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Isolation of cellulose from salacca midrib fibers by chemical treatments

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Abstract. Salacca midrib fibers (SMF) have potential as a new source of cellulosic material. The method of isolate cellulose from SMF carried out by the different stages of chemical treatment. First stage is alkalization using sodium hydroxide and then second stage is bleaching treatment using hydrogen peroxide. The chemical composition analysis of SMF was determined at different stages of chemical treatments. Physical analysis was carried out by fourier transform infrared spectroscopy (FTIR) and x-ray diffraction (XRD). Change in surface morphologies of fibers were characterized by scanning electron microscopy (SEM). Isolation process of cellulose from SMF by alkali and bleaching treatments successfully increased content of α-cellulose, decreased content of lignin and increased percentage of index crystallinity of fibers.

1. Introduction

Natural fibers can be grouped based on the source that comes from plants, animals or minerals. The natural fibers of the cultivated plants have the advantage of obtaining and having a shorter harvest time. Lignocellulosic fibers, also known as cellulose-based fibers, can be devide into nonwood and wood or plants fiber. Natural fibers are an alternative reinforcement for composite application due to the environmental issues [1]. The main content of natural fibers is cellulose, hemicellulose and lignin [2]. Cellulose is renewable, biodegradable and the most abundant natural polymer. Salacca midrib fibers (SMF) is one of prospective tropical plants to produce fibers from their bast (figure. 1). SMF has a diameter of 3 to 3,5 cm with a length of 110 cm. The fibers extracted from the bast of salacca midrib are generally 4-5 years old The chemical composition of raw SMF contain α-cellulose 47.18%, hemicellulose 31.89%, and lignin 22.27% [3].

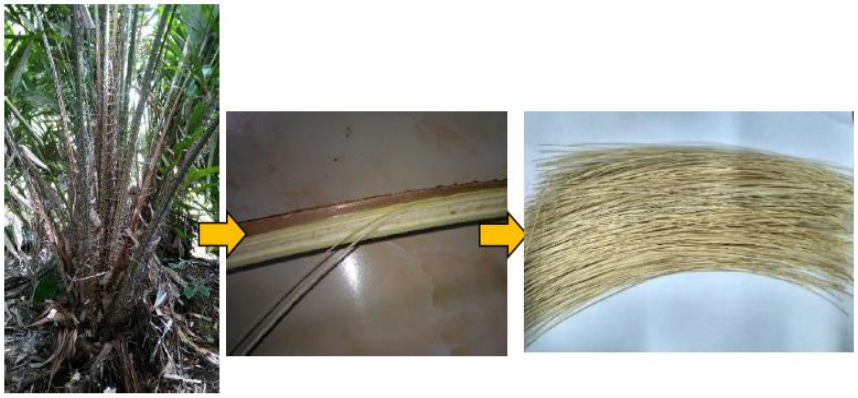

Figure 1. Salacca midrib fibers.

The isolation proses to separate the cellulose from the extractive substances attached on the fiber. The extractives are hemicellulose and lignin. Hemicellulose is one of the plant tissue that serves as water reserves. This network of hemicellulose is made up of clusters of double and branched polysaccharides. Lignin is a support network of plants and protects plants from the surrounding environment. Lignin acts as a matrix to plants and has thermoplastic properties that are not readily biodegradable by water or other compounds. The alkali treatment of natural fibers aims to defibrillate and reduce impurities on the fiber surface [4]. The cellulose is a new material that can be used as an reinforcement in the polymer [5]. With the addition of cellulose from SMF to polymer is expected to improve the composite strength. The process of isolating cellulose by chemical treatment is to soak the SMF in a solution of Sodium hydroxide followed by immersion in hydrogen peroxide. The results of subsequent chemical treatments will be characterized by testing of chemical composition, scanning electron microscopy (SEM), Fourier transform infrared (FTIR) and X-ray diffraction (XRD).

2. Experimental

2.1. Material

Salacca midrib fibers were obtained from Turi, Sleman, Daerah Istimewa Yogyakarta, Indonesia. Sodium hydroxide (NaOH) with a purity of 99% and Hydrogen peroxide (H2O2) with a purity of 3%.

2.2. Isolation and chemical analysis of cellulose

SMF were separated from the bast, then dried at the temperature 50°C for 36 hours, the chemical composition of α-cellulose and holocellulose was determined by chlorite acid modification method and lignin refers to SNI 0492:2008. The fibers were immersed in 2% wt. of NaOH solution [6] at the temperature 70°C for 120 minute at atmospheric pressure and then continuing by bleaching treatment with 3% (v/v) of hydrogen peroxide solution at temperature 60°C, PH10, for 60 minute [7].

2.3. Characterization

The morphology of each stage of chemical treatment on SMF fibers is seen using SEM model JEOL–JSM 6510LA operated at 15 kV. The samples were coated with Au using the sputtering technique. Stages of chemical treatment performed are alkalization and bleaching. Supporting data ensuring that fibers are clean from impurity or other extractive substances require FTIR testing. FTIR aims to detected the functional group, the FTIR model is Shimadzu operate in the range of 400-4000 cm-1. The XRD aims to identify the structure of fiber. The XRD model is Rigaku Miniflex600 operating with Cu Kα radiation (λ=1.5418 Å) ranging from 2θ = 4° - 40°. The Crystallinity index can be calculated using the segal method [8] as shown in Eq. (1):

$$\mathrm{CrI} = \frac{(\mathrm{I}_{002} - \mathrm{I}_{\mathrm{amorph}})}{\mathrm{I}_{002}} \times 100\% \qquad (1)$$

Generally the maximum peak intensity located at 2theta = 22° which represent the crystalline cellulose. Iamorph is the valley intensity (minimum) located at 2 theta is around 18° which represent amorphous cellulose.

3. Results and Discussion

3.1. Chemical analysis and morphology

The purpose of purification process was to separate the cellulose fibers by removed lignin and other extractives. Table 1 shows the purified process of cellulose from SMF. Table 1 shows that the lowest percentage of α-cellulose and the raw fiber has the highest percentage of hemicellulose and lignin. After purification processes indicated that increasing α-cellulose content from raw fibers 47.18%, alkali 49.14%, and bleached 51.81%. The effect of the different chemical treatments shown in Figure 2. from the change colour of fibers. The bleaching process conducted after the alkali treatment step contributed to modifying the fiber color because it removed the lignin. Figure 3 shows the SEM images of the raw SMF compare by the chemical purified cellulose fibers. After chemical pretreatment, the cellulose fibers were defibrillation into individual micro sized fibers.

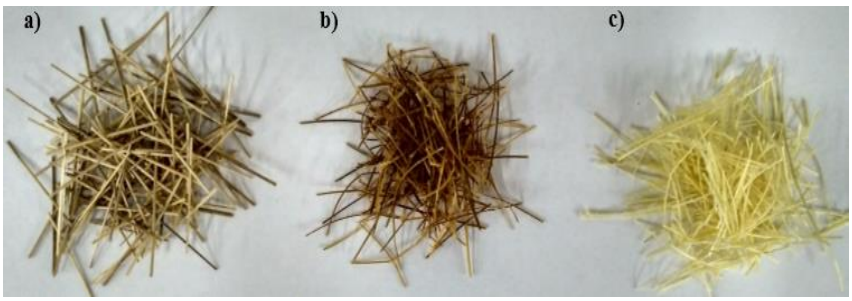

Figure 2. Optical photographs showing the different chemical treatments a) untread SMF, b) alkali treated, c) bleached fibers.

Table 1. Chemical composition of fibers at each stages of treatment

Material	Alpha-Cellulose (%)	Hemicellulose (%)	Lignin (%)
Raw SMF	47,18	31,89	22,27
Alkali	49,14	19,76	20.48
Bleached	51,81	23,12	18.83

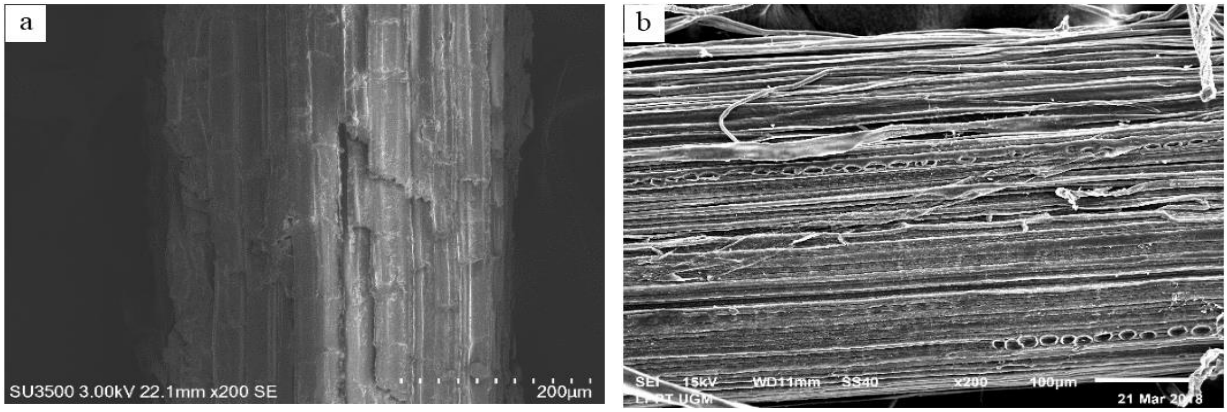

Figure 3. SEM images of the a) untreated SMF and b) bleached fiber.

3.2. FTIR analysis

Infrared measurements were performed on chemically treated fibers to follow the removal of impurities material. Figure 4 compares the FTIR spectra of raw SMF fibers, alkalization and bleached treatments. The band approximately at 1735 cm−1 in the spectrum of raw SMF is attributed to C═O stretching of the acetyl and uronic ester groups of hemicellulose. The band approximately at 1512 cm−1 that could be seen in the spectrum recorded for raw SMF was ascribed to the aromatic C═C stretching from the aromatic ring of lignin [1] [9]. Lignin presented characteristic at the other band in the range 1250–1319 cm−1 indicate that stretching of acetyl C-O and aromatic skeletal vibration CH2 Wagging (lignin). The band located at 1635 O-H (water content).

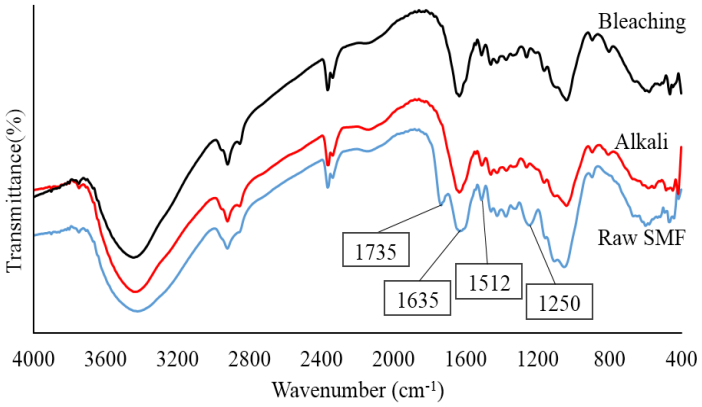

Figure 4. FTIR spectra of fibers at each stage of treatment.

3.3. XRD analysis

The x-ray diffractogram of the raw SMF, alkali, and bleached treatments are shown in Figure 5. Calculation of the crystallinity index based on the Equation 1. For all samples showed that crystalline structure of cellulose 1 in the major intensity peak 2θ around 22.6°, with the intensity of amorphous region peak at a 2θ around 18° [1]. The crystallinity indices increasing from 62,4% for raw fibers, alkali 69.55%, and bleached 70.04%. During the step of purification (raw SMF-alkali-bleaching) progressively increasing the crystallinity index of fiber. This was considered to the progressive removal of amorphous material.

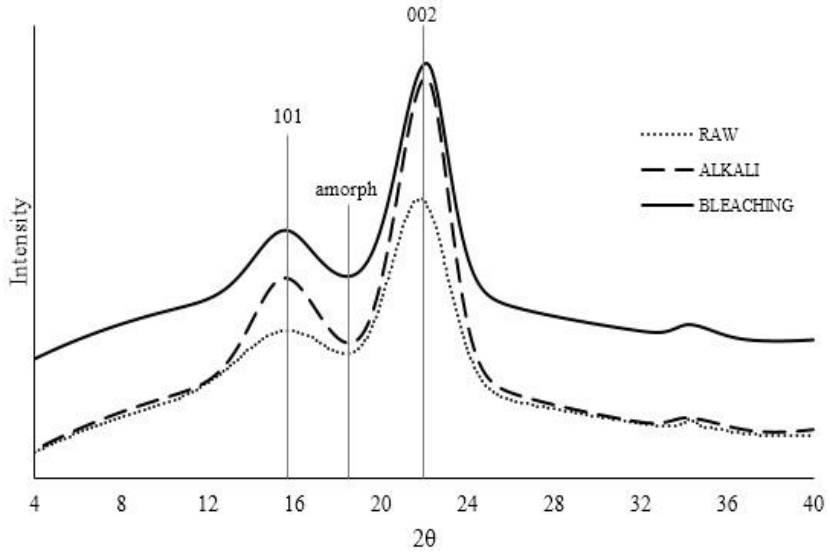

Figure 5. XRD Diffractogram of each stages of chemical treatment.

4. Conclusions

Salacca midrib fibers as a new source of renewable materials contained α-cellulose has been isolated with the yield of 51,81%. The crystallinity index of the cellulose has been extracted with the yield of 70,04%. Effective isolation of cellulose in salacca midrib fibers has been demonstrated by alkalization and bleaching treatment. We have successfully increase content of α-cellulose and decrease amount of non-cellulosic content by chemical treatment using 2% NaOH at the temperature of 70°C for 120 min and bleaching with 3% of H2O2 at the temperature of 60°C for 60 min with PH 10.

Acknowledgments

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References

[1] Sheltami R M, Abdullah I, Ahmad I, Dufresne A and Kargarzadeh H 2012 Extraction of cellulose nanocrystals from mengkuang leaves (Pandanus tectorius) *Carbohydrate Polymers* ***88***(2) 772-779

[2] Kaushik A and Singh M 2011 Isolation and characterization of cellulose nanofibrils from wheat straw using steam explosion coupled with high shear homogenization *Carbohydrate research* ***346***(1) 76-85

[3] S Darmanto, H S B Rochardjo, Jamasri and R Widyorini 2017 Effects of Steaming and Steam Explosion on Mechanical Properties of Snake Fruit (Salacca) Fiber *International Journal of Engineering and Technology (IJET)* **9** pp. 150-157

[4] M Li, S Zhou and X Guo 2017 Effects of alkali-treated bamboo fibers on the morphology and mechanical properties of oil well cement *Construction and Building Materials* **150** pp. 619-625

[5] Ng H M, Sin L T, Tee T T, Bee S T, Hui D, Low C Y and Rahmat A R 2015 Extraction of cellulose nanocrystals from plant sources for application as reinforcing agent in polymers *Composites Part B: Engineering* ***75*** 176-200

[6] Darmanto S, Rochardjo H S, Jamasri and Widyorini R 2017 Effects of alkali and steaming on mechanical properties of snake fruit (Salacca) fiber *AIP Conference Proceedings* **1788**(1), p. 030060)

[7] Yudhanto F and Rochardjo H S 2018 Application of taguchi method for selection parameter bleaching treatments against mechanical and physical properties of agave cantala fiber In *IOP Conference Series: Materials Science and Engineering* **352**(1) pp. 012002 IOP Publishing

[8] Segal, L G J M A, Creely J J, Martin Jr A E and Conrad C M 1959 An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer *Textile Research Journal* ***29***(10) 786-794

[9] Chen W, Yu H, Liu Y, Hai Y, Zhang M and Chen P 2011 Isolation and characterization of cellulose nanofibers from four plant cellulose fibers using a chemical-ultrasonic process *Cellulose* ***18***(2) 433-442