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Microcellulose From Pineapple Leaf Fiber As A Potential Sustainable Material: Extraction And Characterization

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Abstract

Pineapple leaf fiber is consideredan agricultural waste during the harvesting process. Pineapple leaves are rich in cellulose, which made it applicable in many industrial applications. This study aims to extract and characterize microcellulose from pineapple leaf waste in Kampar district, Riau. Pineapple fibers were isolated by alkalization (5% NaOH) at 70ºC for 150 minutes, followed by bleaching treatment (3% H_2O_2) at 60°C for 60 minutes. Microcellulosewascharacterized through various techniques, namely chemical composition analysis, Fourier transform analysis (FTIR), X-ray diffraction (XRD), and thermogravimetric analysis (DT/TGA). The results reveal that the extractedmicrocellulose has significantcellulose content (72.45%) with a crystallinity index of 73.48%. The FTIR spectra of microcelluloseindicatethat alkalization and bleaching treatments removed lignin and hemicellulose in varying degrees. Moreover, the extracted microcelluloseshows high thermal stability, the maximum decompositionoccurredat 347ºC (weight residue 16.37%).Pineappleleaf fiber in Kampar district, Riau, can be a source of microcellulose as a renewable, eco-friendly, and sustainable material for future industrial applications.

Keywords:
Pineapple leaf, microcellulose, crystallinity index, thermal stability, alkalization, bleaching.

1 Introduction

In recent decades, natural fibers have been widely used as an alternative to synthetic fiber reinforcement in polymer composites for industrial applications (such as aviation, automotive, marine, and structures). Natural fibers have attractive properties over synthetic fibers due to their low-density, strength, better stiffness, low cost, eco-friendly, and renewable[5]–[8]. Natural fibers contain main elements namelylignin, hemicellulose, wax, cellulose,etc[9][10]. These elements vary depending on the types of fibers, the plant's age, and the geographical location in which it is cultivated [11][12]. Cellulose has a crystalline structure compared to other elements and cellulose molecules are bonded by hydrogen bonds. Cellulose provides strength to the natural fiber under loading conditions[13].Different types of natural fibers namely: rami, coir, bamboo, kenaf, hemp, wheat straw, and pineapple leaves are sources of cellulose[9], [14]–[17].

Pineapple leaf fibers have a high cellulose content of about 70%-80% and a low microfibrillar angle. As a result, they have superior mechanical properties [18]. In Kampar district, the average pineapple production is 2,343 tons per year. The data suggested that pineapple leaf waste is abundant and has not been utilized optimally.It should be noted that pineapple leaf fibers contain a high amount of cellulose, which could be the source of nanocellulose and microcellulose. Micro cellulose from agriculture waste (namely pineapple leaf fiber) is a good candidate as a reinforcing composite in thermoset and thermoplastic matrix

Composite reinforcing material from pineapple leaf fibers has been widely studied[1], [19]–[22]. Lopattananon et al.[19] investigated the mechanical properties of natural rubber composites reinforced short pineapple leaf fiber. Gaba et al. [20] studied the mechanical properties and structural of pineapple leaf fibers. Ekoputra et al.[1] found the maximum tensile strength of pineapple leaf fiber as reinforced composite matrix polyester is 29.965 MPa. Hadi et al.[22] studied the utilizing of woven pineapple leaf fiber for polymer composite. This finding indicates that the addition of pineapple leaf fiber woven in 3 layers effectively transfers the load to the matrix. Previous research has commonly studied about the mechanical properties. Meanwhile, few papers regarding the extraction process and characterization of microcellulose from pineapple leaf fibers.

Extraction of microcellulose frequently used chemical treatment (namely alkali treatment and bleaching), mechanical treatment, and chemo-mechanical treatment [23]. Alkaline treatment improves the crystal structure of cellulose by eliminating the amorphous elements, namely lignin, hemicellulose, wax, and pectin. Meanwhile, bleaching treatment removes the residual lignin after alkaline treatment [24]. Previous studies have been reported the extraction and characterization of microcellulose from pineapple leaf fibers[3][4]. Bolio et al. [3] studied extraction and characterization of microcellulose from crown pineapple leaf fiber. Chemical treatments (namely: acid hydrolysis (H_2SO_4), chlorination (3.5% NaClO), alkali (20% NaOH), and bleached (0.5% NaClO)) were used as extraction cellulose. The result showed that microcellulose from crown pineapple leaf fiber has index crystallinity of 43.05%. Gnanasekaran et al.[4] studied the production microcellulose from Pineapple Leaf Fiber (PALF). PALF is treated with alkali (NaOH), bleached ($NaClO_2$), and followed by acid hydrolysis (HCl) for extraction microcellulose. They concluded that PALF was successfully produced into microcellulose by alkali treatment, bleaching, and acid hydrolysis. Microcellulose PALF reveals high thermal stability and has maximum thermal decomposition at 349°C.

Based on the previous studies, the extraction of microcellulose from pineapple leaf fibers is still limited. In this study, pineapple leaf fiber waste from Kualu Nanas Village, Kampar district, Pekanbaru was extracted into microcellulose using chemical treatment (alkalization and bleaching). The obtained microcellulose was evaluated for its chemical composition, functional properties, crystallinity, and thermal stability. This study is expected to illustrate the potential of microcellulose from pineapple fiber leaf waste as an eco-friendly, renewable, and sustainable material for future industrial applications.

2 Research Method

2.1 Material

The raw material of micro-cellulose was obtained from pineapple leaf waste, which was post-harvested from Kualu Nanas Village, Kampar district, Pekanbaru, Indonesia. The chemical reagents, namely: sodium hydroxide (NaOH) and hydrogen peroxide (3% H_2O_2), were acquired from Merck, Germany and technical grade chemicals, respectively.

2.2 Fiber Preparation

Pineapple leaves are washed with water to remove sand adhering to the leaf surface. The outer layer of the leaves was peel offmanually from the fibers. Then, the fibers were washed with

water and dried under the sun for about three to four days to remove moisture.

2.3 Microcellulose Extraction

The microcellulose was extracted through alkali treatment and a bleaching process. The extraction procedure is shown in Fig1.

2.3.1 Alkali Treatment

Alkaline treatment was carried out under the following: the fibers were cut into 1 mm lengths. The fibers were immersedinan alkaline solution (5% NaOH) with a solid (g) to solvent (mL) ratio of 1:100 at 70°C for 150 minutes and stirred with a magnetic stirrer. Next, the fiber was filtered and washed in distilled water until it reached a neutral pH.

2.3.2 Bleaching

Bleaching treatment was carried out to remove the remaining hemicellulose and lignin in the fiber. The fiber was bleached using hydrogen peroxide (3% H_2O_2) with a fiber (g) to solvent (mL) ratio of 1:100. The treatment was at 60°C for 60 minutes in a magnetic stirrer. Then, the fiber is filtered and washed with distilled water until it reaches a neutral pH.

2.3.3 Chemical Composition

A crude fiber analyzer (model: CXC-06) was used to quantify the percentage of cellulose from pineapple fibers (untreated, alkalized and bleached). The cellulose content of the untreated and treated fibers was quantified by the Chesson-Datta method. First, measured the Acid Detergent Fiber (ADF) content of pineapple fiber (unprocessed, alkalized and bleached) for each sample as much as 1 mg. The ADF of pineapple fibers was immersed in 72% H_2SO_4 solution for 3 h. The residue was washed using 300 mL of hot water until the foam disappears and rinsed with 25 mL of 96% alcohol/acetone. The residue was dried in an oven at 105°C for 8 h and cooled in a desiccator. Then,the residue obtained was weighed. The cellulose percentage of pineapple fibers (untreated, alkalized and bleached) was determined using Eq. 1.

$$\%\ cellulose = \frac{m_b - m_c}{m_a} \times 100\% \qquad (1)$$

Where m_b (g) denotes the sample mass after the desiccator, m_c (g) denotes the ADF residue mass, and m_a (g) denotes the initial mass.

2.3.4 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR (spectrum two Perkin Elmer) was carried out to analyze the chemical functional group changes of an extracted microcellulose[15][25]. The powder fibers (untreated, alkalized, and bleached) were blended with potassium bromide (KBr) into pellet form.The sample was scanned with a wave length range of 500 to 4000 cm^{-1} at 30 scans per minute.

2.3.5 X-Ray Diffraction (XRD) Characterization

The level of crystallinity of pineapple fiber was analyzed using XRD (brucker D2 phaserdiffractometer). The voltage and current for Cu-Kα radiation were 30 kV and 10 mA, respectively. The intensity counter was set up to scan over a range of 2θ values between 5° to 80° at a constant angular speed of 0.05°/scan. The obtaineddiffractogram was used to calculate the extracted cellulose pineapple fiber's CrystallinityIndex (CI). The crystallinity index is determined using the Segal formula [26]in Eq. 2.

$$CI = \frac{I_{002} - I_{amorphous}}{I_{002}}\ x\ 100\% \qquad (2)$$

Where CI is the Crystallinity Index, I_{002} is the maximum intensity of the peak between 22° and 23° for the crystalline area at a 2θ angle, and $I_{amorphous}$ is the minimum intensity of the peak between 18° and 19° for an amorphous region at a 2θ angle.

2.3.6 Thermogravimetric Analysis (DT/TGA)

The Thermogravimetricanalyzer (Diamond TG/DTA PerkinElmer instrument) was used to determine the thermal degradation of the extracted cellulose from pineapple fibers. The sample of 2 mg weight was heated at a temperature of 30°C to 550°C with a heating rate of 10°C/min in nitrogen atmosphere conditions.

(a) (b) (c) (d) (e) (f)

Fig.1. The process step of extracting microcellulose from pineapple fibers. (a) Extracted pineapple fibers, (b) drying fiber, (c) alkali-treatment, (d) fiber after alkali-treatment, (e) bleaching treatment, and (f) microcellulose.

3 Results and Discussion

3.1 Chemical Composition

The cellulose content of pineapple fibers (untreated, alkali-treated and bleached) is presented in Table 1. The result shows that alkali-treatment increases the cellulose content significantly (26.74% to 69.77%). According to Dinh et al. [27]stated that alkali-treatment of sodium hydroxide (NaOH) breaks the ester bond among lignin, cellulose and hemicellulose. While the hydrogen peroxide (H_2O_2) bleaching treatment at a temperature of 60°C improves the purity of the cellulose content by 72.45%. The combination of alkali and bleaching degraded lignin and hemicellulose effectively without damaging the cellulose structure.

Table 1. Cellulose content of the pineapple fiber before and after chemical treatment

Pineapple fiber	Cellulose content (%)
Untreated	26.74
Alkali treated	69.77
Bleached (microcellulose)	72.45

3.2 Fourier Transform Infrared (FTIR) Spectroscopy

Fig. 2 reveals the FTIR spectra of pineapple fibers (untreated, alkali-treated and bleached). FTIR spectra exhibit the functional group of lignin, hemicellulose and cellulose. All of the samples show high peaks ranging from 3376 to 3401cm^{-1}, which indicates hydrogen-bonded -OH of hemicellulose and cellulose molecules of the fibers[5][28]. An intense peak at the wavenumber of 2916 cm^{-1} occurs in all samples. This occurrence indicates C-H stretching of cellulose, hemicellulose, and extractive (wax and pectin)[28]–[30].The absorption band at 1734 cm^{-1} (untreated sample) reflected the stretching of the C=O bonds of lignin, pectin, and hemicellulose[30][31].

However, this absorption band is not visible in the FTIR spectra of alkali-treated and bleached. It indicates hemicellulose, lignin, and extractive are removed during alkalization and bleaching treatments. These results are in line with the cellulose content, as shown in Table 1. The chemical treatment (alkalization

and bleaching) increased cellulose content and removed amorphous elements. Two absorption peaks of untreated fibers are also detected at 1638cm^{-1}and 1281 cm^{-1}. These values are related to O-H stretching the absorbed water [32][33] and stretching the COO of hemicellulose and lignin, respectively. However, FTIR spectra of alkali-treated and bleached are no longer detected. The absorption peak at 1031 cm^{-1} (untreated fiber) and 1058 cm^{-1} (alkali-treated and bleached) correspond to the bending C-O-C of cellulose, hemicellulose, and extractive (pectin, wax)[34]. Based on the results of FTIR analysis, it verified that alkali and bleaching treatments change the main chemical composition elements namely hemicellulose, lignin and cellulose.

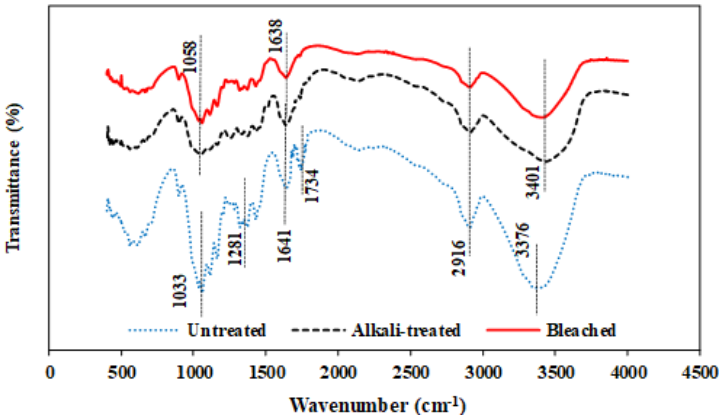


Fig 2. FTIR spectra of pineapple fibers (untreated, alkali-treated and bleached).

3.3 X-Ray Diffraction (XRD)

Fig.3 reveals the peak intensity and the value 2θ of pineapple fibers (untreated, alkali-treated and bleached).Itis noticeable that the three diffractogram is similar. They have three intensity peaks at 2θ =16°, 22°-23°, and 34°-35° with the crystallographic planes of (110), (002) and (004) respectively. These 2θ value corresponds to the typical structure of cellulose-I [35]. The minimum peak intensity indicates the amorphous cellulose. The amorphous cellulose of pineapple fibers (untreated, alkali-treated and bleached) is 2θ of 18.38°, 18.54°, and 18.69°, respectively. A diffraction peak describes the presence of lignin, hemicellulose, and amorphous cellulose at 2θ = 16°. Meanwhile, the diffraction peak of 2θ= 22°-23° indicates the content of α-cellulose[34].The diffraction pattern at 2θ = 34°-35° with the 004 plane represents a low peak of cellulose crystalline[36]. This condition exhibits that NaOH and H_2O_2can maintain crystalline areas at weak peak angles. Based on X-ray diffractograms as shown in Fig. 3.There are diffraction peaks characterization that is the amorphous phase (2θ= 18°) and crystalline phase at 2θ= 16°, 22°-23° and 34°-35°. Therefore, it can be concluded that the X-ray diffractograms are typical of semicrystalline material.

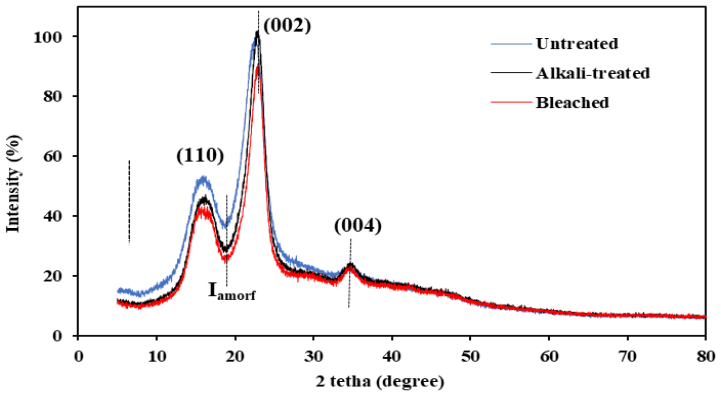


Fig.3. X-RD diffractogram of pineapple fibers (untreated, alkali-treated and bleached).

The calculated crystallinity index of pineapple fiber (untreated, alkali-treated and bleached) using Eq. 2 is shown in Table 2. The crystallinity index of untreated fiber is about 64.21% due to amorphous elements, including hemicellulose, lignin and extractive (pectin and wax). The alkalized and bleached fibers increase the crystallinity index by 72.79% and 73.48%, respectively. Alkalization of NaOH followed by bleaching (H_2O_2) shows much more crystalline structures. The increase in crystallinity index is attributed to the removal of amorphous structures, namely lignin and hemicellulose during the treatment [37]. The greater crystallinity index has rich hydrogen bonds, strong cellulose chains, and a high polymerization degree, which contribute to better thermal resistance and mechanical properties[1][2].

Table 2. Thecrystallinity index of pineapple fiber

Pineapple fiber	Crystallinityindex(%)
Untreated	64.21
Alkali treated	72.79
Bleached (microcellulose)	73.48

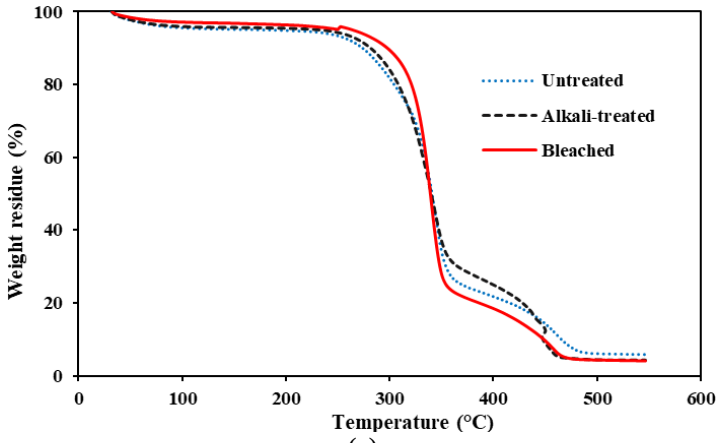
3.4 Thermal Analysis

Fig. 4(a) and 4(b) depict the ThermogravimetricAnalysis (TGA) and Derivative Thermogravimetry (DTG) curves of pineapple fibers (untreated, alkali-treated and bleached). In compliance with Fig. 4(a), all the TGA curves are separated into two weight-loss regions. An initial weight loss reveals below the temperature of 150°C, with the weight loss for untreated, alkali-treated, and bleached being 3.21%, 4.34%, and 4.87%, respectively. Firstly, this fact corresponds to the evaporation of water trapped in the amorphous phase of the cellulose and the initial decomposition of lignin[38]. Secondly, significant weight loss occurs at the temperature range of 240ºC to 377ºC, which is attributed to the decomposition of hemicellulose and the polymerization of the cellulose chain [39]. This result is in line with the previous studies[33][40]that the thermal decomposition of lignin, hemicellulose, and cellulose occurs at temperatures of below 200°C-700°C, 220°C-315ºC, and 315°C-400°C, respectively. Finally, TGA curves over the temperature of 480°C show the residual weight loss (5.84% for untreated, 4.27 for alkali-treated, and 4.08 for bleached). Table 3 reveals that a higher residue of untreated fibers compared to treated fibers (alkali and bleaching) is attributed to lignin and hemicellulose, which induce the formation of char. Meanwhile, alkali and bleaching treatments remove hemicellulose and lignin, resulting in a lower amount of residue. This finding is similar to that reported by Khenblouche et al. [39]. They stated that the fiber after bleaching treatment revealed a lower residue compared to untreated fiber. This is due to hemicellulose and lignin decomposition. Most cellulose, hemicellulose, and partial lignin decompose at a temperature of 600°C[41] and the residue is char, ash, etc.

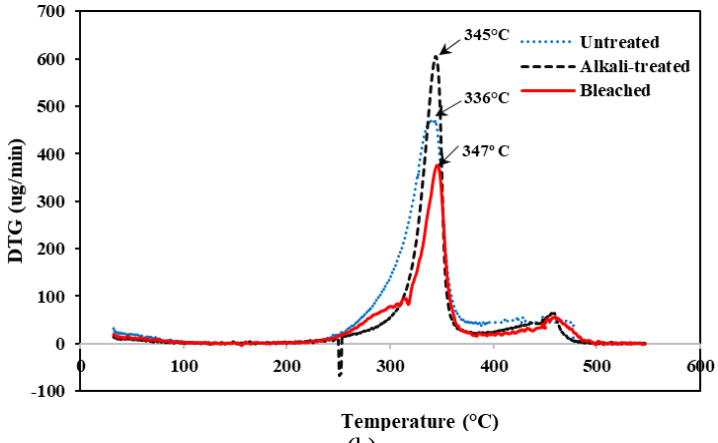
The DTG curve of untreated pineapple fibers reveals the maximum exothermic peaks at 336ºC. These values are attributed to the thermal decomposition of cellulose and lignin. The maximum exothermic peak of alkali-treated and bleached occurs at 345ºC and 347°C, respectively.Alkali and bleached treatments remove hemicellulose, lignin, and surface impurities without changing the structure of cellulose. These treatments enhance the thermal stability of micro cellulose due to the greater crystallinity index (Table 2). The greater crystallinity index indicates an increase in the regularity of the cellulose structure. As a result, microcellulose requires more heat to experience thermal degradation, and heat transfer becomes difficult [42]. The difficulty of heat transfers in micro cellulose causes higher thermal stability [43]. Therefore, it can be concluded from these results that the micro cellulose reveals higher thermal stability compared to the untreated fiber.

Table 3. TGA/DTG of pineapple fibers

Pineapple fiber	Residue (%)	Tmax decomposition (ºC)
Untreated	5.84	336
Alkali treated	4.27	345
Bleached (micro cellulose)	4.08	347



(a)



(b)

Fig.4. (a) TGA and (b) DTG curves of pineapple fibers (untreated, alkali-treated and bleached).

The crystallinity index and TG/DTG analysis of microcellulose Pineapple Leaf Fibers (PALF) obtained were compared with other microcellulose PALF, as reported in Table 4. These results show that the crystallinity index of microcellulose in this study is higher than that of microcellulose PALF as reported in [3], while the maximum thermal decomposition of microcellulose PALF is close to the maximum thermal decomposition in [4].

Table 4. The crystallinity index and TGA/DTG of microcellulose PALF

Fiber	Crystallinity index (%)	Tmax Decomposition (°C)	Ref
Microcellulose PALF	73.48	347	This study
Microcellulose PALF	43.05 ± 2	-	[3]
Microcellulose PALF	-	349	[4]

4 Conclusion

Micro cellulose was successfully isolated from pineapple leaf fiber using alkali and bleaching treatments. The peak diffraction intensity of the crystalline phase was observed at 2θ = 18.29º, 22.7º, and 34º with a crystallinity index of 73.48%. The FTIR spectra revealed the cellulose bond in the microcellulose. Microcellulose also has high thermal stability with a maximum thermal decomposition of 347°C. Based on the results, pineapple leaf fibers in Kampar district, Riau could be a source of microcelluloseas an eco-friendly, Renewable, and sustainable material for future industrial applications.

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