# Characterization of Cellulose from Microwave Extracted Mesocarp Fibre

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***Abstract*— This study was focused to determine and compare the cellulose, hemicellulose, lignin and extractive content of microwave assisted oil palm mesocarp fibre (OPMF). The highest cellulose content obtained is for OPMF is where power level of microwave used is 600 W with irradiation time for 6 minutes while the optimum power level that produced the highest cellulose content of 67.43% at constant power 300W is for 25 minutes. Hemicellulose content was reduced in the range of 30% to 40% compared to the untreated oil palm mesocarp fibre, which is 41.29%. The optimum pretreatment condition for microwave-assisted sterilization in removing optimim lignin content is at 400W for 30 minutes. Higher lignin content is not desirable as lignin acts as a barrier for any solutions by involving both hemicelluloses and cellulose. Only small amount of extractive content reduced in the range of 3% to 4%. The highest extractive content was reduced from 4.45% to 3.45% with 1:0.5 fruit to water is at 800W for 6 minutes. Higher amount of extractive also contributes to poor quality in chemical and mechanical pulping. Removal of extractive is effective with optimum temperature 300W microwave power and prolong irradiation time.**

***Keywords*— *oil palm mesocarp fibre, microwave, cellulose, hemicellulose, lignin, extractive***

## I. INTRODUCTION

Our public has been exceptionally reliant fossil fuels energy sources since the industrial revolution. In any case, the measure of accessible petroleum products worldwide is constrained and its extraction prompts ecological issues including the ozone depletion. Bio-based plant materials have various advantages, such as renewability, biodegradability, and environmental friendliness; therefore, they can be used as suitable substitutions for petroleum-based materials as a means of overcoming environmental problems (Kargarzadeh, 2017).

Cellulose can be considered as one of the most plentiful natural polymers can be found on the earth. It has a great deal of special properties such as biocompatibility, sustainability and biological degradability (Zhao *et al*., 2013). Other than having the biodegradable characteristics as advantage, cellulose fibrils in nanoscales sized which created from biomass is proven to be qualified as one of the new strengthening agent for polymer composites which can lead to producing possible lightweight and strengthens the composite (Cheng *et al*., 2009). This study was objected to determine and compare the cellulose, hemicellulose, lignin and extractive content of microwave assisted oil palm mesocarp fibre (OPMF).

## II. METHODOLOGY

### *A. Oil Palm Mesocarp Fibre (OPMF) Samples*

Oil palm mesocarp fiber was obtained from the oil palm fruit bunch which was acquired from plantation area in Selangor, Malaysia. Oil palm mesocarp fiber was obtained after the oil palm fruit bunch was cut and chopped into smaller pieces in order to facilitate the sterilization to disengage the activity of lipase enzyme. All the smaller pieces of the samples that have been chopped was kept in a dry condition before sterilization. The sample preparation of the oil palm mesocarp fiber was divided into two parts which is microwave assisted sterilization and cum extraction at constant oil palm mesocarp mass at 100g.

For the microwave-assisted sterilization, the paramaters are the ratio, microwave power and the irradiation time on the samples. The selected spikelet were selected and weighed before the sterilization process. Microwave oven (Panasonic model: 1000W frequency, 2450 MHz) was used with the time interval of 2 minutes irradiation time to the microwave power of; which was 4 minutes (800W), 6 minutes (600W) and 8 minutes (400W) for the ratio of fruit to water volume of 1:0. For the ratio of fruit to water volume of 1:0.5, 6 minutes irradiation time was chosen with 800W microwave power.

Meanwhile, for the cum extraction method, different microwave power output was used besides varying the extraction time. The microwave power of 100W, 300W, 400W and 600W were used for the 30 minutes time of extraction. As for the 300W microwave power, the extraction time were varied starting from 20 minutes, 25 minutes, 35 minutes and 40 minutes.

### *B. Measurement*

To analysis the chemical composition between the untreated and treated oil palm mesocarp fibre, the Technical Association of Pulp and Paper Industry (TAPPI) Standard method are used. These methods used are to identify the percentage of the cellulose, hemicellulose, lignin and extractive in the OPMF samples.

### *C. Determination of Cellulose and Hemicellulose*

The determination of cellulose content in the mesocarp fiber from microwave extracted was done according to the TAPPI method test; T 203 cm-99: Alpha-, beta- and gamma- cellulose in pulp. 1.5 g of sample and was placed in a beaker and mixed with 75 mL of 17.5% Sodium hydroxide (NaOH) reagent and stirred. The pulp suspension was stirred using a rod and placed in a water bath at temperature of 25$^{o}$C. After 30 minutes, 15mL of the filtrate was discarded and 100mL of the filtrate was collected in a clean dry filtration flask.

Cellulose determination was done by pipetting 25 mL of the filtrate and 10 mL of 0.5N potassium dichromate ($K_2Cr_2O_7$) and mixed with 50 mL of concentrated sulphuric acid ($H_2SO_4$). After the solution remained hot for 15 minutes, 50 mL of water was added and the solution was cooled to room temperature. 3 drops of

Ferroin indicator was added and the solution was titrated with 0.1N Ferrous Ammonium Sulfate solution to a purple color solution.

The determination of hemicellulose was done by pipetting 50mL of the filtrate into a 100mL graduated cylinder with ground glass stopper and mixed thoroughly with 50 mL of 3N $H_2SO_4$. The cylinder was heated and submerged in a hot water bath at 80°C for 3 minutes. The precipitate in the cylinder was allowed to settle for several hours before being filtered to obtain a clear solution. 50mL of the clear solution obtained and 10mL of 0.5N $K_2Cr_2O_7$ was pipetted and 90mL of $H_2SO_4$ was added cautiously. After the solution remained hot for 15 minutes, 50 mL of water was added and the solution was cooled to room temperature. 3 drops of Ferroin indicator was added and the solution was titrated with 0.1N Ferrous Ammonium Sulfate solution to a purple color solution.

### *D. Determination of Lignin Content*

The determination of lignin content was done according to the Kappa number (K) method attuned with the Klason lignin method (Omar, 2016). 0.05 g weighed treated oil palm mesocarp fiber sample was mixed with 20 mL of 2M $H_2SO_4$ and 5 mL of 0.02M Potassium permanganate solution ($KMnO_4$). The solution was stirred for 5 minutes at 200rpm using a hotplate. The solution then filtered and the filtrate was analyzed by using UV-vis at wavelength of 546 nm. The absorbance of the sample was recorded as $A_c$. A blank sample consisted of $H_2SO_4$ and $KMnO_4$ solution without the sample was used as a controller and recorded as $A_o$.

### *E. Determination of Extractive Content*

10 g of sample was placed in the extraction thimble. The extraction flask was filled with 150mL ethanol-toluene mixture with the ratio of 1:2. The flask was connected to the extraction apparatus and the heaters were adjusted to ensure the boiling rate will cycle the sample specimen for 4 hours. The flask was removed from the apparatus after 4 hours, and the solvent in the apparatus was evaporated partially to 25mL.

The extract was transferred to a weighing dish by washing with small amount of fresh solvent. The contents and dish were dried in an oven at 105°C for 1 hour before being cooled and weighed. A blank determination also was done with the ethanol-benzene mixture.

## III. RESULTS AND DISCUSSION

### *A. Cellulose and Hemicellulose Content*

Table 1 shows that the microwave assisted sterilization method have a significant effect on the cellulose content in the oil palm mesocarp fibre. Based Table 1, the untreated oil palm mesocarp fibre contains 58.71%. The cellulose content of treated oil palm mesocarp fibre slightly increased compared to the untreated oil palm mesocarp fibre. Similar result identified in the work of Long, 2015. It indicated that the microwave pretreatment was efficient in increasing cellulose content. Comparable findings also reported in Binod *et al*., 2015; Zhu *et al*., 2005 where cellulose content increased by using microwave pretreatment method. The highest cellulose content obtained is for sample 03 where power level of microwave used is 600 W with irradiation time for 6 minutes.

It was observed in Table 1 that the hemicellulose content was reduced in the range of 30% to 40% compared to the untreated oil palm mesocarp fibre which is 41.29%. According to Lai & Idris, 2013, reduction of hemicellulose content was triggered by the capability of microwave-irradiation, which lead to depolymerization of hetero-polysaccharides into oligosachharides. In work reported by Keshwani & Cheng, 2009, hemicellulose content was reduced as the temperature increased.

Table 1: OPMF Samples, parameters and cellulose, hemicellulose, lignin and extractive content obtained

| Sample | Ratio (m:v) | Microwave Power (Watt) | Irradiation Time (minute) | Cellulose % | Hemicellulose % | Lignin % | Extractive % |
|---|---|---|---|---|---|---|---|
| **1** | Untreated Oil Palm Mesocarp | | | 58.71 | 41.29 | 14.20 | 4.45 |
| **2** | 1:0 | 400 | 8 | 64.83 | 35.16 | 5.98 | 3.98 |
| **3** | | 600 | 6 | 67.37 | 32.62 | 4.47 | 3.83 |
| **4** | | 800 | 4 | 65.54 | 34.45 | 5.18 | 3.57 |
| **5** | 1:0.5 | 800 | 6 | 62.00 | 37.99 | 4.80 | 3.45 |

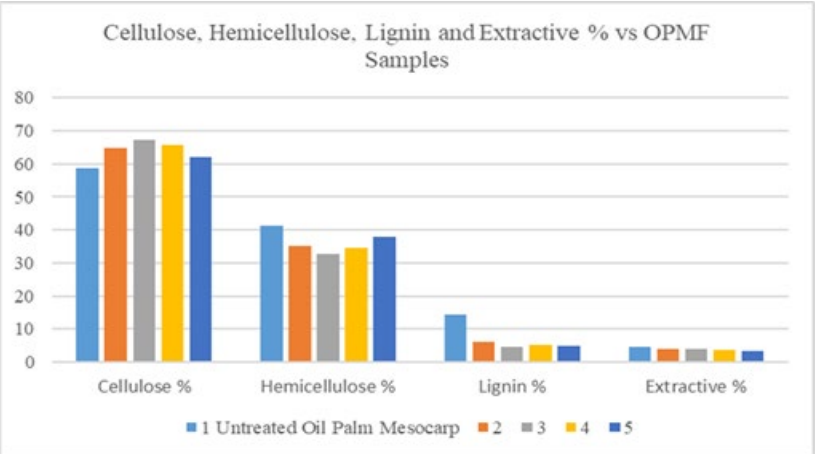


Fig 1: Comparison of cellulose, hemicellulose, lignin and extractive content

Similar finding was found by Nor, 2012, as the untreated oil palm fiber consist of 66.74% of cellulose and did not show significant difference after pretreatment process which was only 66.74%. Microwave-irradiation was proven to alter the structure of hemicellulose or lignin, thus destroyed them which lead to accessible area for hydrolysis caused by reduced cellulose crystallinity (Nomanbhay *et al*., 2013).

Mosier *et al*., 2005 achieved 24.5% for hemicellulose with the optimum microwave-irradiation condition was at 180W for 12

minutes. The effect between microwave power and irradiation time plays significant role in influencing the lignocellulosic materials digestibility by increasing hemicellulose removal (Chen *et al*., 2012; Kabel *et al*., 2007). According to Kabel *et al*., 2007, higher microwave power with prolonged exposure time may prompted the biomass digestibility as higher microwave power and longer irradiation time may lead decomposition of the oil palm mesocarp fibre whcich caused by the exposure to high temperature.

From Table 2, it can be shown that higher microwave power level at constant irradiation time caused the decreasing in cellulose content. The optimum power level that produced the highest cellulose content of 67.43% is at 300W for 25 minutes. Sabil *et al*., 2013, stated that the oil palm mesocarp fibre begin to breakdown and decompose at 150ºC and it was observed that critical decomposition of oil palm mesocarp fibre happens on the inner structural. Besides that, in previous research stated that by increasing the microwave power level, the time for microwave-irradiation pretreatment time can be reduced along with some restrictions. Degree of polymerization of cellulose deteriorates with higher temperature. Treated oil palm mesocarp fibre contain higher amount of cellulose and lower amount of lignin content compared to untreated mesocarp fibre. Higher amount of lignin loss will contribute to higher loss of hemicellulose content (Nor, 2012)

### *B. Lignin Content*

It can be observed that the lignin content of untreated OPMF is 14.2069. Similar findings found in past work on Shinoj *et al*., 2011, which 13-25% lignin content. Oil palm mesocarp fibre went through microwave assisted sterilization method with the ratio to water volume of 1:0, the highest lignin degradation is at 6 minutes with 600W of microwave power which is 68.48%. Lignin degradation by weight percentage of the sample with irradiation time of 4 minutes and 800W power has the lowest percentage of 35.97%. This might be from the higher temperature of the microwave power, which denatures the composition of the fibre.

Similar findings by Chen *et al*., 2012, where about 46% lignin was successfully removed after the fibre exposed to microwave pretreatment method. The lignin content of the oil palm mesocarp fibre after subjected to microwave pretreatment is in range of lignin content similar to past research by Sun, 1999 where lignin content of fibre found to be in the range from 0.9% to 6.6%.

Table 2 shows that with the extraction time of 30 minutes, the optimum pretreatment condition in removing lignin content is at 300W. Meanwhile, with the time variation of extraction time with constant microwave power at 300W, the optimum condition for the highest lignin degradation is sample 10 which is for 20 minutes.

Higher lignin content is not desirable as lignin acts as a barrier for any solutions by involving both hemicelluloses and cellulose. It is crucial to free the cellulose from the lignin and at the same time to reduce lignin content in order to increase cellulose porosity. Microwave irradiation has proven in changing the ultrastructure of cellulose and hemicellulose, besides aids in lignin degradation in lignocellulose materials (Xiong *et al*.,2002; Hu & Wen, 2008).

The lignin degradation of the OPMF which is in the range of 40% to 50%, is similar to *Chen et al*., 2012. It was reported that as the temperature increases, it was observed that more than 40% successfully removed. Lignin degradation is the measure of effectiveness of pretreatment used. Based on the results obtained, the optimum pretreatment condition for microwave-assisted sterilization in removing optimum lignin content is at 400W for 30 minutes.

Table2: OPMF Samples, parameters and cellulose, hemicellulose, lignin and extractive content obtained

| Sample | Microwave Power (Watt) | ExtractionTime (minute) | Cellulose % | Hemicellulose % | Lignin % | Extractive % |
|---|---|---|---|---|---|---|
| **6** | 100 | 30 | 61.44 | 38.55 | 10.10 | 4.17 |
| **7** | 300 | | 67.12 | 32.88 | 8.25 | 3.81 |
| **8** | 400 | | 66.83 | 33.16 | 8.73 | 3.96 |
| **9** | 600 | | 64.38 | 35.62 | 8.58 | 3.99 |
| **10** | 300 | 20 | 65.91 | 34.08 | 8.55 | 3.87 |
| **11** | | 25 | 67.43 | 32.56 | 8.52 | 3.78 |
| **12** | | 35 | 67.13 | 32.87 | 8.24 | 3.71 |
| **13** | | 40 | 66.85 | 33.14 | 8.27 | 3.36 |

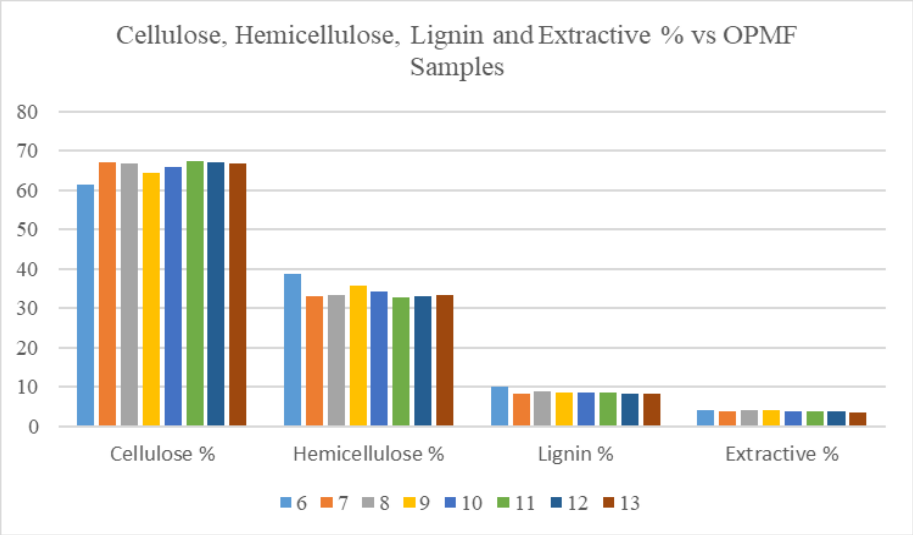


Figure 2: Cellulose, Hemicellulose, Lignin and Extractive Content

Table 2 shows that the increase of heating time affected the loss of lignin from 20 min to 40 min and there was no significant effect of heating time after 35 minutes pretreatment.

### *C.. Extractive Content*

Table 2 shows the extractive content obtained for untreated and treated oil palm mesocarp fibre at different ratio of oil palm fibre with water, microwave power and irradiation time. Based on the table, it was found that the extractive content of untreated oil palm

mesocarp fibre is 4.45% similar to findings by Abdul Khalil *et al*., 2008. Based on Figure 2, only small amount of extractive content reduced in the range of 3% to 4%. The highest extractive content removal from the oil palm mesocarp fibre with 1:0.5 fruit to water is at 800W for 6 minutes. The extractive content reduced from 4.45% to 3.45%. The trend has shown a relationship whereas the microwave of the power increases and the irradiation time reduce, the higher extractive content is being removed.

Based on Table 2, it can be shown that the extractive content is slightly reduced. Sample 6 has the highest extractive content which is 4.17%. This may caused by low power level of microwave of only 100W. Higher amount of extractive content in any lignocellulosic materials are undesirable because its ability to obstruct the formation of microbes in early stage of cloning hence reducing the ability of oil palm fibre to engage and attain water molecules via absorption or adsorption (Bruce, 1998). Furthermore, higher amount of extractive also contributes to poor quality in chemical and mechanical pulping. Sample 13 has the lowest extractive content, which is 3.36% microwave power at 300W for 40 minutes. Removal of extractive is effective with optimum temperature 300W microwave power and prolong irradiation time as the extractive content is decreasing from 3.87% for sample 10 to 3.36% with longer irradiation time.

## IV. Conclusion

Utilization of microwave-irradiation has proven to be effective in pretreatment method of OPMF with its capability to reduce lignin and hemicellulose content, hence increasing cellulose content, which is desirable. Microwave-irradiation pretreatment is possible to speed up the rate of reaction thus reducing the reaction time and energy consumption due to improved configuration of molecules, which lead to enhanced reactivity and lower activation energy. The highest cellulose content obtained is for OPMF is where power level of microwave used is 600 W with irradiation time for 6 minutes while the optimum power level that produced the highest cellulose content of 67.43% at constant power 300W is for 25 minutes. Hemicellulose content was reduced in the range of 30% to 40% compared to the untreated oil palm mesocarp fibre which is 41.29%. The optimum pretreatment condition for microwave-assisted sterilization in removing optimim lignin content is at 400W for 30 minutes. Higher lignin content is not desirable as lignin acts as a barrier for any solutions by involving both hemicelluloses and cellulose. Only small amount of extractive content reduced in the range of 3% to 4%. The highest extractive content is reduced from 4.45% to 3.45% with 1:0.5 fruit to water is at 800W for 6 minutes. Higher amount of extractive also contributes to poor quality in chemical and mechanical pulping. Removal of extractive is effective with optimum 300W microwave power and prolong irradiation time. The morphological analysis on the OPMF can be done for future research.

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