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PREPARATION AND CHARACTERIZATION OF CELLULOSE PULP FROM FIBER OF DURIAN PEEL

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ABSTRACT

Cellulose pulp is a biomolecule that obtained as a bundle of lignocellulose fiber. Durian peel is one of the most agricultural wastes that can be found in one province of Indonesia. This study aimed to prepare cellulose pulp from durian peel fiber. The separation of bundle durian peel fibre was performed using the grinding method. The extraction process is the most important step due to during this process any unwanted materials (hemicellulose, lignin, etc) can be removed from the cellulose matrix. The extraction was performed by using a chemical treatment that combined with steam explotion. The yield of cellulose pulp obtained by this process was about 40-45%. Cellulose pulp was categorized as short fiber or lead bleached craft pulp. Bright degree, extractive content (dichloromethane), and water content meet the minimum value of SNI 6107. The morphological analysis of durian cellulose pulp showed a smooth surface with pores in several places. The crystallinity index of cellulose pulp durian after treatment was an increase to 60.67% from 40.07%.

Keywords: Cellulose Pulp, Durian Peel, Fiber, Extraction, Lignocellulose

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INTRODUCTION

The pulp is also called cellulose and starting material for several kinds in the industrial process, i.e. paper, composite materials, a derivate of cellulose (e.g. viscose rayon, cellulose ester, cellulose ether) and other cellulose-based products (Nano- and microcrystalline cellulose). 1, 2

The wide application of cellulose in the industrial level makes cellulose to be an interesting natural product for characterizing and developing. Silviana and Subagio,³ made cellulose-based bioplastic from bamboo fiber. Siregar.et.al, ⁴ reported a successfully of cellulose-based biofilm preparation, with antibacterial activity, that isolated from Gebang leave (Coryphautan). The obtained biofilm was prepared through a crosslinked process between modified cellulose (CMC) and PVA. In other study, Mahendra et. al⁵ reported oil palm-based nanocrystal cellulose can be used as co-agent to help the emulsification of cyclic natural rubber. Also, Zeng et.al, ⁶ reported, cellulose that obtained from corn stem can be modified with acrylonitrile, and it can act as an adsorbent for heavy metal, i.e. cadmium. Another study also performed to develop a specific adsorbent for lead, modifying cellulose with benothiazole.⁷

The raw material of cellulose pulp mostly dominated by wood-based products, i.e. short and long wood fiber. The percentage of wood and non-wood-based cellulose pulp is about 90-95 and 10-5%, respectively. This data showed the dependence on the wood-based product, the impact of this activity is deforestation and replanting. Thus, the alternative raw material form a non-wood-based product is urgently needed.^{8, 9} Durian peel waste is



one potential alternative to substitute wood-based cellulose pulp. The use of durian peel waste for cellulose pulp production is believed can reduce the cost production and improve the value of durian fruit itself.

Durian peel is categorized as waste from the agriculture sector and has a high potential to develop as fiber sources. This based on its availability - Statistics Indonesia claimed the production of durian fruit in Sumatera Utara was an increase from 65529 (the year 2015) to 74811 tons (the year 2017). This number is predicted will continue to increase to meet markets need for the next 20 years, it's about 90000 ton/year ¹⁰. The increase in production numbers will have a direct impact on the waste number that will be produced. The waste from durian can be classified into the seed (20-25%) and peel (70-85%). This percentage showed that durian has a quite high organic waste. ^{3,11,12,13} Based on its chemical composition, durian contained by cellulose (60.45%), hemicellulose (13.09%), and lignin (15.45%). ^{11,12,14}

The pulp can be used for starting material for any products after passes the extraction step. This process is needed to remove any impurities attached to cellulose pulp surfaces, i.e. lignin and hemicellulose (Fig.-1). In the plant tissue, cellulose can be found together with starch, lignin and hemicellulose.

Several studies on the cellulose pulp extraction of durian peel have been done. Rachtanapun et al,¹⁴ had performed the extraction in the presence of 10w.% NaOH at 100°C and followed by the bleaching process using hydrogen peroxide.¹¹ had done the extraction of durian peel powder, and the extraction was conducted using chlorination and maceration. Dewi and Putri¹⁰ had successfully isolated cellulose from durian peel. Those studies isolate the cellulose directly without any treatment of fiber separation.

The objective of this study was to prepare pulp from durian peel waste. The first treatment was fiber separation using a mechanical technique. The obtained fiber was continued by the pulping process in the combination of hydrolysis (HNO₃ solution), alkalization (NaOH solution at 70°C), and bleaching in the presence of NaOCl 1.75wt%. Acid hydrolysis is performed to remove the presence of starch, wax, and fatty acid in the durian fiber ^{15,16,17}. Then the alkalization is conducted to remove lignin and hemicellulose that bent with cellulose. ¹⁸⁻²⁰

EXPERIMENTAL

Material and Methods

Durian peel waste was obtained from the traditional market in Binjai, Sumatera Indonesia, Indonesia. Chemical in this study was pro analytical grade from Merck, i.e. nitric acid, sodium hydroxide, sodium perchlorate, sodium hypochlorite, hydrogen peroxide, and sodium meta periodate.

Fiber Separation of Durian Peel Waste

The separation of durian peel fiber was performed mechanically, based on our previous study.²¹ Durian peel waste was washed several times to remove any contamination on its surface. The dried peel was continued with the grinding process and soaked in water for 24 h. The soaking treatment was performed for removing the gum from the durian surface through the microbial process (Fig.-2c).

Cellulose Pulp Preparation

75 g of fiber was added into 1000 mL of HNO $_3$ 3.5wt.% and placed in the autoclave. The steam explosion was conducted for 30 min (126°C and 0.15 MPa). The obtained fiber then neutralized using distillate water, and the fiber was refluxed with 750 mL of NaOH 2wt.% at 70-80°C for 30 min. Treatment was continued by the bleaching process using NaOCl 1.75wt.% at 70-80°C for 30 min. The obtained cellulose was continued into alkalization using 500 mL of NaOH 17.5wt.% for obtaining α -cellulose and then neutralized using distillate water. The obtained α -cellulose was bleached using 500 mL hydrogen peroxide 10% at 70°-80°C for 30 min and washed with distillate water to remove the scent of hydrogen peroxide.

Cellulose Pulp casting

The casting of pulp was performed using a hand sheet (Fig.-1). About 200 g of wet cellulose pulp was dispersed in 2000 mL distillate water. The obtained solution was poured into the hand sheet and dried using a vacuum system (the final weight of cellulose pulp was 84-88 g) (Fig.-2f).

Analysis of Cellulose Pulp

The obtained cellulose pulp was analyzed using several methods based on the Indonesian National Standard (SNI):

SNI 01-1840-1990 to measure the length of fiber from wood and non-wood.

SNI ISO 5264-1, Pulp – beating process in the laboratory - Part 1: Valley beater method.

SNI ISO 5267-2, Pulp – determination of drain ability - Part 2: Canadian Standard Freeness method.

SNI ISO 2470-1, Paper, board, and pulp - Measurement of diffuse blue reflectance factor – Part 1: indoor daylight condition (ISO brightness).

SNI ISO 5350-3, Pulp - specifies a procedure for the estimation of the visible dirt and shaves by reflected light using the Equivalent Black Area (EBA) method in pulps.

SNI 8401, Soluble extractive of wood and pulp (T-2014 cm-07, IDT)

SNI ISO 52691, Pulp – procedure for sheet preparation for physical testing – Part 1: Conventional method SNI 08-7070, Procedure to determine the water content of pulp and wood using the oven method.

SNI ISO 536 Paper and board – Procedure for determining the grammage of paper and board

SNI ISO 1974 Specifies a method for determining the (out-of-plane) tearing resistance of paper – Elmendorf method

SNI ISO 2758, Specifies a method for measuring the bursting strength of paper

SNI ISO 1924-2, Specifies a method for measuring the tensile properties – Part 2: Constant rate of elongation method.

FT-IR Analysis

The spectra of the functional group on cellulose pulp and modified cellulose pulp was determined by the FT-IR spectrophotometer using Cary 630 FT-IR Agilent. All obtained spectrum was recorded in the following condition: the resolution of 4 cm⁻¹ and wavenumber range of 4000-650 cm⁻¹ and scanned 32 times.

SEM Analysis

The surface morphology characterization of cellulose and modified cellulose were determined using a scanning electron microscope (SEM) JEOL/EO JSM-6510LA Version 1.0.

XRD Analysis

The XRD analysis was performed using Lab X XRD-6100 (Shimadzu) with the following condition: voltage of 40 kV, current of 30 mA and CuKα radiation. The scan process was performed in the range of 20 7-70°. Crystallinity index was determined using equation (1) below:

$$I_{cr} = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \tag{1}$$

Where, I_{cr} , I_{002} , and I_{am} , referred to the crystallinity index, the intensity of the crystalline region (22°), and intensity of amorphous region (18°). $^{22-28}$

Thermal Analysis

Thermogravimetric (TG) and differential thermogravimetric analysis (DTA) was performed using Extra TGDTA7300 (Hitachi). The measurement temperature was set from room temperature until 600° C with heat rate 10° C /minute.



Fig.-1: Hand Sheet for Cellulose Pulp Casting 1769

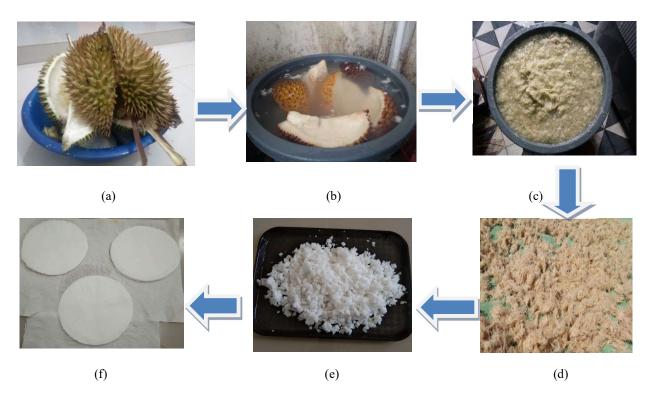


Fig.-2: Pulp Preparation, (a) Durian Peel, (b) Soaked Durian Peel, (c) Soaked Durian Peel after Grating, (d) Dried Durian Peel Fiber, (e) Cellulose Pulp, and (f) Casted Cellulose Pulp

RESULTS AND DISCUSSION

Cellulose Pulp

Cellulose is a natural biopolymer that can be extracted from any kind of lignocellulose sources. The presence of cellulose in nature can be found as a matrix that cross-linked with any kind of polysaccharide. The most component of that matrix is cellulose, hemicellulose, and lignin. ²⁹ Cellulose extraction from that matrix was performed to remove those unwanted components. Figure-2 shows the process for separating cellulose from those unwanted components (hemicellulose, lignin and extractive components). The process was started by soaking the peel durian fiber in water to remove any dirt on the surfaces of peels (Fig.-2b). The process was continued by the grinding process and soaking again in water for 24h (Fig.-2c). The drying process was performed to remove any water that absorbed by peels (Fig.-2d). And at Fig.-2e, cellulose pulp was obtained from peels fiber after the pulping process through the hydrolysis process, alkalization, and bleaching. The casting of cellulose pulp was performed by a conventional technique using a hand sheet (ISO 5269-1:2005). The yield of cellulose pulp obtained through this process was about 40-45%. The result can vary depends on the method that used, for example in the study performed by Penjumras, et,al, ¹¹, at the end of the process the yield was about 33,12 %.

Characteristic of Cellulose Pulp

The testing to determine of fiber length was performed following SNI 0698 NBKP (Needle Bleached Kraft Pulp). Needle Bleached Kraft Pulp is produced form length wood-fiber and the minimum value was determined must be 2.1 mm. Table 1 showed the obtained pulp has a lower value than minimum value that needed to meet based on the SNI 0698 NBKP. Based on that value, the obtained pulp can be categorized as short fiber.

Table-1: Fiber Length of Durian Peel							
No.	Parameter	Unit	Result	SNI 0698 NBKP			
1	Average fiber length	mm	0,67	min. 2,1			

The obtained cellulose pulp was then compared with the SNI 6107 LBKP (Leaf Bleached Kraft Pulp). The data were summarized in Table-2. Based on those parameters, durian peel pulp meets 5 of 8 parameters, i.e. the brightness degree has a higher value than the SNI 6107, this is due to the impact of the bleaching process using NaOCl and hydrogen peroxide during the pulp preparation. But the contrary result was found on the visible dirt parameter (Fig.-3), this value quite high than the maximal value that needed to be meet. This result can be caused by completing treatment during the hydrolysis and alkalization.

Tuber 2: The Quanty of Burian Feet Condition Full						
No.	Parameter	Unit	Result	SNI 6107 LBKP		
1	Degree of Initial Beating	mL CSF	540	min. 430		
2	Degree of Brightness	% ISO	91	min. 85		
3	Visible Dirt	mm^2/m^2	89	max. 5		
4	Extractive Content (Dichloromethane)	%	0.3	max. 0.4		
5	Water Content (AD)	%	7	max. 10		
6	Tearing Index with eating degree of 300 ml CSF	mNm²/g	2.8	min. 5.5		
7	Bursting Index with beating degree of 300 ml CSF	kPa m²/g	0.3	min. 2.5		
8	Tensile Property with beating degree of 300 ml CSF	Nm/g	5.3	min. 45		

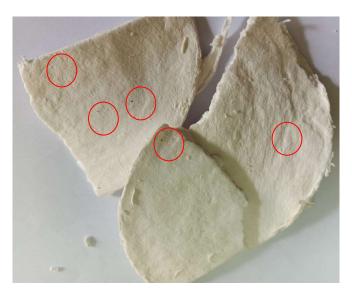


Fig.-3: Cellulose Pulp of Durian Peel and Some Visible Dirt (Red Circle)

FT-IR Analysis

Durian peel fiber consists of lignocellulose, this means it has several components, i.e. cellulose, hemicellulose, and lignin. Cellulose is constructed by D-glucopyranose units. Hemicellulose is constructed by three kinds of sugar, i.e. D-xylose, D-glucose, and mannose, the lignin is constructed by phenyl propane unit. This unit categorized as aromatic compounds, i.e. guaiacyl, syringyl, and phenyl propane (Fig.-5).²⁸

Typical band of lignin was found in the range of 1500-1600cm⁻¹, specifically at 1616 cm⁻¹, it identified as aromatic C=C vibration. Another band was found at 1830 and 1730 cm⁻¹ that referred to the vibration of methoxy (O-CH₃) and aromatic C=C vibration.^{28,30,31} But these bands were not found in the FT-IR spectrum of cellulose pulp (Fig.-4), this result indicated the delignification was successfully removed the lignin, also the xylose band around 1765-1715 cm⁻¹ that identic to the presence of hemicellulose was not found. It is believed the alkalization treatment successfully cut the hemicellulose chain.

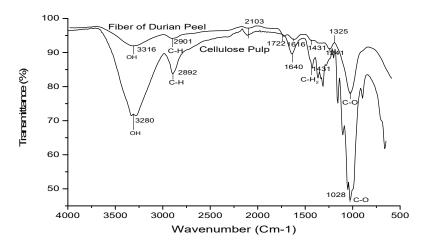


Fig.-4: FT-IR Spectra of Durian Peel Fiber and Cellulose Pulp

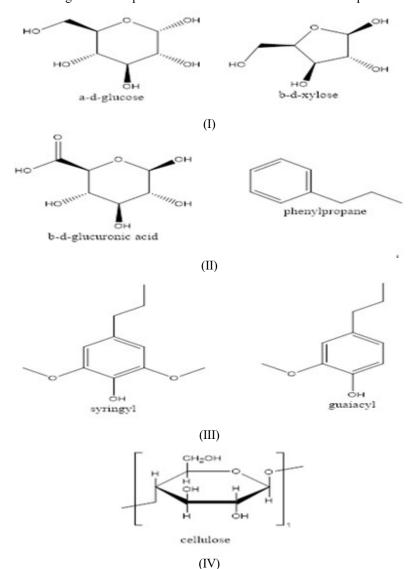


Fig.-5: (I) D-Glucose, D-Xylose, (II) D-Glucoronic Acid, Phenylpropane, (III) Syringyl, Guaiacyl, (IV) Cellulose 1772

Cl-O vibration of syringyl and Guaicyl

C-O-C stretching of aryl-alkyl ether

C-O and C-O-C stretching of β-glycoside

Wavenu	mber (cm ⁻¹)	Functional Group	
Fiber	Cellulose Pulp		
3316	3280	O-H stretching	
2901	2892	C-H stretching	
1722	-	Unconjugated C-O of Xylan	
-	1640	Conjugated C-O	
1616	-	C-Ph vibration	
1431	1431	C-H ₂ scissoring	

Table-3: Functional Group of Durian Peel Fiber and Cellulose Pulp

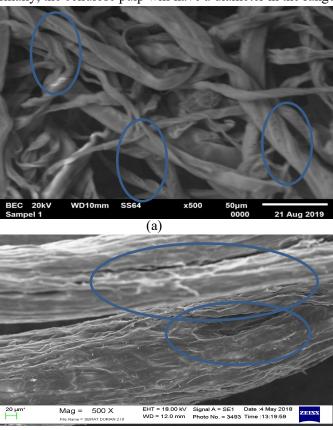
Morphological Analysis

1325 1241

1025

1028

Figure-6a showed the image of morphological surface of durian peel cellulose pulp. The image showed the durian peel cellulose pulp as a long fiber. Many pores also can be found on the fiber surface. This micrograph has a different result with the untreated fiber of durian peel (Fig.-6b). The untreated fiber has a rough surface. This result confirmed a significant result of acid hydrolysis, alkalization, and bleaching treatments. Those treatments also can remove the presence of amorphous region on the cellulose surfaces. The success of treatment also can be seen from the diameter size of fiber, durian peel pulp has an average diameter of 12.41 µm. Normally, the cellulose pulp will have a diameter in the range of 2-20 µm. ³²



(b)
Fig.-6: Scanning Electron Micrograph 500x (a) Cellulose Pulp(b) Durian Peel Fiber

Those three have showed a positive result to the physical properties of durian peel fiber. This treatment can be assumed that able to improve the defibrillation of fiber by detaching the process of lignin and hemicellulose. Defibrillation enhances the possibility of fiber having a smaller diameter size. The presence of NaOCl and hydrogen peroxide during the bleaching treatment has a big role to remove the lignin by oxidizing lignin into carboxylate or carbonyl. 15,33,34

XRD Analysis

Crystallographic of durian peel fiber and cellulose pulp was shown in Fig.-7, which has a crystallinity index of 47.07 and 60.68%, respectively.

The crystallography (Fig.-7) of pulp is significantly different from the untreated fiber. This can be indicated that the pulping process can improve the crystallinity degree removing amorphous region that originated from hemicellulose and lignin.

Thermal Analysis

Based on the Fig.-8, the degradation process was done through 2 steps, i.e. dehydration and degradation. Dehydration and degradation were confirmed in the range of 60-140 and 250-450°C, respectively. Degradation involved several reactions, i.e. decarboxylation, depolymerization and decomposition of glycoside units and the formation of ash as the residue. At 200°C the durian pulp has lost the weight up to 7.9% due to dehydration of pulp into de hydrocellulose. At 400°C durian pulp has lost the weight up to 91.7 due to degradation that involving depolymerization. DTG curve showed a single peak at 350°C, this peak can be associated to the degradation of hemicellulose. The presence of acetyl group in hemicellulose cause the degradation undergoes at the lowest temperature than cellulose and lignin.

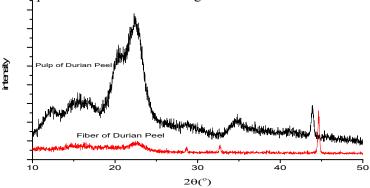


Fig.-7. X-ray Diffractogram of Durian Peel Fiber and Cellulose Pulp

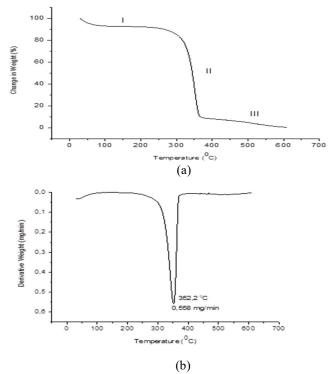


Fig.-8: Thermogram of Cellulose Pulp,(a) TGA; (b) DTG

CONCLUSION

The result of this study showed that durian peel fiber can be used as an alternative for cellulose pulp production. The obtained cellulose pulp was classified into *Leaf Bleached Kraft Pulp* (LBKP) based on SNI 6107. The crystallinity index of durian cellulose pulp is higher than the untreated fiber due to the hydrolysis, alkalization, and bleaching process.

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