

Isolation of Cellulose from Oil Palm Solid Waste

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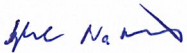
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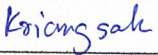
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Abstract

Recently, palm oil production scale has been remarkably increased due to high demand of biodiesel as renewable fuel. As a result, the amount of solid waste from oil palm tree such as palm frond (PF), empty fruit bunch (EFB), and palm pressed fiber (PPF) has been significantly increased. Since this solid waste mainly consists of cellulose, hemicellulose, and lignin, it could be considered as a resource of cellulosic materials. In this study, a simple method consisting of three consecutive chemical treatments to remove wax, lignin, and hemicellulose was used to isolate cellulose from this waste. Efficiency of each step was evaluated from its yield and functional groups of the obtained cellulose was observed. The results showed that lignin and hemicellulose were completely removed from PF by the series of chemical treatments whereas additional physical separation was necessary for the isolation of cellulose from EFB and PPF. The yields of cellulose from PF, EFB, and PPF were 27.1%, 23.5%, and 15.7%, respectively. Furthermore, based on fourier transform infrared spectroscopy (FT-IR) spectrums, all the isolated solids showed characteristics of functional groups (O-H and C-H) without the characteristics of functional groups of lignin (C=C) and hemicellulose (C=O). This result confirmed that the cellulose isolated from PF, EFB, and PPF has high purity. Therefore, PF-, EFB-, and PPF-cellulose obtained from this simple procedure should have high potential for food and pharmaceutical applications.

Keywords: Solid waste, Oil Palm, Cellulose

เรื่อง	การแยกเซลลูโลสจากของแข็งเหลือทิ้งจากต้นปาล์มน้ำมัน
โดย	นาย สราวุฒิ สิ้นพิชัย
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สาขาวิชา	วิศวกรรมปิโตรเคมี
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บทคัดย่อ

ปัจจุบันกระบวนการผลิตน้ำมันปาล์มมีกำลังการผลิตเพิ่มขึ้นอย่างเห็นได้ชัด เนื่องจากมีความต้องการที่เพิ่มขึ้นในการใช้ไบโอดีเซลเป็นพลังงานทดแทน ด้วยเหตุนี้ปริมาณของแข็งเหลือทิ้งจากต้นปาล์มน้ำมัน เช่น ทางปาล์ม, ทะลายปาล์ม, และเส้นใยปาล์ม จึงมีปริมาณเพิ่มขึ้นอย่างมีนัยสำคัญ โดยของแข็งเหลือทิ้งดังกล่าวมีส่วนประกอบสำคัญคือ เซลลูโลส, เฮมิเซลลูโลส, และลิกนิน จึงถูกพิจารณาเป็นทรัพยากรในการผลิตวัสดุเซลลูโลส ในการศึกษานี้ได้ใช้วิธีแบบง่ายซึ่งประกอบด้วย 3 ขั้นตอนในการใช้สารเคมีเพื่อกำจัด แวกซ์, ลิกนิน, และเฮมิเซลลูโลส ตามลำดับ โดยวิธีนี้ถูกใช้เพื่อทำการแยกเซลลูโลสออกจากของแข็งเหลือทิ้งดังกล่าว ประสิทธิภาพของแต่ละขั้นตอนได้รับการประเมินจากเปอร์เซ็นต์ผลผลิตของแต่ละขั้นตอนและกลุ่มฟังก์ชันของเซลลูโลสที่แยกออกมาได้ จากผลลัพธ์แสดงให้เห็นว่า ลิกนินและเฮมิเซลลูโลสได้ถูกกำจัดอย่างสมบูรณ์จากทางปาล์มโดยการใช้สารเคมีตามลำดับขั้นตอน แต่ทว่า จำเป็นต้องมีการแยกทางกายภาพเพิ่มเติมสำหรับการแยกเซลลูโลสออกจากทะลายปาล์มและเส้นใยปาล์ม เปอร์เซ็นต์ผลผลิตของเซลลูโลสที่ได้จากทางปาล์ม, ทะลายปาล์ม, และเส้นใยปาล์ม คือ 27.1%, 23.5%, และ 15.7% ตามลำดับ นอกจากนี้โดยอ้างอิงจากเส้นฟูเรียรทรานสฟอร์มอินฟราเรดสเปกโตรสโกปีสเปกตรัมจะพบว่าของแข็งที่แยกออกมาได้แสดงกลุ่มฟังก์ชัน O-H และ C-H โดยไม่มีกลุ่มฟังก์ชันของ ลิกนิน (C=C) และเฮมิเซลลูโลส (C=O) จากผลการทดลองนี้แสดงให้เห็นว่าเซลลูโลสที่แยกได้จากทางปาล์ม, ทะลายปาล์ม, และเส้นใยปาล์ม มีความบริสุทธิ์สูง ดังนั้นเซลลูโลสจากทางปาล์ม, ทะลายปาล์ม, และเส้นใยปาล์มที่ได้รับจากวิธีแบบง่ายนี้ น่าจะมีศักยภาพสูงสำหรับการนำไปใช้ทางด้านอาหารและยา

คำสำคัญ: ของแข็งเหลือทิ้ง, ต้นปาล์มน้ำมัน, เซลลูโลส

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NOMENCLATURE

W_0	dry basis weight of sample before reaction	g
W_1	dry basis weight of sample after reaction	g

CHAPTER I

INTRODUCTION

1.1 Background

In the year 2014, in Thailand, oil palm (17% oil) was harvested around 12.50 million tons and it was used to produce around 2.00 million tons of crude palm oil (CPO). 42.1% and 51.4% of CPO were used in production of biodiesel and vegetable oil, respectively. Large amount of solid waste, approximately 10.38 million tons, was generated from CPO production process [1]. The proportions of empty fruit bunches (EFB) and palm pressed fiber (PPF) are 22% and 18%, respectively [2]. Palm frond (PF) is also solid waste from palm oil production process since PF will be generated together with every harvest of EFB.

Solid waste from palm oil production process are useful. EFB can be used for mushroom cultivation and combustion to produce in steam generation in palm oil mills [2, 3]. PPF can be used to produce both steam and electrical power in factories. PF is usually left on the ground to decompose and fertilize the soil [4].

Cellulose has interesting properties such as hydrophilicity, non-toxicity, facile chemical modification, potential as a sorbent, and good mechanical properties [5]. Therefore, cellulose has been used in various fields such as pharmaceutical, food, and polymer composites industries [6]. Cellulose can be isolated from biomass such as aquatic weed [7], rice straw [8], corn cob [9], and sugarcane bagasse [10]. Aquatic weed can be used to produce cellulose, although the amount of cellulose is low, around 25%. Amount of cellulose in PF, EFB, and PPF are 62.34% [11], 23.7%-65.0% [3], and 17.2%-29.2% [12], respectively. Then, PF, EFB, and PPF have chance to be used to produce cellulose.

This project aims to isolate cellulose from oil palm solid waste in order to evaluate possibility of using PF, EFB, and PPF as resource of cellulose. A simple method previously applied by Lu and Hsieh [8] to isolate cellulose from rice straw was used in this study.

1.2 Objectives

1.2.1 To isolate cellulose from palm frond (PF), empty fruit bunch (EFB), and palm pressed fiber (PPF).

1.2.2 To compare efficiencies of the cellulose isolation from PF, EFB, and PPF.

1.3 Scope of Work

PF, EFB, and PPF obtained from small scale mills. Cellulose isolated by three consecutive chemical treatments to remove wax, lignin, and hemicellulose. Yield of each step and overall yield were evaluated. The purity of isolated cellulose were determined by FT-IR spectrum.

1.4 Expected Output

Alternative method for utilization of oil palm solid waste as a resource of cellulosic materials.

CHAPTER II

LITERATURE REVIEW

2.1 Palm oil production process

Palm oil mill (Figure 2.1), fresh fruit bunches (FFB) are sterilized in step 1. Then, the palm oil fruits are stripped from FFB. Palm oil fruits are digested to make them easily to press in pressing machine to obtain palm oil from mesocarp, in step 2. Nut is separated from pressed cake by cyclone for using in production of palm kernel oil, in step 3.

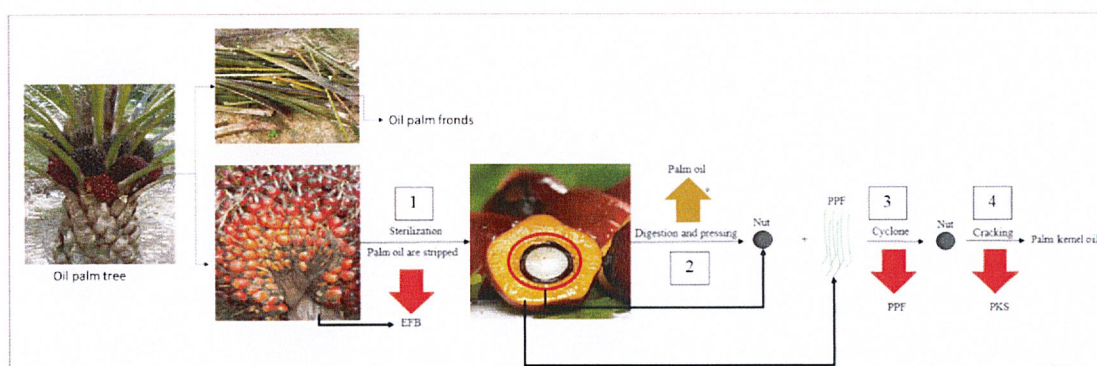


Figure 2.1 Palm oil production process.

2.1.1 Solid waste

PF is first solid residue from palm oil production process because of falling out of the oil palm tree. (PF contain 62.34%, 24.19%, and 14.81% of cellulose, hemicellulose, and lignin, respectively [11].)

Sterilization method, in step 1, after palm oil fruits are stripped from FFB, the process generated empty fruit bunch (EFB) around 20-30% of FFB. Moisture content of EFB is 12-14% [2]. (EFB contains 23.7-65.0%, 20.6-33.5%, and 14.1-30.5% of cellulose, hemicellulose, and lignin, respectively [3].)

Cyclone method, in step 3, nut is separated from pressed cake, the remaining is mesocarp of fruit or palm pressed fiber (PPF) around 12-13% of FFB [2]. (PPF contain 17.2-29.2%, 23.4-23.7%, and 33.3-41.0% of cellulose, hemicellulose, and lignin, respectively [12].)

2.1.2 Composition of solid waste

The chemical composition of PF, EFB, and PPF are summarized in table 2.1. The lignin content of PPF, and EFB are high. The amount of cellulose from solid waste has enough potential to produce cellulose.

Table 2.1 Chemical composition of waste (dry basis).

Biomass	Cellulose, %	Hemicellulose, %	Lignin, %	Extractive, %	References
PF	62.34	24.19	14.81	1.8	[11]
EFB	23.7-65.0	20.6-33.5	14.1-30.5	3.2-3.7	[3]
PPF	17.2-29.2	23.4-23.7	33.3-41.0	10.5-11.0	[12]

2.2 Cellulose

Cellulose is a linear carbohydrate polymer that made from repeating of β -(1 \rightarrow 4)-linked D-anhydroglucopyranose as shown in figure 1. The cellulose chain are bundled together to form cellulose microfibrils. The cellulose microfibrils are bundled together to form cellulose fiber.

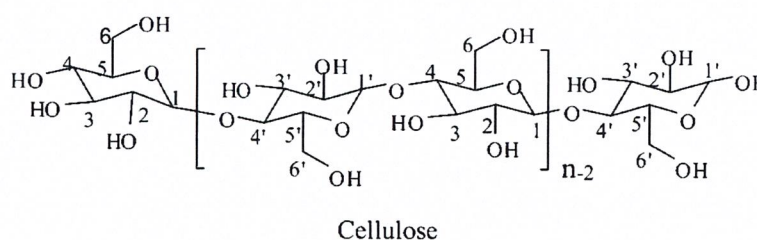


Figure 2.2 Structure of cellulose

There are two route to synthesize cellulose, top-down and bottom-up approach. The top-down approach is the process to produce cellulose from plant or agricultural waste. On the other hand, bottom-up approach is the process to produce cellulose from glucose using bacteria (e.g. *Acetobacter Xylinam*).

2.2.1 Top-down approach

Top-down approach is process to isolate cellulose from lignocellulosic materials by pretreatment and pulping processes. In these process, consist of drying, grinding, dewaxing, purification, delignification, bleaching, filtration, washing, and drying, respectively. Dewaxing, delignification and bleaching are main step used to separate wax, lignin and hemicellulose from biomass.

2.2.2 Application of cellulose

Since cellulose has excellent properties such as hydrophilicity, non-toxicity, facile chemical modification, potential as a sorbent, and good mechanical properties [6], cellulose can be used in various application such as food, pharmaceutical, and adsorbent [5].

Hydrogel made from cellulose and poly vinyl alcohol, hydrogel shows the water absorption capacity of 141.6-392.1% and high opacity [13].

2.3 Related researchs

Sun, J.X. et al [14] study the isolation and characterization of cellulose from sugarcane bagasse by using 3 different procedures. The first method was isolation of cellulose by using mixture of sodium hydroxide (NaOH) and hydrogen peroxide (H_2O_2). The second method was isolation of cellulose by delignification process using acidified sodium chlorite ($NaClO_2$). And last process was isolation of cellulose by using mixture of acetic acid (CH_3COOH) and nitric acid (HNO_3). The yield of cellulose from mixture of NaOH and H_2O_2 , $NaClO_2$, and mixture of CH_3COOH and HNO_3 were 45.9%, 44.2%, and 43.0-43.6%, respectively.

Lu, P. et al study the preparation and characterization of cellulose nanocrystals from rice straw. The pure cellulose isolated by de-waxing, de-lignification, and hemicellulose removing process. The yield of cellulose was 36%. Pure cellulose was used to synthesize cellulose nanocrystals (CNCs) by acid hydrolysis at 30 and 45 min. the CNC suspension were used to assemble into long fibrous structure by freeze-drying. The self-assembled fibers from CNC45 showed higher crystallinity and larger crystallite than self-assembled fibers from CNC30. Moreover, the self-assembled of CNC45 showed extraordinary structural stability, withstanding vigorous shaking and prolong stirring in water.

Oliveira, J. P. D. et al study cellulose fibers extracted from rice and oat husks and their application in hydrogel. Cellulose isolated from rice and oat husks were used to produce hydrogel with poly vinyl alcohol (PVA). The hydrogel from cellulose extracted from rice and oat husks showed water absorption capacity of 141.6-392.1% and high opacity. Rice cellulose hydrogel showed the highest absorption capacity at 25 °C. These results show that the use of agro-industrial residues is promising for the biomaterial field, especially in the preparation of hydrogels.

CHAPTER III

RESEARCH METHODOLOGY

3.1 Materials and Chemicals

Palm frond (PF), empty fruit bunch (EFB), and palm pressed fiber (PPF) were obtained from Krabi Palm Community Cooperative in south of Thailand. Commercial grade toluene ($C_6H_5CH_3$, 99.5%), ethanol (C_2H_5OH , 99.9%), sodium chlorite ($NaClO_2$, 25%), acetic acid glacial (CH_3COOH , 99.8%), and potassium hydroxide (KOH, 85%) were used for isolation of cellulose from biomass.

3.2 Preparation of cellulose from biomass

A simple method, consisting of pretreatment and isolation of cellulose were used to prepare cellulose from PF, EFB, and PPF. Detail of each step was shown in Figure 3.1 and 3.2.

In the pretreatment step, as shown in Figure 3.1, the obtained biomass was washed with tap water. After that, it was dried under sunlight for 1 day and further dried in an oven at 55 °C for 24 h. Then, the dried biomass was cut using a miller to obtained milled biomass (M-PF, M-EFB, and M-PPF) with consisted of powder and fiber with size in the range of 1-42 mm.

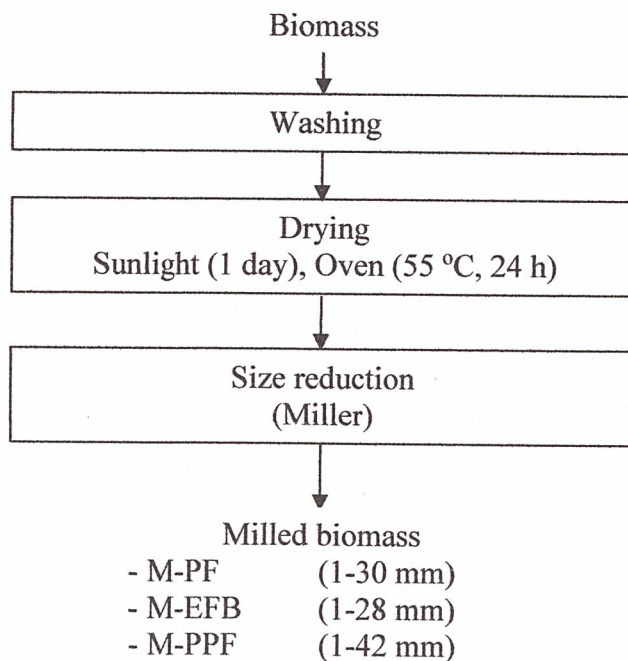


Figure 3.1 Pretreatment of biomass.

In the step to isolate cellulose, as shown in Figure 3.2, the obtained milled biomass was treated with toluene/ethanol solvent using soxhlet at 65 °C for 20 h in order to remove wax. Then, the solid obtained after wax removal was dried in an oven at 55 °C for 24 h. The dried solid after wax removal was called dewax-biomass (DW-PF, DW-EFB, and DW-PPF). The dewax-biomass was treated with 1.4% acidified sodium chlorite at 70 °C for 5 h in order to remove lignin. Then, the solid obtained after lignin removal was washed with distilled water until pH of washed water was 7. The washed solid after lignin removal was called delignin-biomass (DL-PF, DL-EFB, and DL-PPF). After that, the delignin-biomass was treated with 5% KOH at room temperature for 24 h and 90 °C for 2 h in order to remove hemicellulose. Then, the solid was washed with distilled water using vacuum filtration equipment until pH of washed water was 7. The solid cake on the filter paper was collected and stored in bottle. The solid cake was called dehemicellulose-biomass (DH-PF, DH-EFB, and DH-PPF).

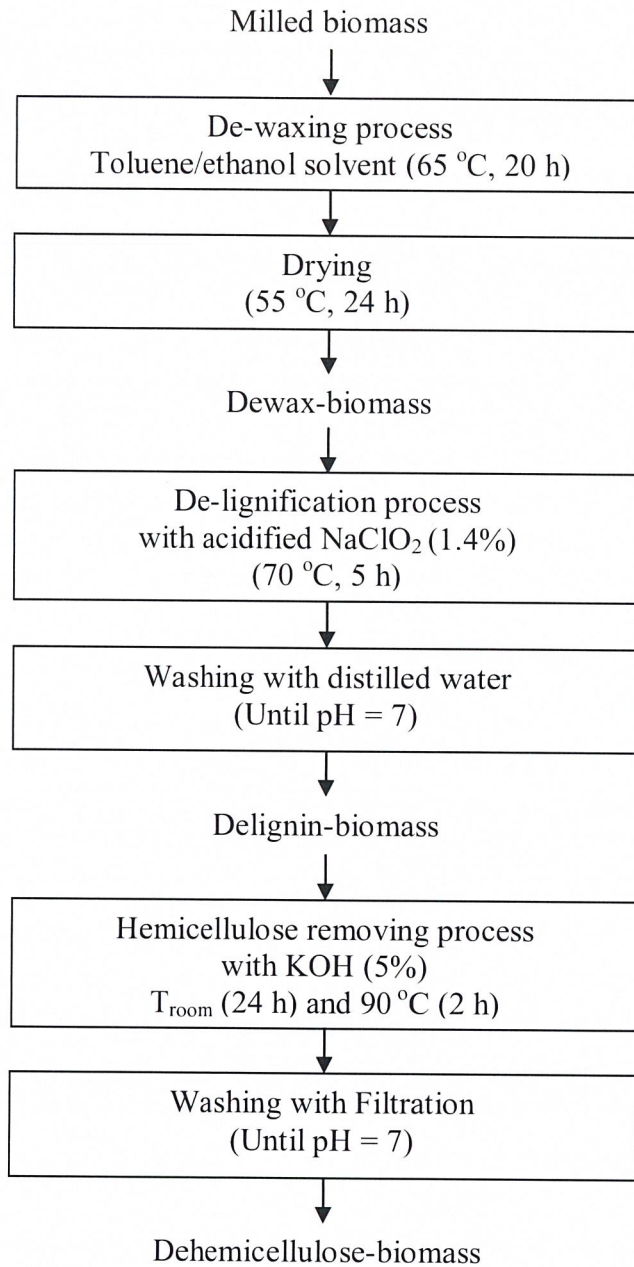


Figure3.2 Isolation of cellulose from milled biomass.

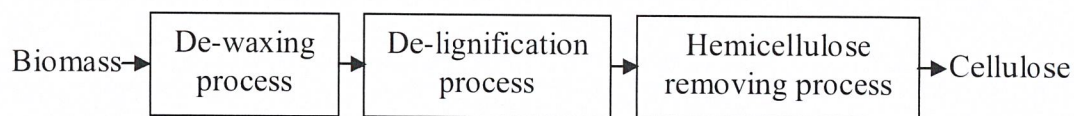


Figure3.3 steps to isolate cellulose from biomass.

3.3 Evaluation of process efficiency

To prepare cellulose from biomass, consist of 3 process as shown in figure 3.3. Yield of the de-waxing, delignification, and hemicellulose removing process were calculated from balance based on dry basis weight of sample at before (W_0) and after (W_1) reaction in each process according to equation 1.

$$\text{Yield} = W_1/W_0 \times 100\% \quad (1)$$

3.4 Characterization of cellulose

The physical appearance of biomass, dewax, delignin, and dehemicellulose were photographed by camera from iphone 6 with resolution of 8 mega pixel.

Functional group of the obtained dehemicellulose-biomass was observed by Fourier transform infrared spectroscopy (FT-IR) in the range of $4000-600 \text{ cm}^{-1}$ using a Shimadzu IRPrestige-21 fourier transform infrared spectrophotometer. The operation was ATR mode with number of scan of 128 and resolution of 4 cm^{-1} .

CHAPTER IV

RESULTS AND DISCUSSION

In the preparation of cellulose from biomass, PF, EFB, and PPF were removed wax, lignin, and hemicellulose, the samples were obtained from the series in preparation of cellulose were called dewax-biomass, delignin-biomass, and dehemicellulose-biomass, respectively. The change of appearance in each step and the characteristic of cellulose for dehemicellulose-biomass of PF, EFB, and PPF were discussed in section 4.1, 4.2, and 4.3.

4.1 Isolation of cellulose from PF

Cellulose was prepared by remove wax, lignin, and hemicellulose in de-waxing, de-lignification, and hemicellulose removing process, respectively. Figure 4.1.1 shows the change of appearance of the sample obtained after each treatment in the isolation of cellulose from PF. The color of sample have change, it changed from brown of PF to yellow of DL-PF due to the lignin was removed, and become white of DH-PF after the hemicellulose was removed (Fig. 4.1.1, (a)-(d)). The size of sample was gradually decreased from PF to DH-PF due to lignin and hemicellulose were removed from outer structure of lignocellulosic biomass (Fig. 4.1.1, (a)-(d)). The weight almost constant for de-waxing process. On the other hand, there are obvious change in weight for de-lignification and hemicellulose removing process. The yield of de-lignification and hemicellulose removing process were 60.0% and 45.2%, respectively.

The FTIR spectrum of DH-PF (Fig. 4.1.2) shown the characteristic peaks of cellulose at $3,434\text{ cm}^{-1}$, $2,897\text{ cm}^{-1}$, and 898 cm^{-1} from stretching of O-H, C-H, and Glycosidic-C₁-H, respectively [5]. Since no characteristic peak of lignin ($1,516\text{ cm}^{-1}$) and hemicellulose ($1,729\text{ cm}^{-1}$) was observed in the spectrum of DH-PF, it can be concluded that lignin and hemicellulose were completely removed from PF.

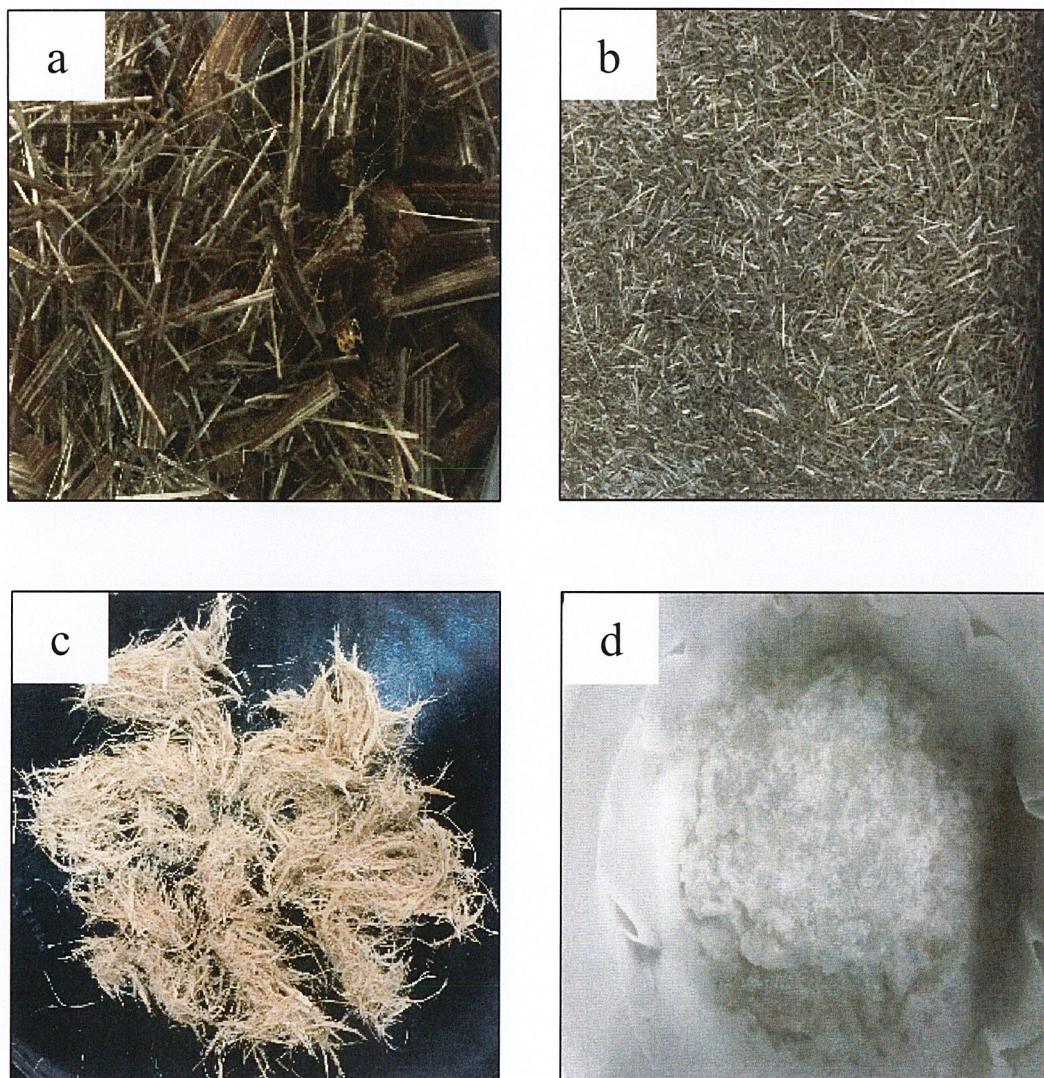


Figure 4.1.1 Physical appearance of PF (a), after remove wax (b), after remove lignin (c), and after remove hemicellulose (d).

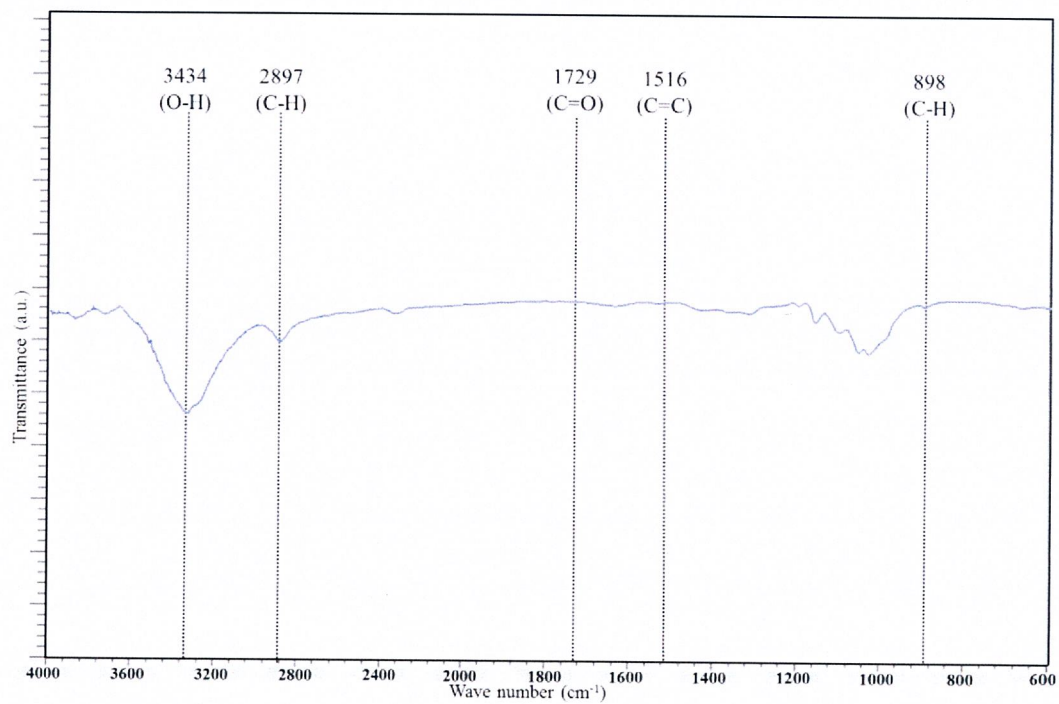


Figure 4.1.2 FTIR spectrum of cellulose from PF.

4.2 Isolation of cellulose from EFB

Cellulose was prepared by remove wax, partial lignin, and hemicellulose in de-waxing, de-lignification, and hemicellulose removing process, respectively. Figure 4.2.1 shows the change of appearance of the sample obtained after each treatment in the isolation of cellulose from EFB. The color of sample have change, it changed from brown of EFB to yellow of DL-EFB due to the lignin was removed, and become white of DH-EFB after the hemicellulose was removed (Fig. 4.2.1, (a)-(d)). The size of sample was gradually decreased from EFB to DH-EFB due to wax, lignin, and hemicellulose were removed from outer structure of lignocellulosic biomass (Fig. 4.2.1 (a)-(d)). There are obvious change in weight for de-waxing, de-lignification, and hemicellulose removing process. The yield of de-waxing, de-lignification, and hemicellulose removing process were 89.1%, 66.7%, and 45.2%, respectively.

The FTIR spectrum of DH-EFB (Fig. 4.2.2) shown the characteristic peaks of cellulose at $3,434\text{ cm}^{-1}$, $2,897\text{ cm}^{-1}$, and 898 cm^{-1} from stretching of O-H, C-H, and Glycosidic-C₁-H, respectively [5]. Since no characteristic peak of lignin ($1,516\text{ cm}^{-1}$) and hemicellulose ($1,729\text{ cm}^{-1}$) was observed in the spectrum of DH-EFB, it can be concluded that lignin and hemicellulose were completely removed from EFB.



Figure 4.2.1 Physical appearance of EFB (a), after remove wax (b), after remove lignin (c), and after remove hemicellulose (d).

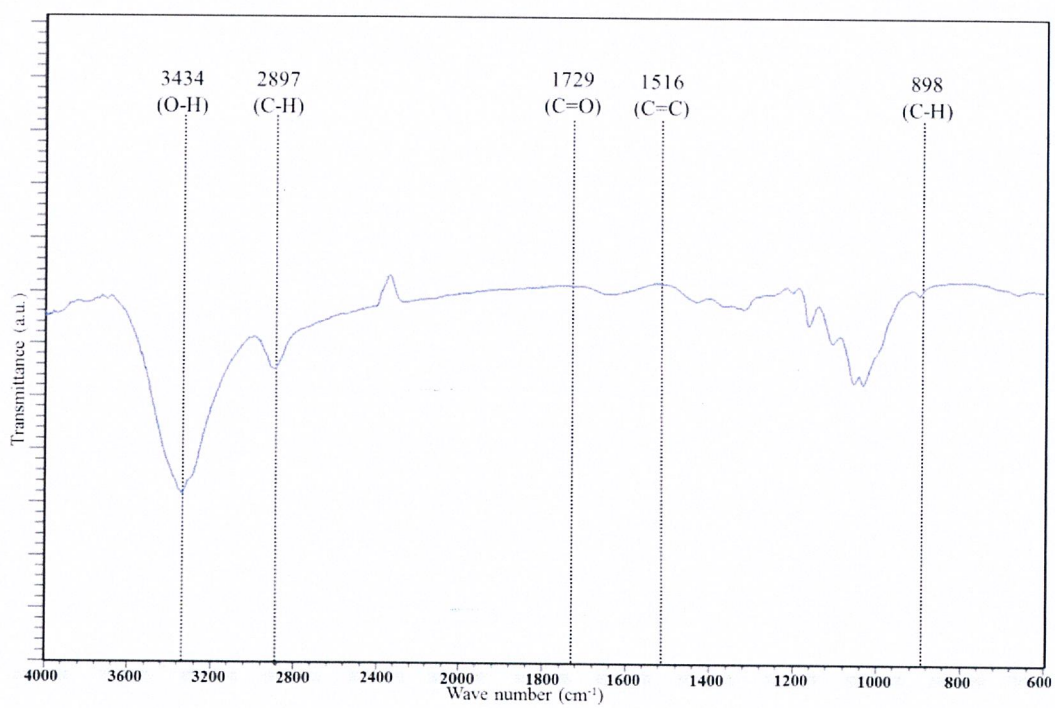


Figure 4.2.2 FTIR spectrum of cellulose from EFB.

4.3 Isolation of cellulose from PPF

Cellulose was prepared by remove wax, partial lignin, and hemicellulose in de-waxing, de-lignification, and hemicellulose removing process, respectively. Figure 4.3.1 shows the change of appearance of the sample obtained after each treatment in the isolation of cellulose from PPF. The color of sample have change, it changed from brown of PPF to yellow of DL-PPF due to the lignin was removed, and become white of DH-PPF after the hemicellulose was removed (Fig. 4.3.1, (a)-(d)). The size of sample was gradually decreased from PPF to DH-PPF due to wax, lignin, and hemicellulose were removed from outer structure of lignocellulosic biomass (Fig. 4.3.1 (a)-(d)). There are obvious change in weight for de-waxing, de-lignification, and hemicellulose removing process. The yield of de-waxing, de-lignification, and hemicellulose removing process were 94.3%, 54.5%, and 30.6%, respectively.

The FTIR spectrum of DH-PPF (Fig. 4.3.2) shown the characteristic peaks of cellulose at $3,434\text{ cm}^{-1}$, $2,897\text{ cm}^{-1}$, and 898 cm^{-1} from stretching of O-H, C-H, and Glycosidic-C₁-H, respectively [5]. Since no characteristic peak of lignin ($1,516\text{ cm}^{-1}$) and hemicellulose ($1,729\text{ cm}^{-1}$) was observed in the spectrum of DH-PPF, it can be concluded that lignin and hemicellulose were completely removed from PPF.

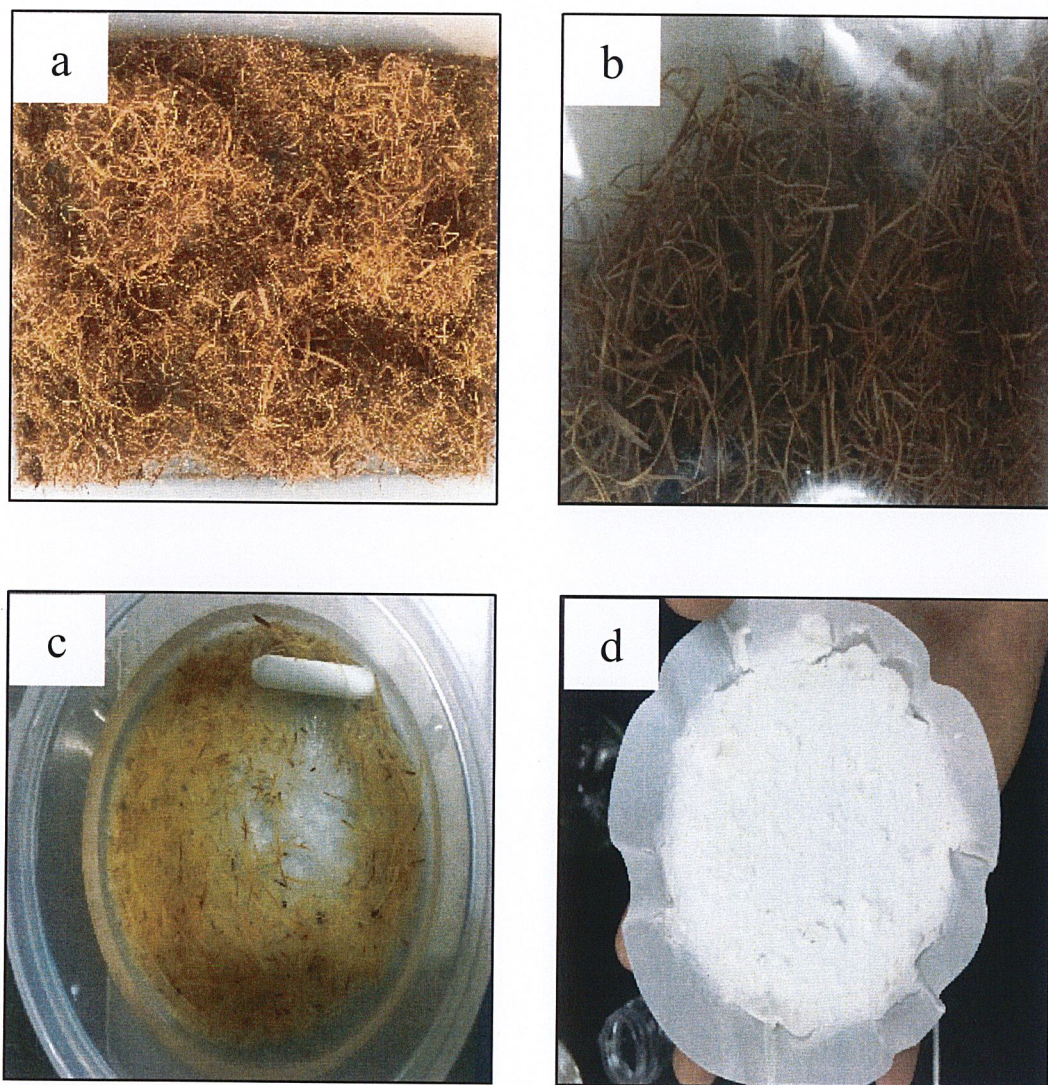


Figure 4.3.1 Physical appearance of PPF (a), after remove wax (b), after remove lignin (c), and after remove hemicellulose (d).

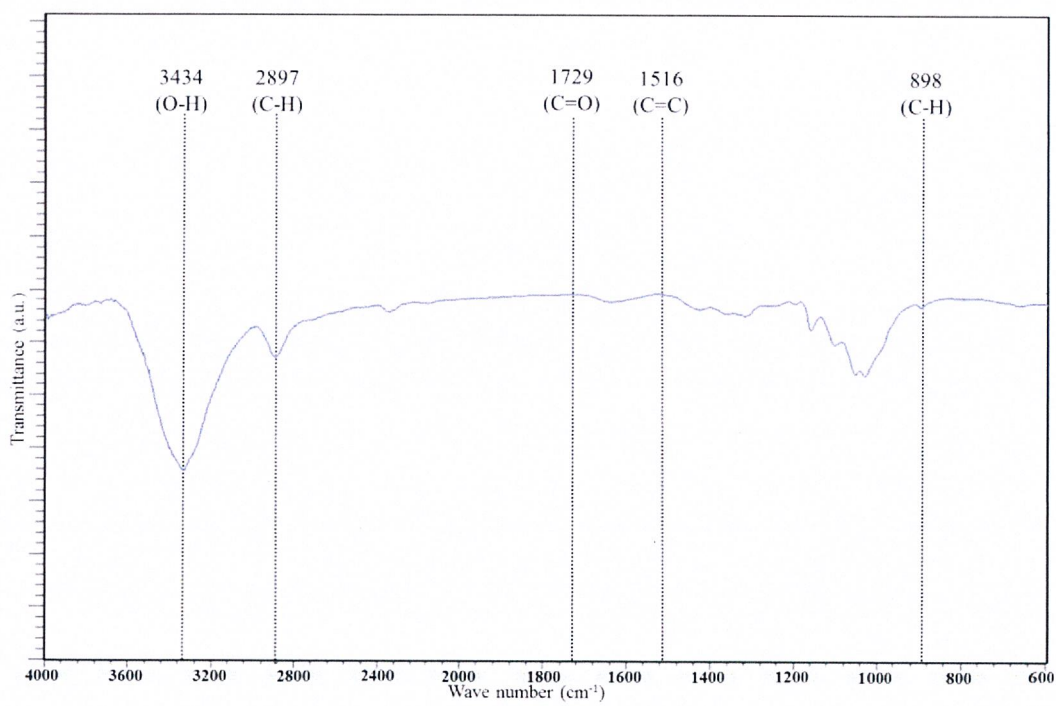


Figure 4.3.2 FTIR spectrum of cellulose from PPF.

Table 4.1 The yield of each process in preparation of cellulose from biomass.

Biomass	Process		
	De-waxing process	De-lignification process	Hemicellulose removing process
PF	100.0%	60.0%	45.2%
EFB	89.1%	66.7%	39.4%
PPF	94.3%	54.5%	30.6%

4.4 Comparative results of cellulose isolated from PF, EFB, and PPF

Cellulose was isolated from PF, EFB, and PPF by de-waxing, de-lignification, and hemicellulose removing process, respectively. Cellulose from PF can be removed completely the lignin and hemicellulose under these condition. On the other hand, cellulose from EFB and PPF cannot be removed completely the lignin under this condition (70 °C, 5 h). However, the cellulose obtained from PF, and the partial cellulose obtained from EFB and PPF shows characteristic peak of cellulose and no characteristic peak of lignin and hemicellulose. These spectrum confirmed that the cellulose isolated from PF, EFB, and PPF were removed completely lignin and hemicellulose.

CHAPTER V

CONCLUSION

The solid waste from oil palm, PF, EFB, and PPF can be used to produce cellulose under these condition. The FTIR spectrum were confirmed cellulose that it isolated from PF, EFB, and PPF. Although cellulose can be isolated from EFB and PPF, but the lignin cannot be removed completely from dewax-*EFB* and dewax-*PPF*. The isolation of cellulose from PF was differentiated from EFB and PPF, cellulose can be isolated from PF by remove completely the lignin and hemicellulose. In addition, the yield in hemicellulose removing process of PF also higher than EFB and PPF. It can be concluded that cellulose can be isolated from PF, EFB, and PPF with the appropriate biomass for these condition was PF.

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APPENDIX

Yield of each process were calculated from the dry basis weight of sample at before and after reaction, the raw data shown in table I.

Table I The dry basis weight of sample before and after reaction in each process.

Biomass	Process					
	Extraction		Bleaching		Treatment	
	Before	After	Before	After	Before	After
OPF	3.50 g	3.50 g	3.50 g	2.10 g	2.10 g	0.95 g
EFB	3.49 g	3.12 g	3.12 g	2.08 g	2.08 g	0.82 g
PPF	3.50 g	3.30 g	3.30 g	1.80 g	1.80 g	0.55 g