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Thermal and Physical Properties of Patchouli Essential Oil Industry Residue as Renewable Feedstock for Bioenergy

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Abstract
Patchouli plant is one of main agricultural commodities in Indonesia with an area of 64.67 ha. The solid waste produced from the distillation industry is around 98% of total feedstock. The aim of this research is to disclose the characteristics of solid waste biomass from patchouli essential oil industry harvested from Lhokseumawe, Indonesia. The properties of patchouli biomass before and after distillation was analyzed by using a number of techniques including proximate, bomb calorimeter, TGA-DTG, DSC and lignocellulosic analyses. Five kilograms of biomass was collected after patchouli harvesting then sorted into four categories i.e. leaves, branches and trunk. and mixture of all. Another set of biomass residue was collected after distillation process and grouped similar to those collected before distillation. All samples were then dried, ground and sieved to 50 mesh size. The analysis results showed that the highest heating value was observed from the sample of patchouli leaves collected before distillation process with a value of 15.65 MJ/kg where its volatile matter content was 81.26%. Compositional analysis of lignocellulosic suggested that 27% in pre-distilled branches. Mixture of all parts was found to contain 35% cellulose that was the highest. Lignin content with 42% value was found in after distilled trunk.
Keywords: Heating Value; Lignocellulose; Proximate Analysis; TGA-DSC Patchouli;

Introduction
There has been increasing concern of researchers to agriculture and farm sectors biomass as alternative source of energy. This is due to depletion of fossil energy source and alternative energy source increasing demand. On the top of that, rise of population also contributes to high energy consumption. Many studies reveal that biomass derived energy source is potential to energy supply, considering the tendency of rising price of fossil fuel. The utilization of biomass as energy source is attractive since it emits zero net CO_2, indicating that it is not a greenhouse gas producing material, which is a carbon neutral. There are many sources of biomass; plants, trees, grass, roots, farming residues, wastes and livestock wastes. One of farming waste biomass that is abundant in Lhokseumawe is patchouli solid waste. Total area of patchouli field in Lhokseumawe is around 42.59-64.67 ha, the percentage of solid waste produced from industrial distillation process of patchouli is around 98-98.5% of raw material.

There have been researches utilizing patchouli distillation waste. Research conducted titled as wax effectivity for fly repellent with patchouli distillation waste as active substance combined with clove oil (Sri Usmiati, 2012). The optimum concentration was stated to be 50% with 87.6% repellent after ten minutes and 100% repellent after thirty and sixty minutes. Another research with title usage of patchouli leaf dregs as compost (T. Salim, 2008); it showed that the shrinkage fulfills the requirement of readily used compost (23.21% for Agrisimba and 28.45% for EM4 types). During its preparation process, it reached the mesophilic phase with its specific microorganism. The third research was forming liquid smoke from dregs of patchouli pyrolysis process (Trianna, 2014). The optimum product was found at 400 °C, 65% xs value after 35 minutes. The reaction kinetics followed pseudo homogenous unreacted core model. This research was aimed to examine physical, thermal and chemical properties of patchouli distillation process waste. The achieved characteristic data will be employed for technological development of patchouli biomass as high valued products.

Materials & Methods
Patchouli biomass was collected from North Aceh district. The used patchouli was Lhokseumawe varieties, which was Pogostemon cablin Benth. The five kilograms raw material was categorized into leaves, branches, trunks and mixed groups. Each of them was dried in oven, mashed with grinder then sieved through 50 mesh filter. After that, lignocellulosic analysis was conducted using Chesson-Datta method aimed to examine the chemicals contained in patchouli plant. On the top of that, proximate analysis standardized with ASTM D 1762-84, heating value and TGA-DSC were to comprehend

the plant's characteristics.

a. Lignocellulosic Content (Chessson-Datta Method)
One gram of dried sample and 120 ml water was refluxed at 100 °C in water bath for one hour. Then it was filtered and the residue was neutralized with hot water. Neutralized residue was dried in oven until constant weight as mass (b). Residue (b) was reacted with 150 mL H_2SO_4 0.5 M, then refluxed at 100 °C for one hour; the product was filtered, neutralized with water and dried until constant weight as mass (c). 10 mL H_2SO_4 72% added to dried residue soaked in room temperature for four hours. After that, addition of 150 mL H_2SO_4 0.5 M before reflux at 100 °C for two hours was done. Rinsing the residue with water and dried in oven at 150 °C before cooled in desiccator until constant weight reached as mass (d). after that residue was dusted and weighted marked as mass (e). Each component was calculated according to the following equations:

$$\text{Soluble Hot Water } (\%) = (a-b)/a \times 100\% \quad (1)$$

$$\text{Hemicellulose } (\%) = (b-c)/a \times 100\% \quad (2)$$

$$\text{Cellulose } (\%) = (c-d)/a \times 100\% \quad (3)$$

$$\text{Lignin } (\%) = (d-e)/a \times 100\% \quad (4)$$

$$\text{Ash } (\%) = e/a \times 100\% \quad (5)$$

b. Water Content (ASTM D 1762-84) The principle of water content examination was through the loss of sample mass after being heated at standard temperature and time. The first step was by weighing sample on porcelain crucible that had been weighed before (A) after that it was heated at 105 °C at two hours then cooled in desiccator with mass (B). The water content was determined with the following equation:

$$\text{Water Content } (\%) = (A-B)/A \times 100\% \quad (6)$$

c. Volatile Matter Content (ASTM D 1762-84)
Volatile matter content was determined by heating the sample without oxidation at standard condition, then the water content value was updated accordingly. The step was done by weighing the sample on porcelain crucible with empty mass had been determined, heated at 105 °C for two hours. The sample then cooled in desiccator with mass (B). The cooled sample was heated in furnace at 500 °C for three minutes and 950 °C for five minutes then weighed with mass (C). The volatile matter was determined with the following equation:

$$\text{Volatile Matter } \% = (B - C)/B \times 100\% \quad (7)$$

d. Ash Content (ASTM D 1762-84)
The principle of ash content determination is by transforming sample completely into ash at standard condition. The first step was by heating the sample on porcelain crucible at 105 °C for two hours. After that, t was cooled for one hour then heated in furnace for six hours at 750 °C. The ash content was determined with the following equation:

$$\text{Ash Content} = \text{Sample mass after heated in furnace} / \text{Sample mass before heated in furnace} \times 100\% \quad (8)$$

e. Fixed Carbon Content
The content of bounded carbon on activated charcoal was from the carbonization process except ash ang other volatile matter. The following equation was used:

$$\text{Fixed Carbon } (\%) = 100 - (IM + VM + Ash) \quad (9)$$

f. Heating Value
Heating value determination was conducted with Bomb Calorimeter. The instrument used to determine heating value was Automatic Calorimeter-K88890. The crucible maximum capacity of calorimetric bomb was 1.1 gr hence the sample weight must be less than maximum crucible capacity. Sample heating value was determined by observing the temperature incrementation in calorimeter bomb facet, length of burnt wire and amount of remaining sample every one-minute time interval.

g. Bulk Density
In order to examine the bulk density of patchouli, a known volume and mass of cylinder was prepared. Having added 50 mesh sized patchouli powder to fill the cylinder fully, the mass of patchouli was weighed. The density of patchouli was determined with the following equation:

$$\rho = m/v \quad (10)$$

h. Thermogravimetric Analysis (TGA)
Thermogravimetric Analysis (TGA) DTA-TGA-DSC-STA, Linseis type TA PT 1600 using 50 mL per minute liquid nitrogen was used to examine the composition of patchouli solid waste sample heated until 1000 °C. Perkin-Elmer Differential Scanning Calorimetry (DSC) was used to analyze the transformation happened to sample during burning from 30 °C to

1000 ºC with 20 ºC incrementation per minute.

Results and Discussion

This research was aimed to examine the characteristics of patchouli waste by analyzing heating value, lignocellulosic proximate analysis and TGA-DSC analysis. Patchouli heating value was determined by summing the heating value and chemical compositions, specifically lignocellulose. From previous study, it was found out that for five kilograms of after distilled patchouli, there was 2.40 kilograms trunk, 1.55 kilograms leaves and 1.00 kilograms branches and remaining was wastes.

Hemicellulose Analysis

Hemicellulose is one of light heterogeneous polysaccharides members. Hemicellulose is usually in fifteen to thirty weight percent from dried lignocellulose. It is relatively easy to be hydrolyzed with acid to be monomers such as glucose, mannose, galactose, xylose and arabinose. Hemicellulose binds cellulosic fiber sheets into microfibrils enhancing cell wall stability.

This research can be seen that the highest hemicellulose contents are 24%, 25% and 27%. High cellulose content in pre-distilled patchouli also affected by volatile matter. During pretreatment process, steam explosion fractured and polymerized hemicellulose producing cellulose and increasing the material porosity (S.H. Soh, 2020). The higher the temperature, the more cellulose achieved. This is due to xylan, which is one of hemicellulose components, polymerized into xylose that is hydrated to be purfural (D. Devi, 2019).

Cellulose Analysis

Cellulose is main component of plant wall cell. It is plain chain polymer of glucoses bounded with ß -1,4 glucoside. The cellulose content can be seen in the range of 12%-35%. The lowest cellulose content was found to be the mixture before distillation, and the highest cellulose content was patchouli branch after distillation process. High level of cellulose may be interpreted as high level of water content [6]. This is due to cellulose fiber is soft and shorted hence it is readily degraded, which shows relatively low level of energy potential (Harunsyah, 2012).

Lignin Analysis

Lignin is three-dimensional polymer that is based on phenyl propane and groups of hydroxyl, carbonyl and methoxy, which acts as natural adhesive material in biomass. Its structure is carbon and ether based bounding forming three-dimensions in which polysaccharides and hemicellulose are found. Lignin purity was found to be 19-41%. This purity is due to the presence of minerals and water in patchouli. The analyzed water and ash contents were found to be 5.56% dan 8.96% respectively. Lignin purity analysis is based on the difference between its dissolved amount and contaminant compound in 72% H_2SO_4 solution. Contaminant in the lignin sample is remaining cellulose or its degrading product. The use of strong concentrated acid dissolves cellulose and leaves saturated lignin, which then weighed in the form of ash. Low water content in lignin is due to its hydrophobic property that is beneficial in synthesizing surfactant in oil refinery with Enhanced Oil Recovery (EOR) system. Besides, lignin has potential as energy source as its natural chemical properties (I. Barmina, 2013).

Water Content

Water is one of considerations in utilizing biomass as energy source. Water content reflects burning heating quality value, burning capacity and produced smog. Water content value was found to be in the range of 3.88-8.41%. The highest value was found to be after-distilled patchouli waste mixture, the lowest value was found to be pre-distilled leaf. Water content after distillation was due to water remaining after the process. High level of water content was due to numerous pores that adsorb water, which is in line with the study done by (R. Faizah H, 2019) who reported that pore numbers and other chemicals such as cellulose, hemicellulose and lignin presence enhances high water content. Patchouli water content is also affected by its hygroscopic properties, which shows its capability in adsorbing water from air. This is also supported by (A. Susanto, 2013), whose study showed that the more lignocellulose with small size content, the more water content presence. In addition to that, carbon and ash presence also increases water content, which results in lower releasing energy. This is due to part of energy usage in vaporizing water rather than fully used to light fire (S. Mustamu, 2016).

Volatile Matter

Volatile matter is non water matter that evaporates as result of material decomposition. The more burning volatile matter, the more smog produced (D. Hendra, 2012). High volatile matter makes less burning efficiency. Volatile matter value in this research was found to be between 69.95-85.51%. The highest volatile matter value was found to be pre-distilled patchouli at 85.51%, while the lowest value was found to be after-distilled patchouli mixture at 69.95%. The characteristic values shows that pre-distilled patchouli has high volatile matter. This is due to decomposing compounds beside water such as aromatic compounds (saponin, terpenoid dan steroid) and hydrocarbon content. High volatile content is affected by high hemicellulose and water content in patchouli. According to (J. A. Fuwape, 1997), high volatile content is affected by chemicals such as hemicellulose, extractives and water that readily evaporate at high temperature. Hemicellulose content of patchouli plant before distillation is 21-27%, while after distillation 9-25%.

Ash Content

Ash content is inorganic residue amount produced from carbonization process. The residue contains minerals that present in burning process. High ash content results lowering heating value of biomass (S. Artati, 2012). This research can be seen that the ash content is in the range of 8.69- 18.97%. The highest as content was found in leaf after distillation that is 18.97%, while the lowest ash content was pre-distillation mixture; 8.69%. high ash content was due to readiness of patchouli plant to adsorb water and oxygen and the presence of minerals after distillation process, hence increases ash content. Low ash content in pre-distilled mixture was due to high organic content that is easily bounded during burning resulting in low ash produced. The ash content is proportional to inorganic material in biomass and residue form burning process that has no carbon inside. The substances that are contained in ash are silica, calcium, magnesium oxide. Thus, it has negative effect on heating value since the materials are not easily burnt.

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Fixed Carbon Content

Fixed carbon is carbon that is bounded to charcoal beside water fraction, volatile matter and ash. The presence of fixed carbon depends on ash content and volatility matter, which are related reciprocally. Fixed carbon content has proportional relation with heating value. Moreover, according to (N. Fitri, 2017) carbon content depends on ash content and volatile matter. At this research, the range of fixed carbon active is in between 0.5989 – 4.1822%. high carbon content in patchouli leaf after distillation is due to low water and ash content. On the other hand, patchouli branch has the lowest carbon content because high amount of water and ash content.

Heating Value

Heating value is main indicator in determining energy source material quality that is based on chemical composition, water content and ash content. Heating value is the results of chemical component and water interaction. This research can be seen that the highest heating value was found to be 15649 kJ/kg, which is the pre-distilled patchouli leaf. The lowest heating value was found to be at 13144 kJ/kg; pre-distilled patchouli mixture. The heating values is dependent on several factors, such as water content, ash content, volatile matter and carbon content. Water content is one of determining factors as the lower its content, the higher heating values (W.D.S. Tavares, 2013). High heating value results in more efficient and less material in burning process (B. Batubara, 2017). This is due to the fact that the higher heating value, the faster burning process (M. T. Ali Sabit, 2019). At this research was, it can be comprehended that carbon content plays important role in determining heating value. The higher carbon content, the higher heating value. Carbon content depends on volatile matter and as content as well; the higher volatile matter and ash content, the less carbon content.

TGA Patchouli After Distillation

Thermal degradation analysis was carried out to determine the decrease in mass weight of the sample during an increase in temperature (at a constant rate). Therefore, with this analysis, information on the increase in weight due to evaporation, decomposition, or perhaps weight gain due to the binding of gas molecules from the atmosphere can be obtained. The results of weight loss on samples with a particle size of 50 mesh can be seen in Figure 1 below:

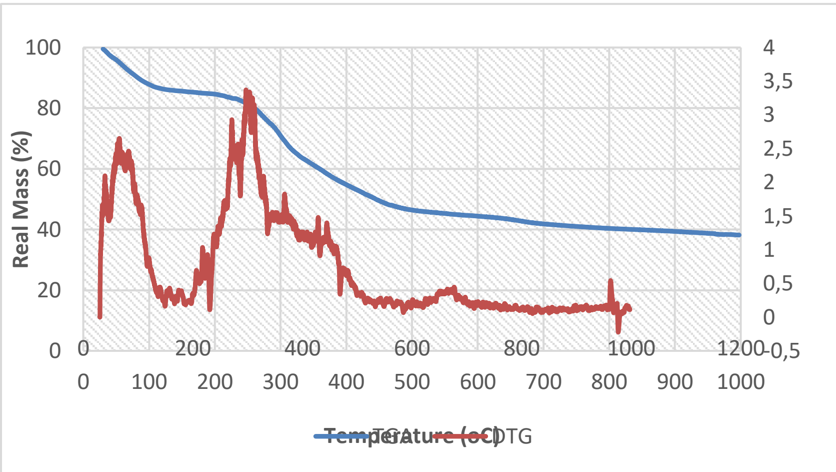

Figure 1. Analysis of Patchouli After Distillation Based on TGA

Figure 1 shows the change in weight indicating that after the distillation started, patchouli decomposed at a temperature of 50 °C.. The shrinkage of the polymer mass occurred due to the release of hydrogen atoms from the polymer hydrocarbon bonds. The release of hydrogen atoms is caused by the input energy that comes from heat. The release of hydrogen from the hydrocarbon bonds will increase with increasing temperature so that the polymer mass will decrease over time.

In this curve, three peaks of the thermogravimetric curve were obtained from the research. The first peak ranged from 50-1000 °C, the second peak from 100-5000 °C, and the third from 620-7000 °C. The test was carried out at a temperature of 1000 °C with the sample of a particle sized 50 with an initial mass weight of 99.48%. At a temperature of 1000 °C, there was a decrease in mass reaching 87.79% due to the large amount of residual water content from the distillation process or the release of water content, resulting in a decrease in weight. The second peak occurred at a temperature of 200-5000C where there was a decrease in mass reaching 46.40% due to the devolatilization process of aromatic compounds that have not been dissolved during distillation which is characterized by a very sharp decrease in mass on the TGA curve and the highest peak at DTG curve. At this peak, decomposition of hemicellulose, cellulose and lignin also occurred. Hemicellulose decomposed at a temperature of 220-3200 °C, cellulose at temperature of 260-3500 °C, and lignin decomposed at 180-5000 °C. There was a decrease in mass reaching 46.40% at the third peak at a temperature of 620-7000 °C due to the carbonation process which was characterized by a decrease in mass which slowed down again and tended to be stable., At a temperature of 10000 °C with a time of 97.96 minutes and left a residual mass of 38.17%.

TGA Patchouli before Distillation

Figure 2 shows the change in weight indicating that before distillation, patchouli began to decompose at a temperature of 300 °C. On this curve, there are three peaks obtained from the thermogravimetric curve generated from the study. The first peak was around 90-1200 °C, the second peak was between 280-3500 °C and the third peak 620-700 °C. This test was carried out at a temperature of 10000 °C, the sample with a particle size of 50 with an initial mass of 98.74%. At a temperature of 1000 °C, there was a decrease in mass reaching 88.59% due to the large release of water content resulting in a decrease in weight. At a temperature of 280-3500 °C there was a decrease in mass at the second peak reaching 45.38% resulted from the decomposition of aromatic compounds such as saponins, flavoids, tannins, steroids and essential oils. The third peak occurred at a temperature of 620-7000 °C and a decrease in mass was 20.44% due to biomass degradation. At a temperature of 10000 °C with a time of 97.96 minutes and leaving a residue mass of 15.30%.

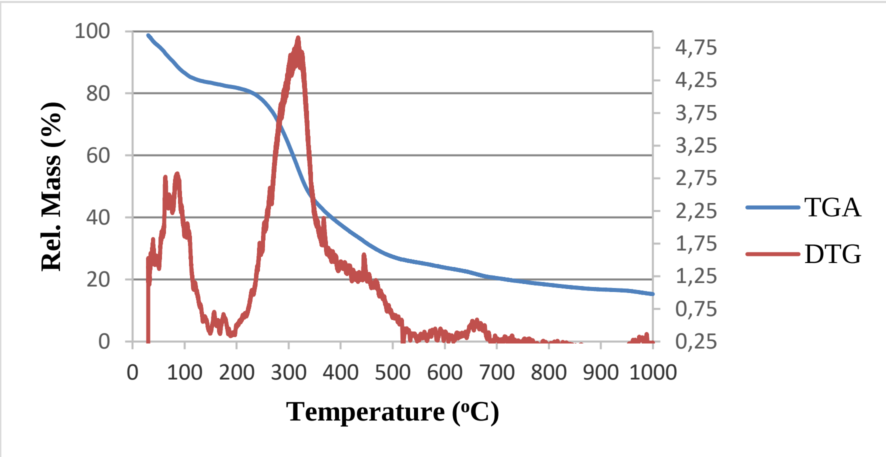

Figure 2. Analysis of Patchouli before Distillation based on TGA

The difference between these two curves is in the residual mass. The patchouli sample after distillation with a particle size of 50 mesh left a residual mass of 38.17%. Meanwhile, in the patchouli sample before distillation with a particle size of 50 mesh, the weight decreased by 15.30% at a temperature of 1000 °C with a time of 97.98 minutes. The amount of mass lost in patchouli before distillation is due to the large amount of decomposition of aromatic compounds.

DSC

Differential Scanning Calorimetry (DSC) is a thermal analysis technique where the difference in heat flow or heat power in the sample and standard (reference) is monitored against time or temperature, while the sample with the atmosphere has been programmed. By doing this analysis, it is hoped that it will be possible to measure changes in the difference in heat flow rates to materials (samples) and to reference materials when they are subject to a controlled temperature program. The results can be seen in Figure 3.

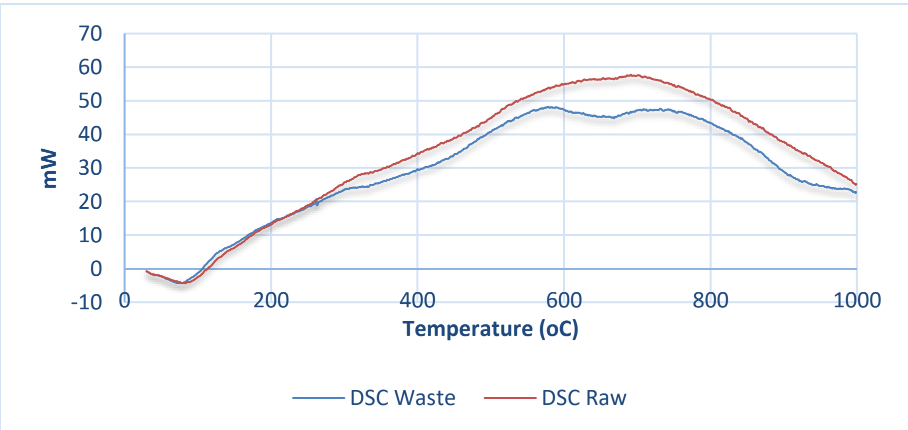

Figure 3. Comparison of Patchouli before and after Distillation Based on DSC

From the graph of the results of the DSC analysis in Figure 3, it can be seen that on patchouli after distillation, a drying stage occurred only at 0-1000 °C with the value of H was -122.83 J/g due to an exothermic reaction where at a temperature of 95.90 °C, energy is released. Meanwhile, on the patchouli before distillation, the drying started to occur at a temperature of 0-1000 °C and an exothermic reaction occurred at a temperature of 89.910 °C with a H value of -118.87 J/g. The second stage took place at a temperature of 100-7800 °C. Here, the degradation process of volatile substances such as saponins, flavoids and tannins took place. In patchouli before distillation, at a temperature of 742.70 °C there was a peak with a value of 47.43 mW,while in patchouli after distillation at a temperature of 679.5 and 694.90 °C there was a peak value of 57.11 and 57.46 mW. During the second stage of heating and continuation (100-7800 °C), the curve continued to rise. This shows that there was a significant weight loss of patchouli before and after distillation with a size of 50 mesh.

Conclusions

Based on the research that has been done, it can be concluded that from the results of the lignocellulose test, the patchouli component that produced the largest hemicellulose content of 27 % is twigs before distillation, the largest cellulose content of 35% was a mixture after distillation, and the largest lignin content in patchouli was contained in stems after distillation (42%). Based on the test results on patchouli components, the part that had the largest fixed carbon value is leaves before distillation (4.18%), while the largest calorific value contained in leaves before distillation (15649 kJ/kg). Patchouli after distillation had a large moisture content and ash content and patchouli before distillation contained a large volatile meter and fixed carbon. Potential processes that can be carried out on patchouli waste include direct combustion, fermentation and gasification.

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