THE EFFECT OF PROCESS PARAMETERS ON LIQUID SMOKE PRODUCTION FROM PALM OIL PYROLYSIS

by
Herlin Sumarna\textsuperscript{1),} Riman Sipahutar\textsuperscript{2) \& Irwin Bizzy\textsuperscript{3)}}
\textsuperscript{1,2,3)Mechanical Engineering, Sriwijaya University}
Email: \textsuperscript{1}herlindarman187@gmail.com, \textsuperscript{2}rimansipahutar@ft.unsri.ac.id \& \textsuperscript{3}gorekayasa@gmail.com

Abstract
Palm kernel shells are waste from Crude Palm Oil (CPO) factories that have not been optimally utilized, so further processing is carried out to increase their economic value by the pyrolysis process. The pyrolysis process is carried out using a reactor combined with a spiral stirrer, and a condensation process that uses a refrigeration system to control the coolant temperature at 18°C. The purpose of this study was to determine the effect of particle size, temperature and residence time on the pyrolysis of palm shells sourced from the Musi Banyuasin area of South Sumatra. The pyrolysis experiments were carried out at pyrolysis temperatures of 300°C, 325°C, and 350°C and the palm shell particle sizes of -3+5 mesh and -5+7 mesh for residence time varied for 3 hours, 4 hours, and 5 hours. The maximum liquid smoke yield obtained is 28.6% at 350°C for palm shell particles -5+7 mesh and a residence time of 5 hours. In terms of temperature influence, the lowest smoke yield is 19% of the total biomass at 300°C. For the residence time variation, the maximum liquid smoke product was 28.6% of the total biomass at a size of -5+7 mesh for 5 hours.

Keywords: Liquid Smoke, Palm Kernel Shell \& Pyrolysis.

INTRODUCTION
The conversion of biomass energy into useful and sustainable forms that best meet human needs is a common concern for scientists, engineers and technologists. From an energy transformation point of view, pyrolysis is an attractive option among the various thermochemical conversion processes due to its simplicity and higher ability to convert biomass to bio-oil. Utilization of biomass provides the possibility to produce value-added products such as chemicals, activated carbon, etc. Which is, it is an attractive economic and technological solution [11]. Based on data from the Central Statistics Agency (BPS) of Palembang City, oil palm production in 2018 was 548 tons with a plant area of 220 hectares. The development of oil palm plantations in Indonesia is progressing very rapidly so that it has an impact on the amount of oil palm waste. The production activities of palm oil factories produce a large amount of waste, both solid and liquid waste [14]. Solid waste from a palm oil mill with a capacity of 100 thousand tons of fresh fruit bunches (FFB) per year will produce around 6 thousand tons of shells, 12 thousand tons of fibers and 23 thousand tons of empty fruit bunches [2]. Palm shell waste has not been used optimally, so it needs further processing to increase its economic value.

In terms of its chemical composition, palm kernel shells have the potential to be used as a source of raw material for making liquid smoke because one of the chemical components contained in palm kernel shells is a lignin compound containing 23% in palm kernel shells [15]. Pyrolysis is a method that can be used to convert biomass into products with higher economic value [16]. Liquid smoke is a pyrolysis product that can be used in various fields. The quality and yield of pyrolysis liquid (liquid smoke) and pyrolysis gas depend on many variables such as operating
parameters (eg. temperature, residence time of raw materials), type and composition of biomass, and pyrolysis techniques used, including the type of reactor. Pyrolysis of biomass with a high lignin content produces pyrolysis fluids with high calorific value [8].

Based on the above, the author will conduct a study that aims to analyze the effect of palm shell size, pyrolysis temperature, and pyrolysis time on liquid smoke products and test the liquid smoke produced using the Gas Chromatography-Mass Spectrometry (GC-MS) tool to determine the components contained in liquid smoke.

LITERATURE REVIEW

Pyrolysis

Pyrolysis is a process similar to gasification, but compared to other thermal conversion technologies for fuels, pyrolysis occurs at a relatively low temperature range (300-600°C) and in the absence of any oxidizing agents [4]. There are two main pyrolysis techniques: slow and fast. Slow pyrolysis has been used for centuries in traditional charcoal making but can also be carried out using modern reactors. Slow pyrolysis techniques are characterized by slow heating rates, relatively long residence times of solids and vapors, and usually a lower reaction temperature than fast pyrolysis. On the other hand, fast pyrolysis is characterized by high heating rates and short residence times. Simple pyrolysis technology that only uses heating will result in the conversion of lignin mainly into solid and gas coke in the disproportionation process, such as lignin-rich materials in nature are converted into coal, not petroleum, during the natural transformation process [9].

Usually there are three products in the pyrolysis process, namely gas, oil pyrolysis, and charcoal, the proportions of which depend on the pyrolysis method, biomass characteristics and reaction parameters. This implies that if the shell is heated without contact with air and given a rather high temperature, there will be a decomposition reaction of the complex compounds that make up the shell and produce substances in three forms, namely solids, liquids and gases [7]. Pyrolysis fluid yields decreased at higher temperatures and with longer residence times [1]. In fast pyrolysis, 50-75% (wt) of the raw material is converted into pyrolysis liquid [8].

Raw materials may consist of small pieces of biomass (wood and grass), plastics, decomposed materials (such as food scraps and paper). This material is then compressed and filtered to reduce the particle size to less than 10 mm for ease of use in pyrolysis experiments [17]. The raw material sizes for slow and fast pyrolysis are 5–50 mm and <1 mm. Smaller biomass particle size gives a higher rate of fast pyrolysis reaction, but particles that are too small are difficult to handle. Reducing the particle size of raw materials can increase the heating rate, so the time needed to get liquid smoke is shorter [11].

Liquid Smoke

Liquid smoke is obtained by condensing the smoke produced through a slow pyrolysis chimney. The process of condensing smoke into liquid smoke is very useful for protecting air pollution caused by this process. In addition, liquid smoke contains a number of chemical compounds which are considered as raw materials for preservatives, disinfectants or as biostimulants [6].

Liquid smoke contains components such as phenols, organic acids and carbonyls which function as antibacterial, antifungal and coagulant. These compounds also have a role as a distinctive taste [5]. Each biomass contains different cellulose and lignin, so the results of pyrolysis will produce liquid smoke with varying specifications [14].

The chemical composition of liquid smoke is determined by many factors, such as the type of biomass, pretreatment of raw materials (particle size and shape, moisture and ash content), pyrolysis conditions (temperature, heating rate, residence time, pressure, gas environment) as
well as vapor filtration and condensation. (filter, condensing media and method, cooling rate). Therefore, the liquid smoke produced from different materials and by different pyrolysis reactors may be very different from one another [10]. Sari’s research results (2007) state that the main components of liquid smoke are 1,2-benzene dicarboxylic acids and diethyl esters, acids (as acetic acid) between 4.27-11.30%, phenolic compounds (as phenols) 2.10–5.13% and carbonyl compounds (as acetone) 8.56-15.23%.

MATERIAL AND METHODS

Raw Material

Palm kernel shells are collected from PT. Hindoli in the Musi Banyuasin area. Before being used as a raw material for pyrolysis, the palm kernel shell is tested to determine the chemical composition contained in the palm kernel shell. Palm kernel shells are crushed and sieved to produce -3+5 mesh and -5+7 mesh sizes. The analysis of the composition of the palm kernel shell was carried out according to the standard test methods of SNI 06-3730-1995 (Moisture Content), ASTM E1755-01 (Ash), In The House (Hemicellulose, Cellulose, and Lignin).

Experimental Procedure

The obtained palm kernel shell is pyrolyzed in a batch reactor type. The reactor has a diameter of 40 cm and a reactor height of 120 cm. To reduce combustion heat loss, the outer reactor is coated with a tube with a diameter of 50 cm, and has a height equal to the height of the reactor. Operating temperatures are carried out at 300°C and 350°C, the cooling system for condensing smoke into liquid smoke uses a refrigeration system with the cooling water entering the condenser there is a thermostat to control the temperature. whereas non-condensable gases are discharged into the atmosphere. Liquid smoke and charcoal products were weighed to calculate the percentage yield. The effect of palm kernel shell particle size, temperature, and pyrolysis time on product yield will be investigated.

Testing of liquid smoke was carried out using the Shimadzu Gas Chromatography-Mass Spectrometry (GC-MS) tool. GC-MS analysis of liquid smoke products was carried out according to ASTM E2997 standard. The oven temperature was started at 35°C for 2 minutes, increased to 250°C at 20°C per minute and held at this temperature for 20 minutes. The injector port temperature and detector temperature are set at 280°C. The carrier gas, helium, was set at a flow rate of 47.5l per minute and the injector port separation ratio was set at 50:1. An appropriate amount corresponding to 0.03 g of liquid smoke was used and diluted with HPLC content of methanol to a volume of 0.5 ml using a bottle. After that the dough is whipped and filtered. Finally, 1.0 µl of the mixture was injected with a 5.0 µl syringe into the GC-MS apparatus.
Figure 1. Schematic of experimental set-up

RESULTS AND DISCUSSION

the results of testing the composition of the palm shell

Below are the results of testing the composition of the palm kernel shell.

Table 1. Palm Shell Composition Test Results

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Results</th>
<th>Unit</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cellulose</td>
<td>11.52</td>
<td>% dry basis</td>
<td>In House Method</td>
</tr>
<tr>
<td>Hemicellulose</td>
<td>2.68</td>
<td>% dry basis</td>
<td>In House Method</td>
</tr>
<tr>
<td>Lignin</td>
<td>76.22</td>
<td>% dry basis</td>
<td>In House Method</td>
</tr>
<tr>
<td>moisture content</td>
<td>12.18</td>
<td>%</td>
<td>SNI 06-3730-1995</td>
</tr>
<tr>
<td>Ash</td>
<td>3.19</td>
<td>% dry basis</td>
<td>ASTM E1755-01</td>
</tr>
</tbody>
</table>

Effect of particle size on the pyrolysis yield.

Figure 2 shows the mass percentages of liquid smoke, charcoal and gas products for different particle sizes (-3+5 mesh and -5+7 mesh) of the palm kernel shell raw material at a maximum temperature of 350°C and residence time of 5 hours. It was observed that the maximum percentage of liquid smoke collection was 28.6% of the total palm shell feed for a particle size of -5+7 mesh while a smaller amount of liquid smoke was obtained at larger particle sizes. It is possible that the results obtained are that the smaller particles are thoroughly supplied with heat during the pyrolysis process. Conversely, in larger particle sizes, the heat supply during pyrolysis is still not comprehensive due to the small particle surface area, causing a reduction in product yield. As shown in Figure 2, the highest char yield of 55.2% was obtained using a particle size of -3+5 mesh, while the lowest char yield was 46.6% using a particle size of -5+7 mesh. Likewise, the highest and lowest gas yields were obtained using particle sizes of -5+7 mesh and -3+5 mesh, respectively. The increase in particle size causes a larger temperature gradient inside the particles so that at certain times the core temperature of the particles is lower than the surface temperature [12]. In addition, the use of stirrers also helps in the heat distribution process for raw materials. The particle size of raw materials is known to affect the results of liquid smoke from pyrolysis products, the particle size is small enough to facilitate the heating process uniformly.

Effect of temperature on the pyrolysis products yield

Figure 3 shows the variation in the weight proportions of liquid smoke, charcoal and gas at different pyrolysis temperatures (300, 325, and 350°C) with the optimum palm shell particle size at -5+7 mesh as obtained in this study. In this way, it is found that at the lowest pyrolysis temperature (300°C), the decomposition process is relatively slow and charcoal is the main product. With temperatures from 300°C to 350°C, the yield of liquid smoke
was obtained 28.6% of the total palm shells used while the lowest smoke yield was 22.4% of the total used shells. Charcoal yield decreased gradually when the pyrolysis temperature was increased from 300°C to 350°C. The highest yield of charcoal was 55.8% of the total oil shells used at a temperature of 300°C and the lowest yield was 46.6% of the total shells obtained at 350°C. A decrease in char yield with an increase in temperature can be approximated from the greater decomposition of the palm shell at a higher temperature or through the decomposition of the rang residue [13]. The highest and lowest gas yields were 24.8% and 21.8% at temperatures of 350°C and 300°C, respectively. Product gas increases with increasing pyrolysis temperature.

**Figure 3. Effect of temperature variation on pyrolysis products yield**

**Effect of residence time on the pyrolysis products yield**

Figure 4 shows the product yield (%) of liquid smoke against variations in pyrolysis time. The maximum liquid product is 28.6% of palm shell feed at -5+7 mesh size and 5 hours residence time with 350°C pyrolysis temperature. Meanwhile, at the time of pyrolysis 3 and 4 hours the resulting liquid smoke product was lower than the pyrolysis time of 5 hours. The lowest yield of liquid smoke was obtained during pyrolysis for 3 hours with a pyrolysis temperature of 300°C. The longer pyrolysis time can cause a higher gas release rate and can cause more liquid smoke products, therefore, the cooling water control in the condenser must be strictly maintained because the greater the gas release rate the higher the heat rate of the gas.

**CONCLUSIONS**

The effect of particle size on the yield of liquid smoke shows that the palm shell size of -5+7 mesh is the optimum palm shell size to produce liquid smoke of 28.6% and palm shell size -3+5 mesh is the optimum particle size to produce charcoal at temperature 350°C. In addition, the effect of temperature and pyrolysis residence time on the product showed that the temperature of 350°C at a residence time of 5 hours was the optimum temperature to produce liquid smoke.

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