The effects of temperature on alpha-cellulose content and extraction result of tobacco stem

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Abstract. This research aims to obtain levels of cellulose and α-cellulose from the tobacco stems. Tobacco rods are extracted using several processes step, namely the Making of tobacco stem powder, powder characterization analysis, powder extraction with several temperatures, and FT-IR analysis. The results obtained at several temperatures showed that the highest cellulose yield was 76.25% with alpha-cellulose content of 82% at a temperature of 150°C.

1. Introduction
Cellulose and alpha cellulose is the content of the main parts of plants that can be found as the inner skin of fibrous wood. Cellulose is needed as a raw material in various industrial fields, such as paper industry, textile industry, food industry, pharmaceutical industry and biofuel industry. Cellulose derivative products can also be used as emulsifiers, disperse agents, edible coating films, and others. Currently, national demands of cellulose are met by heavily relying on imported cellulose. Cellulose with the molecular formula of \((C_6H_{10}O_5)n\) is a glucose polymer compound that is rarely found in a pure form. Linkage is usually found among cellulose, lignin, and hemicelluloses that act as a binder in woody plant. The long chain of cellulose is connected through hydrogen bonds and van der Waals forces [1]. The Figure 2 shows the structure of cellulose composed of straight and unbranched glucose chains. To obtain high purity levels of cellulose, lignin and hemicellulose must be removed from the raw material. Cellulose isolation from fibrous plants can be done by modifying the mechanical extraction process and the chemical extraction process. Chemical extraction can be done using base or acidic methods. Meanwhile, mechanical extraction is usually carried out during pre-treatment stage using size reduction method aiming to increase the surface area of raw materials. Several studies have been successfully conducted to extract cellulose from various agricultural wastes to be applied as specific cellulose derivatives. Extractions of palm kernel kernels have a cellulose content of 20-30% [2]. Whereas, in 2013, extraction of cellulose from cassava stems using NaOH with a concentration of 25% resulted in cellulose content of 67.69% [3]. In 2012, extraction from cocoa pod husk using sodium hydroxide resulted in a yield of 26.09% cellulose content [4]. Another agricultural waste that can also be potentially used as the source of cellulose is tobacco stem waste. Previous study revealed that, the waste of tobacco stem contains 50% cellulose content [5]. The cellulose is also discovered in tobacco leaf stems [6]. The high cellulose content of tobacco stem waste and its high abundance make the stems as a potential source of cellulose. The Figure 1 shows dried tobacco stems obtained from tobacco farmers. The utilization of agricultural waste as a cellulose source does not only reduce the negative impact of the accumulation of the waste but also can increase the value of the waste itself. All this time, tobacco stems are usually left to accumulate and rot without ever being used. This will bring a negative impact to the environment.
Thus, this research is very important to be carried out for the development of agricultural waste potential in countries, like Indonesia, that are rich with tobacco. Several studies have examined the characteristics of tobacco stems to determine its potential so it can be applied effectively. Study of morphological properties of tobacco stem xylem matic tissue has been carried out by [7]. The characteristics are similar with those of biomass that is usually used to produce biofuel [7]. In 2015, the results of extraction of tobacco rod using petroleum ether, chloroform and ethanol solvents can be used for soundproofing [8].

Higher levels of alpha cellulose and decomposed lignin levels showed high cellulose quality. The alpha cellulose level in a fiber shows the cellulose purity level in the yield produced [9]. Therefore, the content of alpha cellulose is used as a determinant of cellulose purity. Thus, this paper, we determine alpha cellulose levels from the extraction of tobacco rod. Extraction of cellulose from the tobacco rod was carried out using base method. Extraction temperature was varied to assess the effect of temperature treatment on the quality of the extraction results. This is because, to date, studies on the effect of extraction temperature on cellulose quality have not been widely carried out. Therefore, this article examines the effect of temperature treatment on the yield and characterization of cellulose from tobacco stem extract obtained using base method. The treatment of alkaline base in lignocellulose material can selectively remove lignin [10]. Whereas, in 2008, temperature treatment on lignocellulosic material will cause changes in the chemical components of the material by breaking the existing chemical bonds [11]. In 2008, the first component to be degraded is hemicelluloses [12]. However, treatment using temperature above 160°C will degrade cellulose.

2. Methods

2.1. Making tobacco rod powder
At this step, it begins to drying and cutting, as well as refinement of tobacco mesh with a size of 60 mesh which produces fine powder, called powder stubble.
2.2. Characterization of materials
Tobacco stem powder is characterized by several analyses, namely: analysis of cellulose content, lignin content, hemicellulose content, water content and ash content. Analysis of cellulose content was carried out using the SNI method No 0444:2009. Characterization of lignin content using SNI method No 0492:2008, hemicellulose content using ASTM method No D1104-56, water content using SNI method No 712:2015 and ash content using SNI method No 01-22354-2006.

2.3. Extracting tobacco
The process of extracting tobacco stem fibres: This step, powdered tobacco stems are divided into 5 samples weighing each 10 grams of tobacco stems. Each sample was inputted with 50 mL NaOH 12.5% with a ratio of 1: 5. Each sample was heated at different temperatures, namely 100 ° C, 120 ° C; 130 ° C; 140 ° C and 150 ° C and rotated at a speed of 150rpm for 30 minutes. Cellulose, specifically alpha cellulose will not be damaged if the extraction temperature used is suitable namely the temperature is less than 160oC [13].

The samples are cooled and filtered. The residue obtained is washed with distilled water such that the residue has a neutral pH. The sediments of residue which have water content heated in an oven at 105°C.

2.4. Data Analysis
The results of the extraction were analyzed using FT-IR to determine the chemical bonds that exist in the tobacco stem extract. FT-IR analysis was carried out to ensure the product obtained matched with the expected product. This can be determined by examining appeared functional groups wave numbers.

Cellulose content, alpha cellulose content and lignin content of the tobacco stem extract were also analyzed.

3. Result and Discussions
Cellulose extraction of tobacco stems using the base method is carried out with several temperature variations. This base method uses NaOH as a solvent because NaOH can reduce the amount of lignin bound on the sample. NaOH can break down the basic bonds of the lignin structure and form soluble phenolic salts. It was also stated by [14] that NaOH could separate hemicellulose and could break down lignin structure in crystalline and amorphous forms. In addition, NaOH particles are able to break down lignin structures, which make lignin more soluble and cause a decrease in the lignin content detected in the material [15]. The hydroxyl ion from NaOH will react with lignin to form H₂O. Sodium ions will form phenolic salts, which will dissolve when rinsed.

Content analysis of the tobacco stem was carried out to determine the characteristics of the tobacco stem used. The result of the content of tobacco stem is shown in Table 1.

After we analyzing tobacco stem content, then we extracting alpha-cellulose from tobacco powder using temperature variations namely from 100C until 150C. In this research, we only use the temperature less than 160°C because on the temperature of 160°C and more; the cellulose will be damaged [13]. The results obtained, we show in Table 2 and analysis results of tobacco stem extract in several temperature variations.
Table 1. Characterization of tobacco stem fibres

| Parameter     | Composition (%) |
|---------------|-----------------|
| Water content | 11.16           |
| Ash content   | 9.90            |
| Cellulose     | 50.00           |
| Hemicellulose | 22.60           |
| Lignin        | 17.00           |

Figure 4. Tobacco stem powder.

Table 2. Extraction result of tobacco stem from several temperature variations.

| Temperature (°C) | Rendement (%) | Alpha-cellulose (%) | Lignin Content (%) |
|------------------|---------------|---------------------|-------------------|
| 100              | 46.75         | 35                  | 8.7               |
| 120              | 53.00         | 45                  | 8.6               |
| 130              | 67.50         | 48                  | 7.4               |
| 140              | 69.00         | 60                  | 6.0               |
| 150              | 75.25         | 82                  | 1.8               |

The results of the analysis show that the temperature of the extraction process greatly affects the increase in alpha cellulose yield. This implies that there is a decrease in lignin levels from the extraction of the tobacco rod. In addition, the process temperature also affects the reaction rate. The rate of reaction between NaOH and lignin will increase because it is an increase in reaction temperature. This is caused by the increasing breakdown of lignin macromolecules so that we obtain more alkaline soluble lignin. The other words, we obtain more lignin from cellulose. Therefore, this implies that alpha cellulose which obtained to be to increase from extraction. This shows an increase in the purity of cellulose obtained because if alpha cellulose is increasing, then the cellulose will be increasing of purity level of the yield produced [9]. Besides temperature, the increase in alpha cellulose levels was also influenced by the use of NaOH because alpha cellulose has a long chain and is insoluble in a solution of 17.5% NaOH at 20°C.

Figure 3 shows the mechanism for breaking the bonds between lignin and cellulose using NaOH. During the extraction process, NaOH reacts with lignin to form phenolic and H2O salts. The addition of alkaline alkaloids can break down the lignin structure so that lignin is easily separated from cellulose.
Another factor that affects the high extraction yield is the sample size. The sample size will affect the porosity of the sample. This facilitates contact between the sample and the alkali solution to facilitate the separation of cellulose from other components. In this study, we only use a sample size of 60 mesh as shown in Figure 4.

![Figure 5. Trend line of increase in alpha-cellulose and decrease in lignin](image)

On the graph in figure 5 shows the value of $R^2 = 0.997$ for the increase in alpha-cellulose and $R^2 = 0.985$ for the decrease in lignin. This shows that the increase in temperature of the extraction process greatly determines the increase in alpha cellulose levels and decreased levels of lignin.

The average increase in alpha-cellulose in the increase of every 10°C is 11.75%. In other words, an increase of 1°C gives an average increase in alpha cellulose of 1.175%. While the average reduction of lignin is obtained 0.17% for each increase of 1°C. However, the increase in alpha cellulose levels can only apply at temperatures less than 160°C [13].

4. Conclusion
Temperature treatment during extraction does affect reaction rate between NaOH as solvent and lignin of the material. The higher the extraction temperature, the higher the temperament of cellulose. The higher the extraction temperature, the lower the lignin content remaining in the material. Chemical composition, especially high cellulose content in tobacco stems, has the potential to be used as an alternative cellulose source.

5. References
[1] Perez J, Munoz-Dorado J, Rubia T and Martinez J 2002 Biodegradation and Biological Treatments of Cellulose, Hemicellulose and Lignin: An Overview. *Int. Microbiol.* 5 53–63
[2] Bono A, et al., 2009 Synthesis and Characterization of Carboxymethyl Cellulose from Palm Kernel Cake *Advances in Natural and Applied Sciences* 3 1 5–11
[3] Widodo L U, et al., 2013 Pemisahan Alpha-Selulosa Dari Limbah batang Ubi kayu Menggunakan Larutan Natrium Hidroksida *Jurnal Teknik Kimia* 7 2 43–47
[4] Hutomo G S, Djagal W M, Angrahami S, Supriyanto 2012 Ekstraksi selulosa dari Pod husk Kakao menggunakan sodium Hidroksida. *AGRITECH* 32 3 223-229
[5] Handayani S S, Amrullah 2018 Extraction of cellulose from tobacco rod as preparation to bioethanol production *Jurnal Penelitian Pendidikan IPA JPPIPA* 4 2 38-42
[6] Gordana J K and Vesna B R 2011 Analysis of cellulose content in stalks and Leaves of large leaf tobacco *Journal of Agricultural Sciences* 56 3 207-215
[7] Juliano, Daniela, Boaretto, Luis F S, Fernando, Labate, Carlos A 2016 Tobacco stalk as promising feedstock for second generation ethanol production. *Bioenergia em revista*
diálogos 6 2 47-61

[8] Fransiska A W, Hardiansyah, Sari, Nugroho K O, Rifa D, and Riza F D 2015 Pengolahan serat batang tembakau sebagai soundproofing material : alternatif penanggulangan limbah batang tembakau Prosiding seminar nasional PERTETA 262-267

[9] Sutiya B, Wiwin T I, and Adi R 2012 Kandungan Kimia Dan Sifat Serat Alang-Alang (Imperata Cylindrica) Sebagai Gambaran Bahan Baku Pulp Dan Kertas BIOSCIENTIAE 9(1) 8-19

[10] Kim J S, Lee Y Y, and Kim T H 2016 A review on alkaline pretreatment technology for bioconversion of lignocellulosic biomass Bioresource Technology 199 42–48

[11] Esteves B, Marquez A V, Domingos I, Pererira H 2007 Influence of steam heating on the properties of pine (Pinus pinaster) and eucalypt Wood Science Technology 41 193-207

[12] Kocaefe D, Poncsak S, Dore G, Younsi R 2008 Effect of heat treatment on wettability of White Ash and Soft Maple by Water Holz Roh Wurst 66 355-361

[13] Mussatto S I and Roberto I C 2004 Alternatives for detoxification of diluted-acid lignocellulosic hydrolyzates for use in fermentative processes: a review Bioresource Technology 93 1-10

[14] Safaria S 2013 efektivitas campuran enzime selulase dari aspergillus niger dan trichoderma reesei dalam menghidrolisis substrat sabut kelapa Jurnal Kimia Khatulistiwa 2 1 46-51

[15] Elwin L M and Hendrawan Y 2013 Analisis Kandungan Selulosa, Lignin, dan Hemiselulosa Eceng Gondok Jurnal Keteknikan Pertanian Tropis dan Biosistem 2 2 104-110

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