research used pyrolysis methods in a fixed-bed reactor with atmospheric nitrogen and no catalysts to convert *Spirulina sp.* into biofuels [43], these studies found that the optimal temperature for biofuel was 550°C. The results demonstrate that pyrolysis at this temperature yields a high liquid phase. At this temperature, the pyrolysis produces a larger molecular weight, which leads to a condensable gas. Therefore, the bio-oil yield is high at 20% AC because it has a larger contact area than the other. Aswie et al. stated that a higher proportion of liquid production would result from a longer pyrolysis duration. This is because a longer pyrolysis time would result in more contact between the reacting components, which would increase the liquid yield.

By adding 20% AC, the time needed will be less because AC promotes a high contact area and a high heating rate. This can avoid excessive costs and also save time in bio-oil production. Besides that, the longer the pyrolysis process takes, the pyrolysis will reach an equilibrium condition. Once the equilibrium condition is reached, the bio-oil production will not change much. So, the longer the reaction time, the greater the inefficiency, as supported by Patil et al. [44] the pyrolysis process lasts too long, it can lead to overheating, which causes excessive gas production, increased char formation, and energy loss during the process. This study demonstrated that the highest liquid and lowest solid residue were obtained at 20% AC loading. Adding AC leads to a very high heating rate and higher contact area, which will produce more tar during the process.

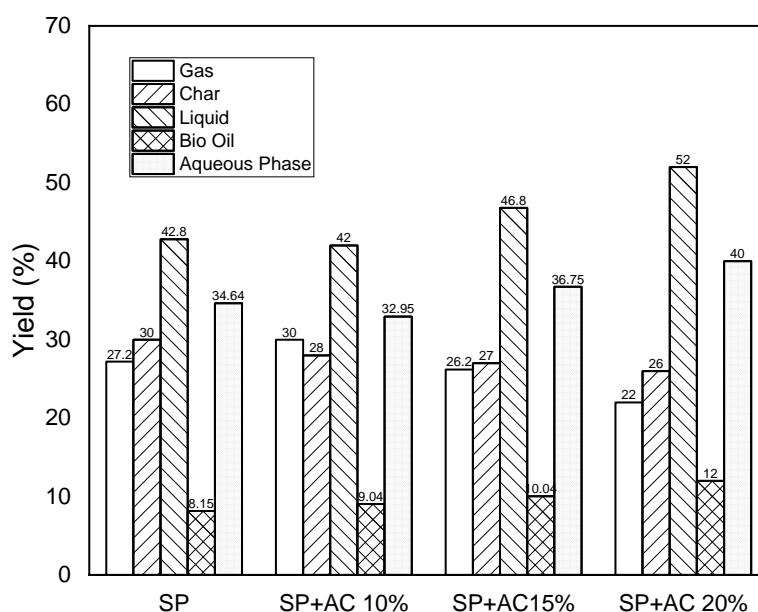


Fig. 3. Product yield of pyrolysis



### Heating Value

As we can see in Figure 3, the heating values of bio-oil from the pyrolysis at temperatures of 550°C using additives have different values. The heating value at an SP with no additives was 35.419 MJ/kg, while at a 10% AC additive was 37.464 MJ/kg. The heating value at a 15% AC additive was 39.345 MJ/kg, and the last 20% AC additive was 36.097. The overall results indicated that the addition of activated carbon (AC) significantly improved the quality of the liquid bio-oil, leading to a higher heating value compared to the bio-oil produced without any AC additive. This enhancement was particularly noticeable at a 15% AC loading, where the bio-oil exhibited the highest heating value. This suggests that the presence of activated carbon plays a crucial role in optimizing the bio-oil's energy content, making it a more efficient and viable option for use as a fuel. The findings highlight the potential benefits of using AC as an additive in the pyrolysis process to improve the performance of bio-oil for various applications.

According to Jena et al. [45], a slow and low pyrolysis process potentially has a higher O element in the liquid product, resulting in a lower energy content than a high-temperature one. Therefore, the addition of activated carbon additives will speed up heating; this will support a faster pyrolysis process and heat up faster compared to low amounts of additive. This process results in a low amount of O in the liquid fuel resulting in an increase of the heating value. The 20% additive has an excellent heating speed, but 20% loading produces more water phase than the other based on Figure 3, which could lead to a decrease in heating value. It is also found that the increase in temperature could increase the water content in bio-oil (Figure 3), possibly caused by secondary reactions [36]. A decrease in heating value can be caused by the high level of oxygenate compounds in crude oil, such as water, hydroxy aldehydes, hydroxy ketones, and phenols, which are highly oxygenated compounds in crude oil. The presence of water in bio-oil significantly impacts its usability as a fuel, as it can reduce the heating value, cause phase separation, and delay ignition. These factors create challenges in efficiently utilizing bio-oil as a reliable energy source [46]. However, the specific mechanisms behind these effects are not yet fully understood, highlighting the need for further in-depth research to clarify the underlying causes. Moreover, the high oxygen content in bio-oil is another critical aspect that warrants detailed study. Comprehensive characterization methods, such as Gas Chromatography-Mass Spectrometry (GC-MS), are essential to analyze and better understand the bio-oil's chemical composition, properties, and potential for optimization. This additional research could help address the limitations and improve the overall performance and application of bio-oil as a sustainable fuel alternative.

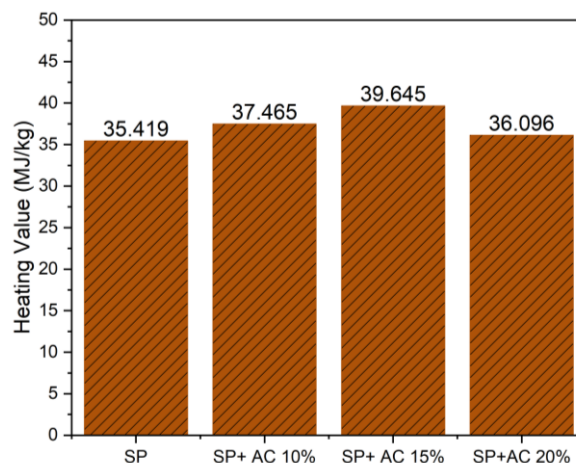


Fig. 4. Heating value of the liquid product

### Thermogravimetry Analysis

Thermoanalysis techniques, including thermogravimetric analysis (TGA), are crucial for evaluating the thermal behavior of bio-oil. TGA provides data on The relationship between the weight loss of bio-oil and temperature under an inert atmosphere, which was analyzed in this study. Thermogravimetric analysis (TGA) was conducted to examine the weight loss stages of the liquid bio-oil. The TGA curves for the bio-oil samples derived from the microwave pyrolysis of *Spirulina platensis* (SP) are shown in Figure 5. For comparison purposes, the thermograms for the same category of additives are displayed together in the figure. As shown in Figure 5, the bio-oil produced through microwave-assisted pyrolysis (MAP) with activated carbon additives exhibited a similar decomposition process.

From Figure 5, it can be seen that the thermal decomposition process of all liquid bio-oil samples is investigated. All liquid bio-oil from pyrolysis using or no AC additives experienced three stages of decomposition, where all samples have similar decomposition mechanisms. Stage I occurs below 120°C, stage II between 120–477°C called the decomposition stage, and stage III is the formation of solid residues at around 535°C. This result is in line with the study that was reported by Liu et al. [47]. Liquid bio-oil from no AC additive has the lowest temperature to decompose with residue under 1%, which is only 0.006 mg. In contrast, the liquid bio-oil sample containing additives such as a 10% AC additive sample having a residue of 3.9% (0.45 mg), a 15% AC additive having a residue of 2.4% (0.27 mg), and a residue of a 20% AC additive that was 2.3% (0.24 mg). Liquid bio-oil of SP with no AC addition has the lowest residue. This phenomenon was due to the low impurities (such as coke) produced during pyrolysis [48]. In contrast, the addition of 10% AC has the highest residue. The increase of the solid residue in bio-oil could be caused by many impurities, and the amount of AC was too small; the SP could not get enough heat to decompose AC. It's critical to keep the solid content in bio-oil to a minimum since they can act as catalysts and speed up the aging process [49]. Conventional boilers have lenient standards for solid content, making bio-oil easily usable in these systems. However, the particulates in bio-oil can adversely affect the precise nature of diesel fuel injection systems, thereby restricting their use in such applications [10].

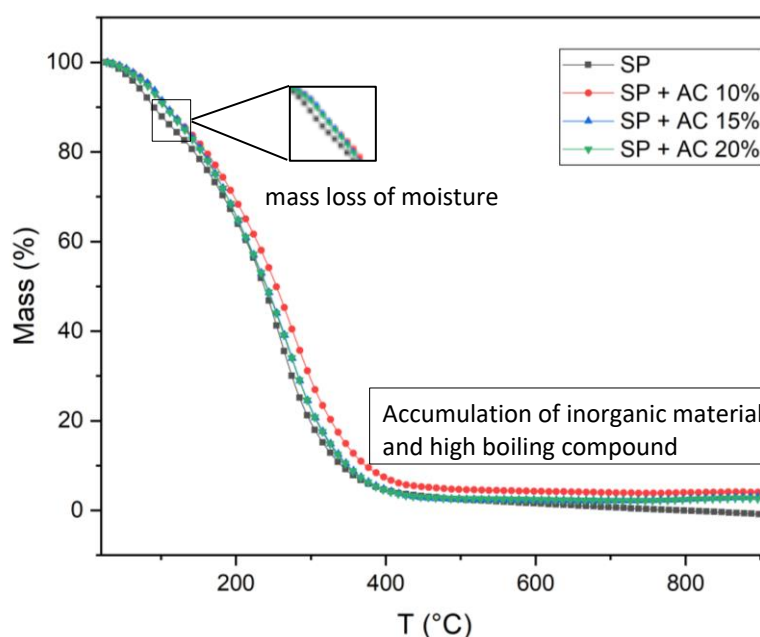


Fig. 5. TG profiles of liquid bio-oil at various amounts of AC additives

In accordance with Figures 5 and 6, it can be understood that liquid bio-oil from SP without AC within the temperature range of 27.27—450°C had a rapid mass loss, where about 98.043 wt.%. Following this decomposition process, the DTG peak was encountered at 261°C, in correlation with the crack of the protein element [7]. The TG curve for liquid bio-oil from SP without AC additive has a second mass loss at 712.94°C, whereby only about 2.85% mass was lost, and a seamless DTG peak was observed at 714°C due to secondary cracking. The red line of Figures 5 and 6 illustrate the curve of liquid bio-oil originating from 10% AC addition. The TG curve shows a direct incline line, indicating there was a massive mass loss. The liquid bio-oil originating from 10% AC addition has experienced a drying process within a range of temperatures of 80—150°C, then the thermal degradation is still going on until reaching the peak at 275.56°C and decreases and finishes at around 550°C.

The blue line of Figures 5 and 6 illustrates the curve of liquid bio-oil originating from 15% AC addition. There are two peaks in the graph, the first peak at 98°C and the second peak at 270°C. The DTG of liquid bio-oil originating from 20% AC addition shows a little difference at the first peak compared to the 10% and 15%; when others have a sharp look, this one has a little slope at 109°C then the rate starts to increase until it reaches the peak at 268.55°C, after that the rate slowed down until the temperature around 430°C.

In accordance with Figures 5 and 6, it was well understood that liquid bio-oil originating from SP no AC additive decomposed earlier than three others, and it seemed fast to decompose at first. The TG-DTG graph of all the samples looks similar, but the shape of the graph is a little bit different at the first peak. It can be seen that 10% of AC addition has the highest temperature to decompose, compared to 15% and 20%, which means that liquid bio-oil originating from 10% AC addition does not easily react and decompose. Generally, a higher temperature corresponding to the peak of sample weight loss indicates better thermal stability [50].

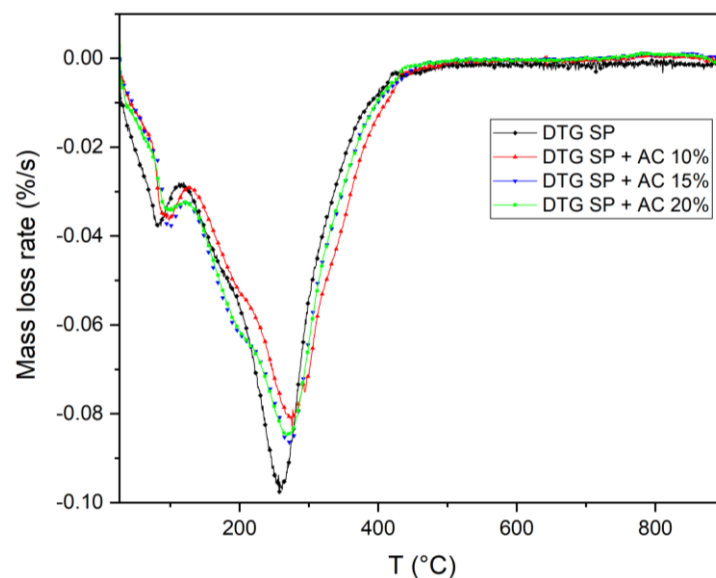


Fig. 6. DTG profiles of liquid bio-oil at various amounts of AC additives

### *Infrared Spectra Changes*

The spectra of SP microalgae and liquid bio-oil originating from SP pyrolysis with AC additives of 10%, 15%, and 20% are presented in Figure 7. FTIR is an effective method for

analyzing small molecules in liquid products. This technique can identify the type of organic matter by analyzing the functional group of organic molecules. The structural variation of liquid biofuel from pyrolysis products can be analyzed using FTIR spectroscopy [51].

Figure 7 presents the FTIR spectra results between the liquid bio-oil originating from SP pyrolysis with and without the AC addition. The complex and long-chain molecules in microalgae are broken down into shorter molecular chains with a variety of functional groups by pyrolysis at 550°C. The peaks that emerged at the specified wavenumber range in the four samples show different compounds presented in liquid bio-oil.

The band spectra at the wavenumber range 3200—3600  $\text{cm}^{-1}$  contain an O-H stretching, which was derived from water, lipids, protein, and carbohydrates. This finding was linear with the study of Barbosa et al. [52] that classified the O-H in the water group. The observed bands at the wavenumber range 3003—3512  $\text{cm}^{-1}$  in correlation with N-H stretching of the amide group [53]. The peak found in the wavenumber range 2750—2950  $\text{cm}^{-1}$  is related to aliphatic C-H stretching of methylene [46], and bands in the wavenumber range 1870—1540  $\text{cm}^{-1}$  strongly linked to C=O stretching of esters that originated from lipids of microalgae. A strong peak at 1750  $\text{cm}^{-1}$  indicates the absence of aldehydes, ketones, and ester compounds. In this wavenumber (1750  $\text{cm}^{-1}$ ), C=O stretching is strongly associated with fatty acid. A peak at 1450  $\text{cm}^{-1}$  is related to C-H bending, which is a vibration of methyl. Unpronounced bands at the range of 1540—1870  $\text{cm}^{-1}$  are linked to the C=O stretching of the carboxyl group [54]. A small peak at 750—810  $\text{cm}^{-1}$  correlates with the C-H group (aliphatic character) and is also associated with the N-H and C=O groups derived from protein [33]. It is observed that hydrocarbons, aliphatic chemicals, nitro compounds, and amide groups are among the functional groups found in the liquid bio-oil that result from SP pyrolysis with AC loading.

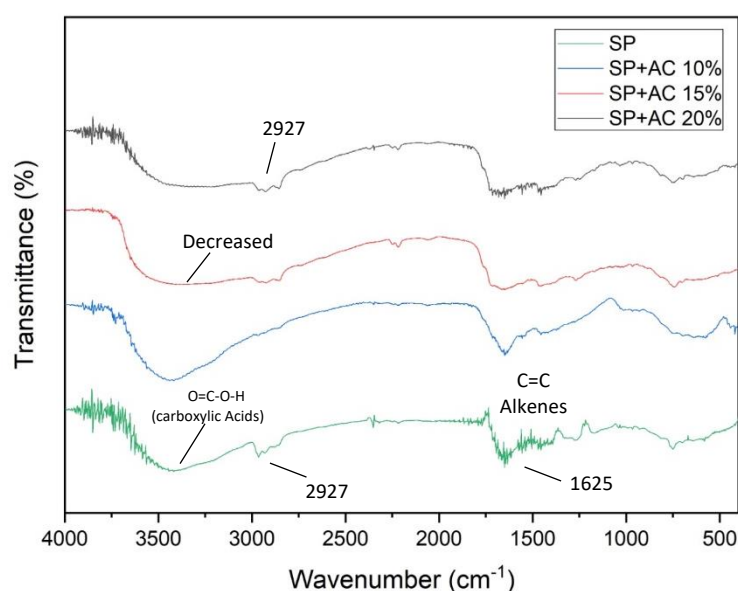


Fig. 7. Infrared spectra of liquid products in different amounts of AC additives

The addition of AC to SP during the pyrolysis process significantly affects the chemical composition of the final products, as evidenced by the FTIR spectrum. The decrease in the band intensity at 3400  $\text{cm}^{-1}$  indicates that AC effectively adsorbs water or hydroxyl compounds from SP. This finding was also supported by the data from the heating value test, whereby the highest heating value was found at 15% AC loading, which was 39.64

MJ/kg. This increase is likely due to the reduction of O-H (water) bonds, as the presence of moisture in the fuel decreases its heating value [45]. Additionally, variations in the intensity and position of bands in the 3000-2800  $\text{cm}^{-1}$  range suggest that AC influences the aliphatic structure in the pyrolysis products. The increased band intensity at 3000  $\text{cm}^{-1}$  with 20% AC indicates high aliphatic stability. Furthermore, the increased intensity of the band at 1700  $\text{cm}^{-1}$  with higher AC concentrations suggests an increase in carbonyl compounds, likely due to further decomposition or reactions with AC. The formation of new compounds is also indicated by the presence of a new band at 2200  $\text{cm}^{-1}$  with 15% AC, suggesting the formation of compounds such as carbon or nitriles during pyrolysis with higher AC concentrations. Moreover, changes in the intensity of bands at 1450  $\text{cm}^{-1}$  and 750  $\text{cm}^{-1}$  indicate that AC affects the stabilization and modification of organic compounds in the pyrolysis products, particularly hydrocarbons and aromatic compounds. Overall, mixing activated carbon (AC) into SP could significantly reduce hydroxyl compounds, modify the aliphatic structure, increase the concentration of carbonyl compounds, and lead to the creation of new compounds that are absent in samples without additives.

#### IV. Conclusions

Using activated carbon (AC) as an additive in the microwave-assisted pyrolysis of SP presents a promising thermochemical conversion method for improving both the efficiency of the process and the yield of bio-oil. AC significantly increases the heating rate and speeds up the drying process during the pyrolysis up to 36% at 20% AC loading. Giving AC 20% to SP pyrolysis could increase bio-oil yield to 52%, but the optimum heating value was at 15% additive loading with 39.6 MJ/kg. Bio-oil without AC additive has the lowest decomposition temperature, adding 10% AC, which results in the highest decomposition temperature among concentrations tested, indicating lower reactivity but better thermal stability. Increasing AC can affect macromolecular bonds by reducing hydroxyl groups and increasing carbonyl groups. Several characteristic tests of SP microalga bio-oil with AC additives have been carried out. Further studies are required to gain a deeper understanding of the properties and composition of bio-oil, including evaluations of its volatile content, water content, and aging characteristics.

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