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Influence of torrefaction on chemical compositions of empty fruit bunch (EFB) biomass using microwave heating

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ABSTRACT

Lignocellulosic biomass is inexpensive, most abundance and provide the large-scale. Lignocellulosic are compose into three structures which are cellulose, hemicellulose and lignin. Empty Fruit Bunch (EFB) has the highest composition of cellulose, hemicellulose and lignin among the abundance fiber like coir, corn, bagasse and kenaf fiber. Torrefaction was the process pre-treatment of biomass materials in inert atmosphere (nitrogen) in temperature range 200 to 300 °C by using microwave heating. Microwave controlled all the parameter which is power level (W), temperature (°C), volume of nitrogen (ml/min) and mass of sample (g) during the torrefaction process. In this study, the analysis of raw and torrefied EFB was done according to TAPPI standard method except hemicellulose which data was collected through equation. Result acquired reveals that the highest percentage in extractive, holocellulose, α- cellulose, hemicellulose and lignin was be found in raw EFB compared to other torrefaction EFB due to the degradation of content during torrefaction process. The degradation of hemicellulose was the takes place in temperature range 200 to 350 °C or even lower, whereas the degradation of cellulose and lignin occurs when 300 °C and above. This study determined EFB as useful alternative resources in feedstock material steam for power plant application.

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1. Introduction

With large-scale industrial development, the total exploitable of fossil fuel decline that are cause increasing of environmental pollution (Chen et al., 2011). In this study, there are more focus on the biomass energy that are one of the alternative way to reduce the air pollutant and greenhouse gasses that are due to high concentration CO_2 in the atmosphere cause by use of fossil fuel (Mohamed et al, 2015). Torrefaction is a thermochemical process which converts biomass into solid, liquid and gases fuel that will replace the using of fossil fuel (Huang et al., 2016). The process of torrefaction generally carried out in the 200-300 °C and to be used to upgrade lignocellulosic biomass to a higher quality fuel. Torrefaction has high potential to becoming a leader of pretreatment technology for exploitation of biomass for energy purpose (Peduzzi et al., 2014).

The oil palm (Elaeis guineensis sp.) fruit bunch is the one of the biomass energy and there generates a lot of lignocellulosic by product, mainly EFB, the fruit fiber and the shell. The types of biomass produce by oil palm are EFB, mesocarp fiber, kernel shells, fronds and trunk (Uemura et al., 2013). The EFB comprises of 17-33% of hemicellulose, 43-65% of cellulose and 13-37% lignin on the dry weight basic (Palamae et al., 2014). Lignocellulosic agriculture biomass consists of cellulose, hemicellulose and lignin, together with smaller amount of pectic substance. Cellulose acts as a major structural component in the plant cell that is for mechanical strength whereas the hemicelluloses macromolecule that is often repeated polymers of pentose and hexoses (Anwar et al., 2014). Then, the lignin contain three aromatic alcohol that are coniferyl alcohol, sinapyl alcohol and p-coumaryl alcohol that are produce by the process of biosynthetic process and

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form a protective seal to the both other component that are cellulose and hemicelluloses (Wang et al., 2015). Hemicellulose are starts to decompose for the chemical composition at the 200 to 250 °C and were totally degraded when torrefied temperature at 300 °C for 2 hours. Whereas, the cellulose and lignin began decompose about 270 to 300 °C. The tensile failure strength and strain energy are clear reduce due to the increasing temperature (Shang et al., 2012). Then, in this study, the effect of torrefaction towards compositions of hemicellulose, cellulose and lignin will be investigated.

2. Methodology

2.1. Empty fruit bunch (EFB) samples

EFB will get from oil palm (Elaeis guineensis sp.) fruit bunch at Felda Kemahang at Tanah Merah, Kelantan. The research handles in the material technology laboratory at Universiti Malaysia Kelantan Kampus Jeli, Kelantan.

2.2. Torrefaction experiment

Process of torrefaction started with the drying of sample to remove the moisture content using the oven drying method for 200 °C in 20 minutes. The samples will be grind to have specific size of particles. After that, the sample of raw EFB will be divided into two parts. One part are the raw sample and the others are undergoes the torrefaction process. Biomass material undergoes the thermochemical process that temperature in the range of 200 to 300 °C by using the microwave heating. Three parameters will be influence the final result of the torrefaction process that are power input selection at 385 Watt, various temperatures at 200 and 300 °C and different residence time (20, 40 and 60 mins).

2.3. Measurement

To analyze chemical composition analysis (Kassim et al., 2015) between raw and torrefied biomass materials, the Technical Association of Pulp and Paper Industry (TAPPI) Standard method are use. There are to identify the percentage of cellulose, hemicellulose and lignin in the biomass material.

2.3.1. Determination of extractive

The experiment will be carried out by using extraction method solubility in Alcohol-Benzene extractive following TAPPI Test Method T264 cm-97 (1997) (Hemmasi, 2012). This experiment started with approximately of 15 g sample of empty fruit bunch weighted.

Then, free thimble and free round bottom flask was weighted. The 5 g sample placed on thimble and the thimble placed at the soxhlet extraction. After that, the 300 ml of Ethanol - Benzene with the ratio (2:1) is place in the round bottom flask. The extraction is running for 6 until 8 hours in chemical fume hood. The extractive solution will be evaporate by using rotary evaporator in order to have only extractive. Then, the extractive will be obtained and put in the glass petri dish and the sample will be dry at 105 °C for 24 hours to get extractive. The calculations of extractive in the sample like below (Eq. 1).

$$\% \text{ extractive} = \frac{\text{Weight of extractive}}{\text{Weight of dry sample(g)}} \times 100 \quad (1)$$

2.3.2. Determination of holocellulose

Holocellulose content was determined by using Wise et al., 1946 method (Wang et al., 2012). In this experiment, 3 g of sample put into 250 ml conical flask and 100 ml of distilled water, 1.5 g sodium chloride crystals and 5 ml 10% acetic acid also added. Then, put the flask into 70 °C water bath. The experiment was conducted in fume cupboard. After 30 minutes, 5 ml 10% acetic acid added and after 30 more minutes, 1.5 g sodium chloride (NaCl) also added. For every 30 minutes, 1.5 g NaCl added which sums to 6 g, 4 hours duration. The sample heated for 30 minutes more. After that, the sample filtered with filter paper number four. Then, the sample washed with cold distilled water 500 ml, acetone 15 ml and left to dry in oven 60 °C for 24 hours. The equation used to determine the holocellulose was show below (Eq. 2).

$$\text{Holocellulose Content}(\%) = \frac{\text{Weight of holocellulose (g)}}{\text{Weight of oven dries (g)}} \times 100 \quad (2)$$

2.3.3. Determination of α-cellulose

The content of α-cellulose determined and compared. In this experiment, there carried out using TAPPI T203 os-74 (1997) method and handle in ice water bath (Hemmasi, 2012). Firstly, 1 g holocellulose mixed with 15 ml 17.5% NaOH in 250 ml beaker. Then, the solution stirred slowly for 1 minute, 10ml 17.5%, and after 45 seconds, the mixture added 10 ml more of 17.5% NaOH. The mixture is then left for 3 minutes and after 2.5 minutes, the mixture added 10 ml more of 17.5% NaOH, and at minute 5 and 7.5 will be added the same amount which sums to 65 ml of 17.5% used. Then, 100 ml of cold distilled water added and stirred quickly for 30 minutes. Next, the cellulose filtered with filter paper number 4. Beaker and leftover will be washed by using 25 ml 8.3 % NaOH. After that, the sample washed by using 250ml of distilled water and then soaked with 15ml of 10% acetic acid for 5 minutes. Samples then dried in 50 °C oven for 24 hours and finally weighted and recorded (Eq. 3).

$$\alpha - \text{ cellulose } (\%) = \frac{\text{Weight of } \alpha\text{-cellulose(g)}}{\text{Weight of oven dry holocellulose(g)}} \times 100 \quad (3)$$

2.3.4. Determination of hemicellulose content

Hemicellulose can be determined by using the following Eq. 4.

$$\text{Hemicellulose} = \text{Holocellulose} - \alpha - \text{cellulose} \quad (4)$$

2.3.5. Determination of lignin content

Lignin determination was carried out based on TAPPI T222 om-88 (2002) method (Saad and Ibrahim, 2014). Firstly, 1 g sample put into 150 ml beaker and placed in iced water bath. The 25 ml of 72% sulphuric acid (H_2SO_4) added and stirred with glass rod for every 10 minutes for 2 hours. Then, mixture transformed into 1 L conical flask that already contains 400 ml distilled water, and heated in hot plate at 170 °C with reflux for 4 hours. The sample cooled overnight and filtered and washed with hot 500 ml distilled water until pH level neutralized. Lastly, the sample dried until the weight is constant. The lignin contents will be determined by using the following equation.

$$\text{Lignin (\%)} = \frac{\text{Weight of Lignin (g)}}{\text{Weight of oven dry extractive-free (g)}} \times 100 \quad (5)$$

3. Results and discussion

3.1. Extractives

Extractive present in plants may be extracted using alcohol-organic solvent. After the drying and torrefaction process into the sample, the sample was subjected into the soxhlet extraction by using (2:1) ethanol: benzene to avoid present of lignin and polyphenolic compound in extractive-free. This extractive- free was needed into the next step that was to find holocellulose (Pelaez et al., 2014). Based on Fig. 1, extractive content is highest at raw EFB that are 22.65% compared to other torrefied sample. The lowest extractive percentage was sample (200 °C, 20 min) that is 14.20%. The trend of the graph above slightly increase from 200 to 300 °C, means that the extractive content produce after extraction process are high in 300 °C compare to 200 °C. The trend for minutes 20 was increased gradually when temperature increase compared to other 40 and 60 minutes. Observation recorded includes, the colour of the extractives obtained after the Soxhlet extraction process made during the study. There are showed darker colour for sample (300 °C, 20 minutes) which is more concentrated, compared to the other samples as extractive content are relatively second higher after raw EFB. As a conclusion the darker the colour of sample after extraction process, the higher the concentrated of extractive product.

3.2. Holocellulose

Chemically, holocellulose is the mixture of cellulose and hemicellulose in EFB fiber, the fibrous residue that remain after the extractive, the lignin, and the ash forming element have been removed (Yaman and Kucukbayrak, 2010).

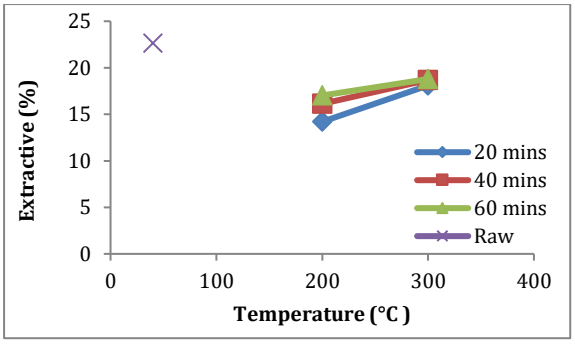

Fig. 1: Effect temperature on percentage of extractive

Holocellulose consist of the highest percentage of chemical content among the others composition in EFB which is 66.97% (Sudiyani et al., 2010). In this study, holocellulose in raw sample recorded the highest percentage content that is 62.08% as shown in Fig. 2. The holocellulose from raw EFB consist of 36.30% of α-cellulose and 25.78% of hemicellulose. The percentage of holocellulose obtain by this study are close with previous study by Ferrer et al. (2011) that come out with EFB consist of 56.49% as the total biomass. There are consist 33.25% of α-cellulose and 22.24% of hemicellulose (Sudiyani et al., 2010). It also proves that the holocellulose of raw EFB was 66.97% that also strongly supports the result obtain in this study. The lowest percentage of holocellulose was sample (200 °C, 20 min) that consists of 24.01% which comprise 20% of α-cellulose and 20.01% of hemicellulose. This degradation of holocellulose happen because the degradation of hemicellulose and the increase the percentage of cellulose obtained due to the rise of temperature in the raw and torrefied sample. The temperature and the times of torrefaction play a main role for final percentage of the holocellulose. The percentage of holocellulose starts to decrease when temperature increases and degradation decrease sharply for the minutes 60 compared to others 20 and 40 minutes. It was proved that the temperature and time of torrefaction process influenced the final percentage of holocellulose (Ferrer et al., 2011). It stated that the heating period 10 to 30 mins caused difficult to determine precise torrefaction time to perform torrefied sample. The samples not reach fully the torrefaction temperature in this period. It could report that the holding time will affect the amount of holocellulose to support the result obtain in this study.

3.3. α-cellulose

α-cellulose is the major component of wood and paper pulp. It was separated from the other components by soaking the pulp in a 17.5% solution of sodium hydroxide. One of three classes of cellulose, alpha cellulose has the highest degree of polymerization and is the most stable. The other two class, known as beta cellulose and gamma cellulose

(Rawangkul et al., 2010). Based on Fig. 3 shows the highest percentage of α-cellulose that are raw EFB with 36.30% whereas sample (200 °C, 20 min) shows the lowest percentage that are 20%. Cellulose was depleted at light torrefaction from 200-235 ˚C and mildly affected at severe torrefaction (275 to 300 °C) (Chen and Kuo, 2010).

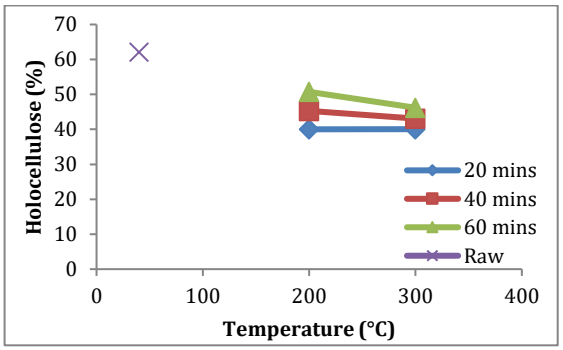

Fig. 2: Effect temperature and time on percentage of holocellulose

The increasing temperature lead to more cellulose consumed. These phenomena happen because of the disruption inside cell wall matrix including the connection between carbohydrate and lignin as well as depolymerization and solubilizing the hemicellulose leaving the α-cellulose content (Ramli et al., 2015). These finding are close to result yielded in this study that are state that the percentage of cellulose at temperature 200 °C show the lowest reading. After 200 °C, the percentage of cellulose started to increase until degradation temperature and the main decomposition of cellulose occur at 315-400 °C (Yaman and Kucukbayrak, 2010). The percentage of cellulose shows the highest at 300 °C. When the sample has longer treated by torrefaction, the percentage of the cellulose was also increase but the percentage of torrefied sample not high as raw EFB. The percentage of cellulose relatively highest at minutes 60 compared to other 20 and 40. This was proved that temperature and times influenced the final percentage of cellulose.

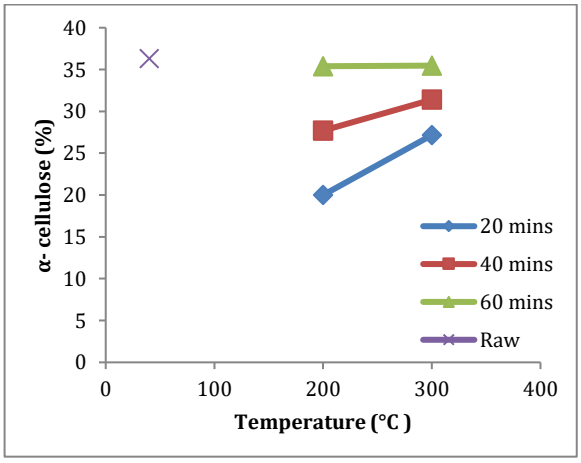

Fig. 3: Effect temperature and time on percentage of α-cellulose

3.4. Hemicellulose

In previous study by Silva et al. (2017), xylan or hemicellulose prepared by alkaline extraction was acidic polysaccharides containing arabinose (50.0%), xylose (38.5%) and uronic acid (9.0%) by using sodium hydroxide (NaOH) and followed by the alkaline filtrate. Hemicellulose plays a fundamental role in linking the fibers of cellulose to each other (Ramli et al., 2015). Based on observation on Fig. 5, the hemicellulose in raw EFB shows the highest percentage that are 25.78% where sample (300 °C, 60 min) shows the lowest reading that was 10.71%. Shen et al. (2015) proved that the degradation of hemicellulose takes place in temperature range 200-350 °C or even lower. These finding was close to the result obtained in this study. From Fig. 4, the percentage of hemicellulose was slightly dropped starting from 200°C. When the trend reached 300 °C, the hemicellulose content was totally low especially for minutes 60 that are from 15.32% at 200°C to 10.71% at 300 °C. The degree of hemicellulose degradation due the increase of time occurs because of the factor of the damage hydroxyl group in hemicellulose (Ferrer et al., 2011). The percentage of hemicellulose was nearly lost, especially for minutes 60 at 300°C due to degradation compared to minutes 20 and 40. This case happen because, the sample was precisely reaches the torrefaction temperature. In conclusion, minutes 60 were the best residence time to hemicellulose to fully reaches the torrefaction temperature and starts the rapidly degradation.

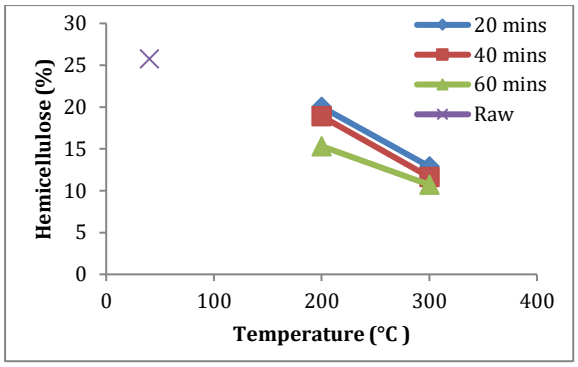

Fig. 4: Effect temperature and time on percentage of hemicellulose

3.5. Lignin

Lignin was particularly important in the formation cell wall and gives strength to the fibrils and polysaccharides, especially cellulose and hemicellulose (Ramli et al., 2015). The lignin was different between the types of wood. The content in EFB was 25.83% where the lignin content in the hard wood is 18-25% that are low compared to the non-woody plant (Ferrer et al., 2011). The high lignin content was the main reason of alkaline-pretreatment which was applied to EFB. By this pretreatment, the lignin will be removed and composition of lignin decreased. Chen et al. (2015) stated that when biomass is torrefied, the

pretreatment been categorize into light, mild and severe torrefaction, corresponding to the temperatures approximately 200-235 °C, 235-275 °C and 275-300 °C respectively. Lignin was most difficult to be thermally degraded because their decomposes was in much wider temperature range compared to the cellulose or hemicellulose (Chen and Kuo, 2010). Based on the Fig. 5, the highest percentage of lignin was in raw EFB that consist of 28% where the lowest value of lignin was for the sample (300 °C, 60 min) with 12.01%. The percentage of lignin has a nearly value for minutes 20, 40 and 60 at 200 °C and there were show that there are only small degradation happen at this temperature. The trend rapidly decreases when there are achieve the temperature 300 °C. Similar trend observable, there was proven that the degradation of lignin was started at 275 °C. Trend percentage of degradation of lignin can see clearly at temperature 300 °C in minutes 60 compare to other minutes. These finding are close to the previous research that prove that holding times more than 30 minutes are the better compare to the less than 30 minutes. From the observation, minutes 60 were the best for the lignin degradation. Lignin is difficult to degrade due to their complex structure that is heterogeneity. More specifically, lignin is complex composed of complicated phenypropane unit that are coniferyl alcohol, sinapyl alcohol and p-coumaryl alcohol are the one most commonly encountered (Popova et al., 2016). When the degradation of the lignin occurs, these chemical properties will breakdown into sugar breakdown product like furfural and 5-hydroxylfurfural.

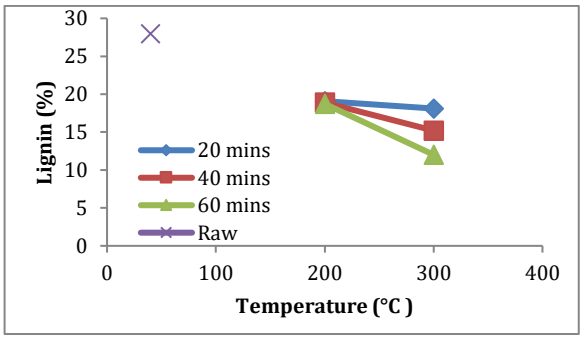

Fig. 5: Effect temperature and time on percentage of lignin

4. Conclusion

The data acquired from this study on comparison chemical properties in raw and torrefied Empty Fruit Bunch (EFB) is useful for the future research. The finding in this study determined that the torrefied EFB was a very useful resource for manufacturing the carbon (Ahmad et al., 2015) product use as feedstock material for steam power plant application. Both of objectives in this study were successfully achieved in this study that was able to produce the torrefied material using microwave heating (Ahmad et al., 2016). Microwave was success to control all the parameter during the torrefaction process that is 200 and 300 °C, residence time (20, 40 and 60 min) and at power level 385 W. Next, the chemical properties which are extractives, holocellulose, α-cellulose, hemicellulose and lignin of raw and torrefied EFB was successful analyzed by using TAPPI standard and equation. During torrefaction, the degradation of hemicellulose occurs in light torrefaction when the temperature reaches 200 °C and degradation of cellulose and lignin occurs in severe torrefaction at 300 °C. The best torrefaction sample that suitable to use to as feedstock material in steam power plant application was (300 °C, 60 min) due to the lowest lignin content, and highest holocellulose content. Lignin act as a binder between cellulose and hemicellulose and when the lignin was degraded, the structure of sample was brittle and will improve their grindibility. Besides that, the sample (300 °C, 60 min) contain the high mass loss and easy for transportation compared on raw EFB.

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