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CHARACTERIZATION OF PALM SHELL-DERIVED BIO-OIL THROUGH PYROLYSIS

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Abstract. *Lignocellulosic biomass is a renewable resource used to produce energy, fuels, and chemicals. This study aimed to determine the effect of pyrolysis temperature on product yield and product characterization of bio-oil. In this study, palm shells were selected and prepared as raw materials for bio-oil production. Palm shells were first soaked in 10% HCl and then pyrolyzed at temperatures of 300 °C, 350 °C, 400 °C, and 450 °C in a fixed bed reactor. Afterward, the reactor will emit smoke which later will condense into bio-oil. The experimental results show that a temperature of 450 °C will be a better choice for higher bio-oil yields (44.59%). The characteristics of the bio-oil obtained are density (905 – 1015.17 kg/m^3), Kinematic Viscosity (1.21 – 1.5 mm^2/s), and flash point (60 – 68.7 °C).*

Keywords: *palm shell; bio-oil; pyrolysis; temperature; delignification*

1. Introduction

Due to the large concentrations of lead compounds, hazardous gases, photochemical oxidants, and dangerous particulate matter in the atmosphere, the excessive usage of fossil fuels to supply current society's energy demands has a detrimental effect on the environment and contributes to global warming (Mota *et al*., 2014). Hence, the development of alternate energy sources is pushed by modernity, including using lignocellulose to create biofuels and electricity (Sabarman, 2019). Bioenergy and biofuels can be generated from the pyrolysis process, which undergoes condensation and produces liquid smoke, which contains phenolic compounds, carbonyls, and acids—one of the lignocellulosic biomass sources that can be found in palm shell (Sahrani, 2019). Currently, Indonesia produces the most palm oil worldwide. Crude Palm Oil (CPO) is processed in palm oil refineries to create palm oil (Badan Pusat Statistik, 2020). Palm shells are one of the largest palm oil processing wastes, reaching 60% of oil production (Ahmad, 2014). Palm shell from palm oil mills is a low-efficiency boiler fuel since it is available, cheap, and easy to get. Thermal processing of palm shells into bio-oil improves utilization as biomass energy to replace fossil fuels and minimizes disposal challenges (Sulhatun, 2019). Over the next three years, the Sub-Directorate for Plantation Crop Statistics estimates that the area of oil palm plantations will grow by about 2.3 %. The Agricultural Director General of Plantations at the Ministry of Agriculture stated that Indonesia makes the most palm oil of any country in the world (Pramudya, 2017).

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Lignocellulosic biomass, such as palm shells, can be processed chemically, biochemically, or thermochemically to produce products with economic benefits (Ghenai *et al.*, 2020). Thermochemical pathways, known as pyrolysis, have received much interest since it is an economical and environmentally acceptable technique (Van Schalkwyk *et al.*, 2020). Biomass pyrolysis produces bio-oil, bio-char, and syngas without oxygen (Ogunkanmi *et al.*, 2018). The heat conversion of organic materials without oxygen is referred to as pyrolysis. Pyrolysis is a process that uses heat to turn biomass into liquids (bio-oil), solids (char), and gases at temperatures between 300 °C to 800 °C. One of the newest sustainable energy techniques; pyrolysis, offers significant liquid yields (75 %) with little gas and biochar in a controlled setting (Sukiran *et al.*, 2011 & Radha *et al.*, 2011). However, compared to gasification and combustion, pyrolysis is the best thermochemical conversion technique for producing bio-oil as the primary product. The volatile compounds in the pyrolysis process condense into bio-oil when the components are promptly quenched. In many industrial applications, bio-oil may replace fuel oil, such as power-generating turbines, internal combustion, and boilers. It can also undergo additional processing to generate secondary products, better fuel, or chemicals (Ahmad *et al.*, 2014).

This study devised the palm shell pyrolysis technique. The purpose of this study is to analyze how different pyrolysis temperatures (300 °C, 350 °C, 400 °C, and 450 °C) and also delignification affected product yields and attributes. Experiments were done in a fixed-bed reactor to see if bio-oil could be used as an alternative to traditional fuels. Palm shell will be given pretreatment with 10% HCl under previous studies where numerous academics have researched pretreatment techniques to lower lignin content and enhance biomass quality because the extensive number of lignin could hamper the pyrolysis process (Nematizade *et al.*, 2012; Dai *et al.*, 2019; Liu *et al.*, 2011). Meanwhile, other studies have revealed that, under optimal conditions, the value of bio-oil produced from four different agricultural wastes resulting into a different variation in the content of the bio-oil as well. (Biswas *et al.*, 2017). Since palm shell has a high value of lignin, reducing the lignin is urge in order to get the better result of bio-oil.

2. Methods

2.1. Material Preparation

Sumatera Selatan local plantations provided the palm shell waste used in this research. Palm shell was sun-dried for two days to remove unbound moisture. Palm shell was pulverized and sieved to 1-2 mm size. After that, as many as 3 kgs samples that will be used as the thermal cracking material were dried for 24 hours at 105°C. Gravimetric analysis was used to estimate the amounts of cellulose, hemicellulose, and lignin in the lignocellulosic components of palm shell. The result was attained, as demonstrated in Table 1. The processed sample was characterized using proximate and ultimate analysis. The standard and method used in the ultimate and proximate analysis process were from the previous study (Rusdianasari *et al.*, 2022). The analysis data are listed in Table 2.

Table 1. Palm Shell Contents

Parameter Analysis	Value (%wt)
Cellulose	25.2
Hemicellulose	23.7
Lignin	44.6

Table 2**.** Proximate and Ultimate Analysis of Palm Shell

		Unit	Method	Current Study	Comparative Literature*
Proximate Analysis	Moisture Content	%	ASTM D 3173-17a	15.21	19.58
	Ash	%	ASTM D 3174-12	7.75	2.75
	Volatile Matters	%	ASTM D 3175-18	58.78	69.78
	Fixed Carbon	%	ASTM D 3172-13	18.15	19.37
Ultimate Analysis	Carbon (C)	%	ASTM D 5373-16	48.68	47.53
	Hydrogen (H)	%	ASTM D 5373-16	4.98	5.11
	Nitrogen (N)	%	ASTM D 5373-16	0.29	0.32
	Sulfur (S)	%	ASTM D 4239-18e1	0.06	0.05
	Oxygen (O)	%	ASTM D 3176-15	37.83	36.14

*(Rusdianasari *et al.*, 2022)

Table 3. Lignin Levels in 1 Gram of Palm Shell

Delignificator	Lignin (%)
Before Treatment	45.37
Aquadest	43.43
HCl 10%	26.23

2.2. Pretreatment

As many as 3 samples were prepared; each sample contained 10 grams of palm shells. Sample 1 was filled with 50 mL of distilled water, while samples 2 and 3 were filled with 100 mL of HCl solution with a concentration of 5% and 10% (v/v), respectively. The sample was heated on a hotplate at 121 ^{o}C for 30 minutes. The sample was filtered and washed with water until the pH was neutral. The resulting residue was dried in an oven at 105 ^{o}C for 1 hour. The results were obtained as shown in Table 3.

2.3. Pyrolysis Process

In order to conduct the pyrolysis experiments, 3 kg of palm shell were loaded into a stainless steel tubular reactor with an internal diameter of 214 mm and a length of 360 mm. An internal K-type thermocouple was used to regulate the reactor's temperature. The reactor was heated externally by a vertical electric furnace and arranged alternately with different temperatures. Palm shell were vaporized under a high temperature and then flowed into condenser where it turned into liquid oil. The set-up for pyrolysis is described in Figure 1. Characterization of bio-oil from palm shell pyrolysis results was studied by looking at the effects of temperature (300 °C, 350 °C, 400 °C, and 450 °C). The percentage of yield was calculated by weighing bio-oil products. The density, kinematic viscosity, and flash point of bio-oil were determined by ASTM D 4052, ASTM D 445, and ASTM D 93, respectively.

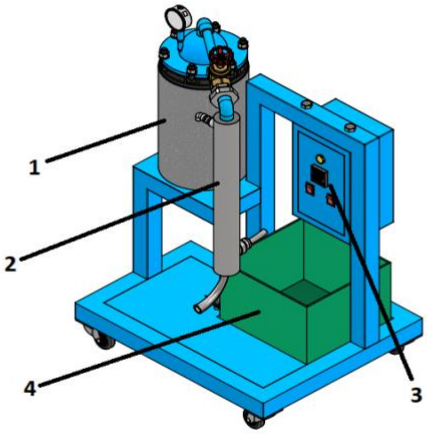

Figure 1. Pyrolysis set-up used in the experimental work (1) reactor, (2) condenser, (3) control panel, and (4) water tank

3. Results and Discussion

3.1. Pretreatment of Palm Shell

Palm shell in this study was pretreated before being used as feed for pyrolysis. Pretreatment in this study is divided into two processes, the size reduction process and the delignification process. Reducing the size of palm shell will increase the contact between the raw material mixture and the reactor wall which will increase the rate of heat transfer during the pyrolysis process (Zhang *et al*., 2018).

Palm shell was also pretreated using 10 % of HCl solution as a delignification medium. The result of before and after the delignification process can be seen in Table 3. Based on Table 3, it can be seen that after the delignification process with 10 % HCl, there was a change in the content of lignocellulosic compounds from palm shell. Lignin content decreased by 19,14% after treated with 10 % HCl.

3.2. Physical Appearance of Bio-Oil Produced

Generally, bio-oil is dark in colour and smells like smoke (Achmad *et al*., 2022). Those characterizations are because the product produced from the pyrolysis reactor is liquid smoke. As seen in Fig. 2, the bio-oil produced was dark in colour and had a precipitate at the bottom of the storage bottle. In Fig 2(c), the precipitate has started to decrease, and the resulting colour is slightly more transparent than in Fig 2(a-b). In many ways that bio-oil can be changed, heating is needed.

Organic polymerization and cracking in bio-oil are caused by the heating process, which tends to make coke. The biggest obstacle with using bio-oil, which involves heating it, is that coke could reduce bio-oil's carbon conversion efficiency and prevent access to the reactor chamber. If the surplus palm shell is not proportional to the temperature increases and is not entirely transformed into bio-oil products, it will result in residue or coke. Therefore, the residues were decreased as the temperature increased. The proportion of coke was also formed from lignin-derived compounds in biomass pyrolysis (Hu *et al*., 2020). Coke formation was observed for all pyrolysis of biomass (Chang *et al*., 2013). This is seen in the products' darker hues and scents.

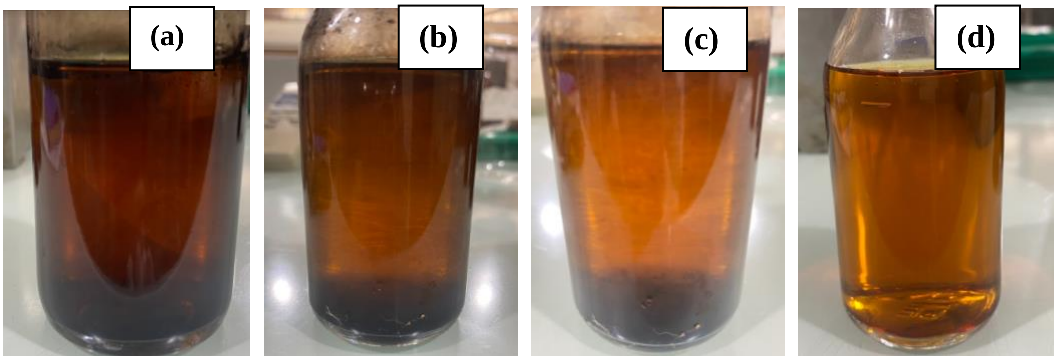

Figure 2. Bio-oil at (a) 300 °C, (b) 350 °C, (c) 400 °C, and (d) 450 °C.

The effects of pyrolysis temperatures (300°C, 350°C, 400°C, and 450°C) on the % yield, density, kinematic viscosity and flash point of palm shell that was treated with 10% HCl, 90 minutes reaction time were shown in Fig.3(a-d).

3.3. Yield

Yield equals the quantity of raw palm shell used divided by the weight of the condensate collected. Yield is also a metric used to assess a process' overall performance, the more yield produce, the better (Sandika *et al*., 2021). Figure 2-a shows the effect of temperature (300, 350, 400, and 450 ^{o}C) on the bio-oil yield. It can be concluded that temperature significantly influences the yield of bio-oil. The highest bio-oil yield, 44.59 %, was achieved at 450 ^{o}C, the optimum temperature. The results revealed that when the temperature rose from 300 ^{o}C to 450 ^{o}C, the bio-oil output increased maximally from 29.04 % to 44.59 %. This is due to the fact that cracking will more readily and effectively occur at higher temperatures. Adding more heat energy at a higher temperature was also a factor in improving product yield. (Heriyanto *et al*., 2018). Meanwhile, previous studies showed that the product dropped at a temperature of 450-550 ^{o}C due to a decrease in specific and organic products. The secondary cracking of volatile compounds occurred at temperatures above 500^{o}C. Beyond this point, the yield decreases as more gaseous products are realized due to secondary cracking (Lin *et al*., 2015). Considering the yield factor and the results, the temperature of 450°C was determined to be the ideal pyrolysis temperature because it constantly gives highest yield percentage among other temperature below or above it.

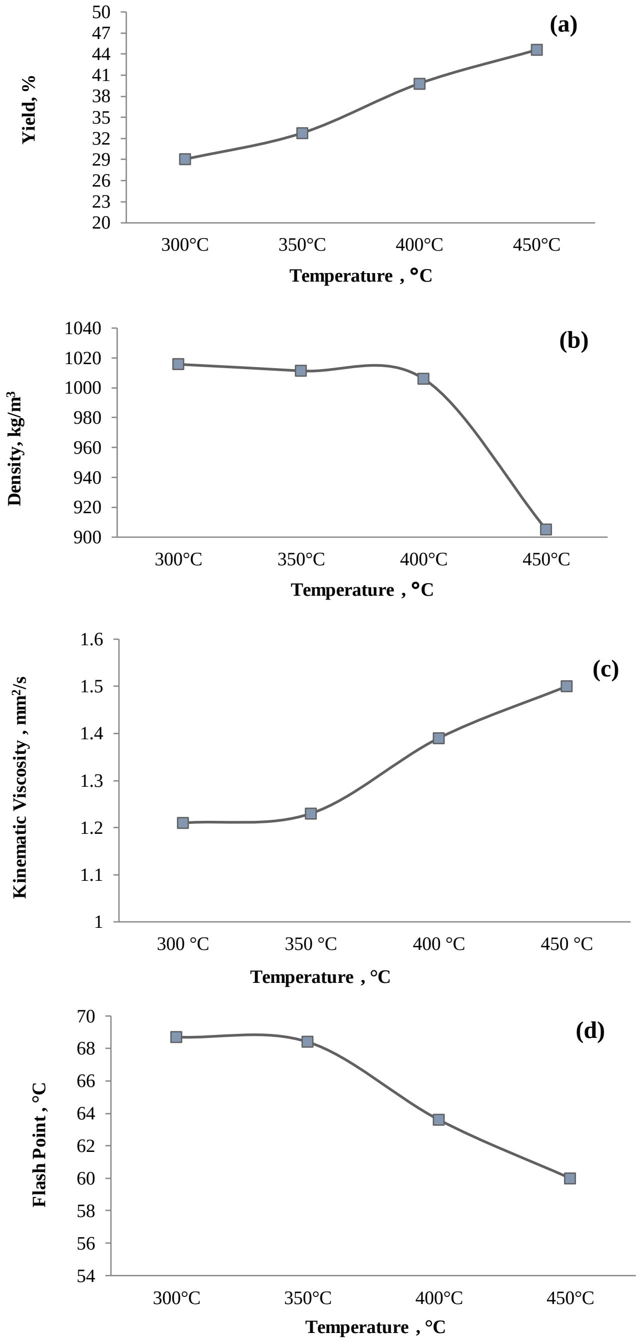

Figure 3**.** The temperature of pyrolysis's impact on the (a) yield, (b) density, (c) kinematic viscosity and (d) flash point of bio-oil.

3.4. Density

The occurrence of secondary reactions that result in the generation of light products will rise at high deoxygenation temperatures (Arend *et al*., 2011). According to the research, the density of the final product decreases as temperature increases. Density relies on the number of carbon chains; fewer carbon chains, lesser density. The number of carbon atoms produced influences the density itself; the longer the c chain, the more c atoms are made simultaneously at a given density. The density of the product obtained in this study ranged from 905 – 1015.17 kg/m^3. The density range obtained is greater than the diesel density range of SNI 3506-2017 (815 – 879 kg/m^3) because there are still many other compounds in bio-oil products, such as tar compounds. Most are paraffin, naphthenic, or aromatic hydrocarbons, each with unique chemical and physical properties (Asikin-Mijan *et al*., 2017).

3.5. Kinematic Viscosity

The kinematic viscosity of bio-oil can be seen in Fig. 2-c. The increase in kinematic viscosity is proportional to the chain length of the hydrocarbon compounds. The number of paraffinic hydrocarbons generated significantly impacts the product's viscosity; value increases with chain length. Fig. 2-c shows that the measured viscosity value drops as the heating temperature is raised. With less viscosity, a minor product portion will occur (Aziz, 2019). Too much viscosity will increases pipe friction, makes the pump work harder, and complicates filtering, which increases dirt deposition and gasoline fogging (Irawan *et al*., 2021). The kinematic viscosity of the product acquired is in the range of 1.21 – 1.5 mm2/s which is smaller than the kinematic viscosity of SNI 3506-2017 (2 – 4.5 mm^2/s). This difference in viscosity is due to the fact that when testing the resulting bio-oil product, the drop in the falling ball was not hampered by the viscosity of the bio-oil.

3.6. Flash Point

One crucial practical aspect is flash point; the higher the flash point, the higher the level of safety during operation, transportation, and storage (Özener *et al*., 2014). The oil will ignite when exposed to a spark at a temperature below its flash point (Afriansyah *et al*., 2022). Based on the flash point test findings, it was obtained between 60 – 68.7 °C. Things that can affect the number of a product's flash point is the volatile matter content, in. The less volatile the bio-oil contains, the harder it is to ignite the product. In this case, palm shell has 58.78 % volatile matter as shown in Table 2. A compound's flash point increases linearly with viscosity (Zaher and Gad, 2017). In addition, viscosity also affects the size of the flash point. The inversely proportional graph can also prove this in Figures 2c and 2d. The flash point obtained from this study meets the SNI 3506-2017, which is greater than 52 °C.

4. Conclusion

Pretreatment of palm shell in 10 % HCl affect the content of lignin compounds from palm shell. Lignin content decreased by 19,14%. The optimum conditions obtained from the research results are at temperature of 450 ^{o}C. Because under these conditions, the pyrolysis reactor can produce a maximum yield of 44.59%. The characteristics of bio-oil obtained are density (905 – 1015.17 kg/m^3), Kinematic Viscosity (1.21 – 1.5 mm^2/s), and flash point (60 – 68.7 ^{o}C). Those value are not in accordance yet with SNI 3506-2017 because further processing is needed so that bio-oil can be used as fuel.

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