Isolation and Characterization of New Cellulosic Microfibers from Pandan Duri (Pandanus Tectorius) for Sustainable Environment

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Isolation and Characterization of New Cellulosic Microfibers from Pandan Duri (*Pandanus Tectorius*) for Sustainable Environment

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ABSTRACT

Cellulose is the main component of natural fibers whose content varies greatly depending on the type of plant and its treatment. Therefore, it is necessary to examine the effect of chemical treatment on natural fiber properties. This study successfully extracted the content from Pandan duri (*Pandanus tectorius*) fiber through alkalization, bleaching, and acid hydrolysis. The effect of these chemical treatments on the characteristics of fiber surface, shape, chemical composition, crystallinity, and thermal properties was examined and analyzed. Subsequently, the cellulose components in the structure and the removal of lignin groups were characterized by Fourier transform infrared (FTIR) spectroscopy spectrum analysis. The surface morphology analysis was done by scanning electron microscopy (SEM) that showed the raw fiber surface was still in the structure of bundles. However, it was decomposed into cellulose fibrils after chemical treatment with a diameter of about 2 µm – 20 µm. The chemical composition for cellulose content increased by 90.5%, while hemicellulose decreased by 89.6% after acid hydrolysis treatment. Also, X-ray (XRD) analysis showed crystallinity increased from 39.5% for raw fibers to 67.7% after the hydrolysis. Thermal gravimetric analysis (TGA) showed higher degradation temperature of micro cellulose offered better thermal stability compared to raw fibers. In conclusion, the cellulose from *Pandanus tectorius* fiber can be used to reinforce biocomposites as an alternative to synthetic fibers for sustainability of environment.

摘要

纤维素是天然纤维的主要成分，其含量因植物类型及其处理方式的不同而有很大差异. 因此，有必要研究化学处理对天然纤维性能的影响. 本研究通过碱化、漂白和酸水解，成功地从盘状杜丽(Pandanus tectorius)纤维中提取出含量. 研究和分析了这些化学处理对纤维表面特性、形状、化学成分、结晶度和热性能的影响. 随后，采用傅里叶变换红外光谱(FTIR)对纤维素组分的结构和木质素基团的去除进行了表征. 通过扫描电子显微镜(SEM)进行表面形貌分析，表明原始纤维表面仍为束状结构. 然而，经过化学处理后，它被分解为直径约为2µm–20µm的纤维素原纤维. 酸水解处理后，纤维

KEYWORDS

Pandanus tectorius; chemical treatment; cellulose fiber; crystallinity; thermal stability; chemical composition

关键词

盖盘菌; 化学处理; 纤维素纤维; 结晶度; 热稳定性; 化学成分

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素含量的化学成分增加了90.5%，半纤维素含量下降了89.6%. X射线衍射(XRD)分析表明，水解后纤维结晶度从原纤维的39.5%提高到67.7%. 热重分析(TGA)表明，与生纤维相比，纤维素降解温度越高，热稳定性越好. 综上所述，为了环境的可持续性，覆盆子纤维中的纤维素可以用来增强生物复合材料，作为合成纤维的替代品.

Introduction

Residues from agricultural waste are not optimally used due to lack of waste utilization and management to improve their use-value, therefore, they are usually buried or burnt (Sanjay et al. 2018; Vinod et al. 2020). The primary components of plant residues in natural fibers are natural biopolymers, such as cellulose, hemicellulose, and lignin (Manimaran et al. 2018; Yang et al. 2007; Zhang et al. 2015). Cellulose can be used as reinforcement in specific applications like food packaging (Edi et al. 2019, 2019, 2018; Mahardika et al. 2019; Mochamad et al. 2018; Rangappa et al. 2021), biomedical field, and electro-conductive (Ul-Islam et al. 2015). The superior characteristics of its fibers include high mechanical strength or stiffness, biodegradability, good thermal resistance properties, recyclability, abundant availability in nature, low density, and cost (Mahmud et al. 2021; Masmoudi et al. 2016; Nagarajan et al. 2021). With these advantages, it can be used as a reinforcement and filler in biocomposite polymers, structural applications, insulation materials, and automotive applications, such as car panels as well as other interior applications (Adesina et al. 2019; Madhu et al. 2020; Muthu Chozha Rajan et al. 2020; Rangappa et al. 2022; Sari et al. 2021).

Several previous studies that have utilized this component in the manufacture of biocomposites include cellulose nanocrystals with chitosan (Adel et al. 2019), polyester with bacterial cellulose reinforcement (Panaitescu, Nicoleta Frone, and Chiulan 2016), PLA with cellulose nanocrystal reinforcement (Yin et al. 2017), and others. Recently, various new sources of cellulose have been identified and characterized, namely cellulose in bark *Thespesia populnea* fiber (Kathirselvam et al. 2019), *roselle* fiber (Kian et al. 2017), *oil palm mesocarp* (Chieng et al. 2017), *Acacia nilotica* L. (Kumar et al. 2020), *Citrullus lanatus* (Khan et al. 2020), *Phaseolus vulgaris* (Babu et al. 2020), *Vachellia farnesiana* (Vijay et al. 2020), *Momordica Charantia* (Anish et al. 2020), *Eucalyptus* spp. (Carrillo-Varela, Pereira, and Teixeira Mendonça 2018) and other fibers. Pandan duri *(Pandanus tectorius)* grows a lot in tropical areas like Indonesia and its fiber potentially serves as auspicious source of cellulose, considering its abundant availability in the country.

Several chemical treatments that have been successfully used for cellulose extraction include alkalization, bleaching, and acid hydrolysis (Afolabi et al. 2019; Hairul et al. 2019a; Ilyas, Sapuan, and Ishak 2018; Mahardika et al. 2018; Owolabi and Megat-Yusoff 2018; Sanjay et al. 2019). Ilyas, Sapuan, and Ishak 2018 reported that alkalizing treatment succeeded in increasing the cellulose content of sugar palm fibers by 46.7% compared to those without treatment (Ilyas, Sapuan, and Ishak 2018). Previous studies on the extraction of cellulose from *Pandanus tectorius* (Screwpine) only used alkali and combined alkali-bleach treatment. (Owolabi, Sri Melor Megat-Yusoff, and Syahmi Hamizol n.d.). The results obtained a maximum cellulose content of 78% at the concentration of 2 wt% NaOH for 120 minutes and 2 wt% $NaClO_2$ (Owolabi, Sri Melor Megat-Yusoff, and Syahmi Hamizol n.d.).

Furthermore, the process of extracting the component using this study's method has not been examined by previous research teams. This study aims to determine the characteristics of the fiber before and after chemical treatment. The fiber was extracted by different chemical treatments, such as alkalization, bleaching, and acid hydrolysis. Furthermore, it was analyzed for XRD, FTIR, thermal properties, and its surface morphology was observed using SEM. This study will help compare Pandanus tectorius fiber's characteristics with other natural fibers and determine the optimal cellulose content with various chemical treatments. After being processed, the material properties of the species showed that the development of the business was minimal.

Materials and methods

Materials

The primary material used in this study was Pandan duri (PD) (*Pandanus tectorius*) leaves as the primary source of cellulose fiber. The leaves were obtained from a plantation area in Harau, Kabupaten Lima Puluh Kota, West Sumatera. The thorns on its edges were cleaned and cut 5–10 cm long, it was subsequently dried in the sun for four days with a moisture content of approximately 9 to 10%. Furthermore, the leaf's fibers were decomposed using a blender.

The Pure Analysis chemicals used include sodium hydroxide (NaOH 98% Sigma-Aldrich brand, HCl (KOH brand Millipore), sodium chlorite ($NaClO_2$ brand Pubchem), and glacial acetic acid (CH_3COOH).

Fiber extract

The chemical methods used for fiber extraction include alkalization, bleaching, and acid hydrolysis treatment. Lignin and hemicellulose were extracted by alkaline treatment of 5% (w/v) NaOH for 2 hr at 80°C on a hotplate. The brown-colored fibers were washed until they were alkali-free with a pH of 7.

Alkalized PD fibers were bleached using a solution consisting of equal parts (v:v) acetic buffer (27 g NaOH and 75 mL glacial acetic acid, diluted to 1 L distilled water), and dilute sodium chlorite (1.7 wt% NaClO2). This treatment was repeated twice for 1 hr at 80°C, resulting in white PD fibers which were subsequently hydrolyzed with 5 M HCl solution for 1 hr with a ratio of fiber weight to a solution of 1:20. This process was carried out at a hotplate temperature of 50°C with 200 rpm. The hydraulic fibers in particles are known as Cellulose Micro Fibers (CMF) Pandan Berduri (PD). The extraction process is shown in Figure 1.

Analysis of chemical composition

Chemical composition analysis was based on the test method developed by Van Soest. Natural fiber consists of fiber soluble in neutral detergent (*Neutral Detergent Fiber*/NDF), soluble in acid detergent (*Acid Detergent Fiber*/ADF), hemicellulose, cellulose, and lignin. The Van Soest method can determine the content of cellulose, hemicellulose, and lignin in the PD fiber.

Figure 1. PD fiber leaves (a), dried PD fiber after blending with a blender (b), alkalized PD fiber (c), bleaching PD fiber (d), hydrolyzed PD fiber (e).

Scanning electron microscopy (SEM)

SEM observations were used to determine the shape of the fiber surface before and after the chemical treatment. Meanwhile, the surface morphology of the cellulose was observed using Scanning Electron Microscopy (SEM), Model: S-3400 N, Hitachi, Ltd., Japan, with a voltage of 20 kV and a current of 8 mA probe. The test sample was placed on the SEM sample stub. Meanwhile, the preparation which was previously coated with gold to reduce the electron charge and obtain image clarity.

X-ray diffraction (XRD)

The crystallinity index of the fibers before and after chemical treatment was measured using the X-ray diffraction (XRD) technique through X'pert PROPANalytical (Model: PW3040/60) with Cu Kα radiation (λ = 0.1542 nm). The spectrum was recorded between 5° and 50° at 40 kV and 30 mA. The crystallinity index (I_{cr}) was calculated using this formula:

$$CI=[(I_{002}-I_{am})/I_{002}]x100 \quad (1)$$

Where I_{002} = Intensity for 2θ = 22.3°, which indicated the crystal region. I_{am} is an amorphous region at Intensity 2θ = 18° (Segal et al. 1958).

Fourier transform infrared (FTIR)

FTIR characterization using PerkinElmer FTIR spectrometer (Frontier instrument, USA). This test was used to identify free functional groups from the fibers before and after chemical treatment. Spectrum scans were recorded with 4 cm^{-1} over a wavenumber range of 4000–600 cm^{-1} (Hairul et al. 2019a).

Thermogravimetric analysis (TGA)

Measurement of the fiber's thermal stability without and after chemical treatment was carried out using the DTG-60 SHIMADZU (Kyoto, Japan). Furthermore, thermal analysis was conducted in a nitrogen atmosphere at a flow rate of 50 mL/min, a heating rate of ten °C/min, and a range temperature of 30–550°C.

Results and discussion

Chemical composition

The chemical composition of the PD fiber is presented in Table 1. According to the table, the cellulose content was 43.21% which is lower compared to other natural fiber sources, such as *Eucalyptus* (50.30%) (Carrillo-Varela, Pereira, and Teixeira Mendonça 2018), banyan tree roots (67.32%) (Ganapathy et al. 2019), and sugar palm fibers (43.88) (Ilyas, Sapuan, and Ishak 2018). This is due to its several irregular amorphous areas (Abhilash et al. 2018; Saravanakumaar et al. 2018; Senthamaraikannan et al. 2018).

After the 5% NaOH alkalizing treatment, the cellulose content increased by 64.92%, while the lignin content decreased by 68.10% compared to raw fiber. The reduction in this content was due to the dissolution by alkalizing treatment. The bleaching process succeeded in dissolving hemicellulose with a reduction of 89.62% compared to raw fiber. Most of the lignin and hemicellulose were extracted from the pandan duri fiber, hence, increasing the degree of cellulose crystallinity as well as the strength and thermal properties of the fiber (Asrofi et al. 2018; Ilyas, Sapuan, and Ishak 2018).

Table 1. Chemical Composition and Crystallinity Index (I_{cr}) Pandan Duri Fiber.

Fiber Treatment	Cellulose (%)	Lignin (%)	Hemicellulose (%)	I_{cr} (%)
Raw PD Fiber	43,21	5,11	38,81	39,49
Alkalized PD Fiber	71,26	1,63	17,72	54,89
PD Fiber Bleaching	77,86	1.69	15,08	61,02
Acid Hydrolyzed PD Fiber	82,30	7,04	4,03	67,65

Morphological analysis

The fiber's surface looks rough, as shown in Figure 2a with a magnification of 750 times. According to the image, the diameter of its bundle before chemical treatment was ± 20 µm. Figure 2a showed the presence of fibril-shaped fiber bundles that are still bound to lignin, pectin, and hemicellulose (Mahardika et al. 2018). Meanwhile, Figure 2b showed a smoother surface due to alkalization treatment. The alkalization process can remove impurities, such as wax and fatty substance on the surface (Kathirselvam et al. 2019). After the bleaching process, the bundle's length, as well as the lignin and hemicellulose levels were reduced, as shown in Figure 2c. These results were supported by testing the chemical composition of the fiber according to previous studies. Meanwhile, Figure 2d showed a very smooth fiber surface with the shape of fibrils after acid hydrolysis treatment. The results indicated that the treatment increased the cellulose content by 90.5% compared to raw fiber.

Infrared spectroscopy analysis

The hydrophilic nature of cellulose with free hydroxyl groups regulates the water absorption of the fiber and its water absorption ability (Asrofi et al. 2017). The fiber's functional groups were identified through FTIR spectrum results, as shown in Figure 3. The wavelength in the range of

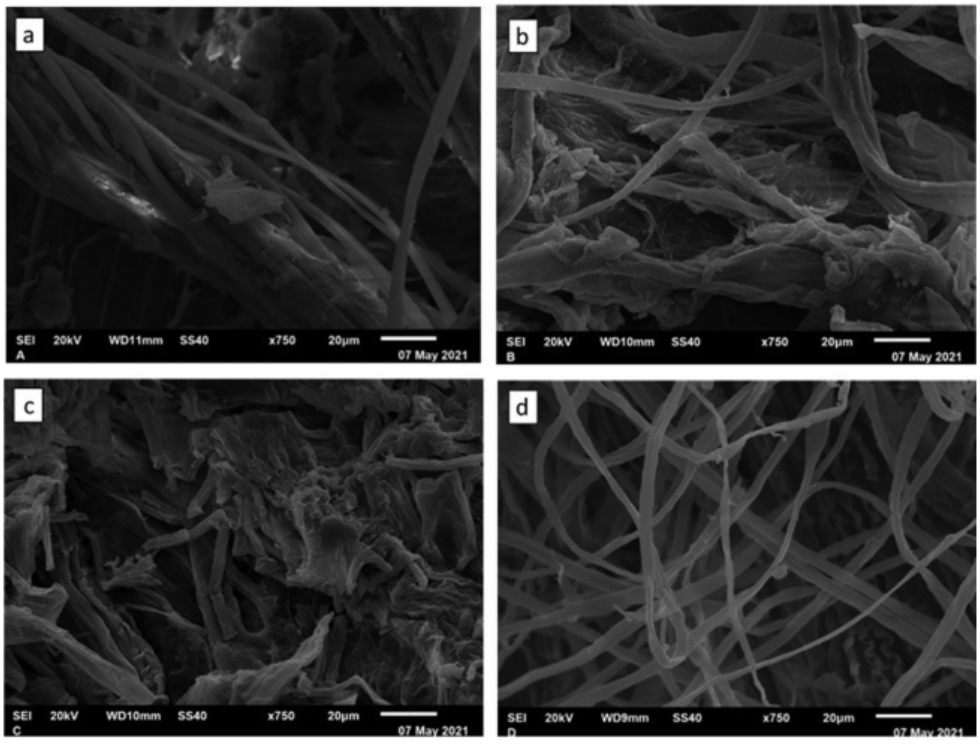

Figure 2. Analysis of SEM of Pandan duri raw fiber (a), Alkalization (b), Bleaching (c), Acid hydrolysis (d).

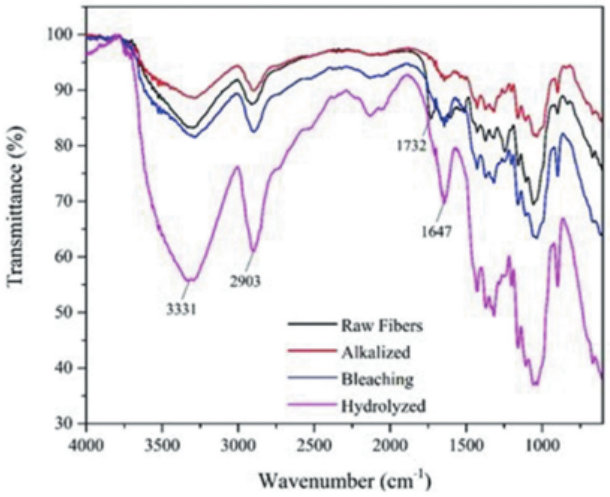

Figure 3. FTIR spectra of all pandan duri fiber samples.

3331 cm^{-1} showed hydroxyl group (O–H) of cellulose compounds in the structure (Zhang et al. 2015). The presence of cellulose was also indicated by the bands of 2903 cm^{-1} which is the C–H strain (Anish et al. 2020). According to these results, the intensity of the band was reduced after chemical treatment compared to raw fiber. The strain of the carbonyl group (C–O) in lignin and hemicellulose was shown at wavenumbers 1732 cm^{-1} and 1647 cm^{-1} (Yang et al. 2007). Figure 2 showed the disappearance of the peak at a wavenumber of 1732 cm^{-1} which indicated that the treatment succeeded in extracting the lignin content. These results were also supported by chemical composition testing (Table 1) and previous studies (Mahardika et al. 2018). The following peak, identified in the region 1400 cm^{-1} to 1425 cm^{-1} for both raw and chemically treated fibers, confirms the vibrational stretching of CH_2 denotes the presence of aromatic rings in hemicellulose (Vijay et al. 2019). The vibration of C-O attributed to the acetyl groups in lignin is shown by a small intensity peak in the region 1200 cm^{-1} to 1225 cm^{-1} (Vinod, Sanjay, and Siengchin 2021). The last intensity peak, which can be found in the range of 1025 cm^{-1} to 1035 cm^{-1}, reflects the stretching and vibration of C-H in the presence of cellulose (Shravanabelagola Nagaraja Setty et al. 2020).

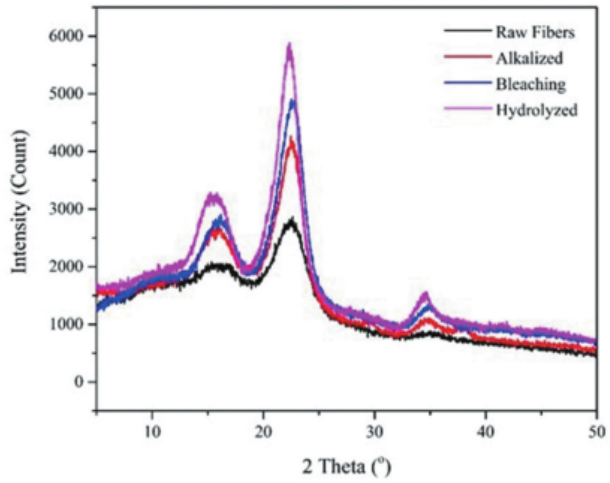

Figure 4. XRD curves of all pandan duri fiber samples.

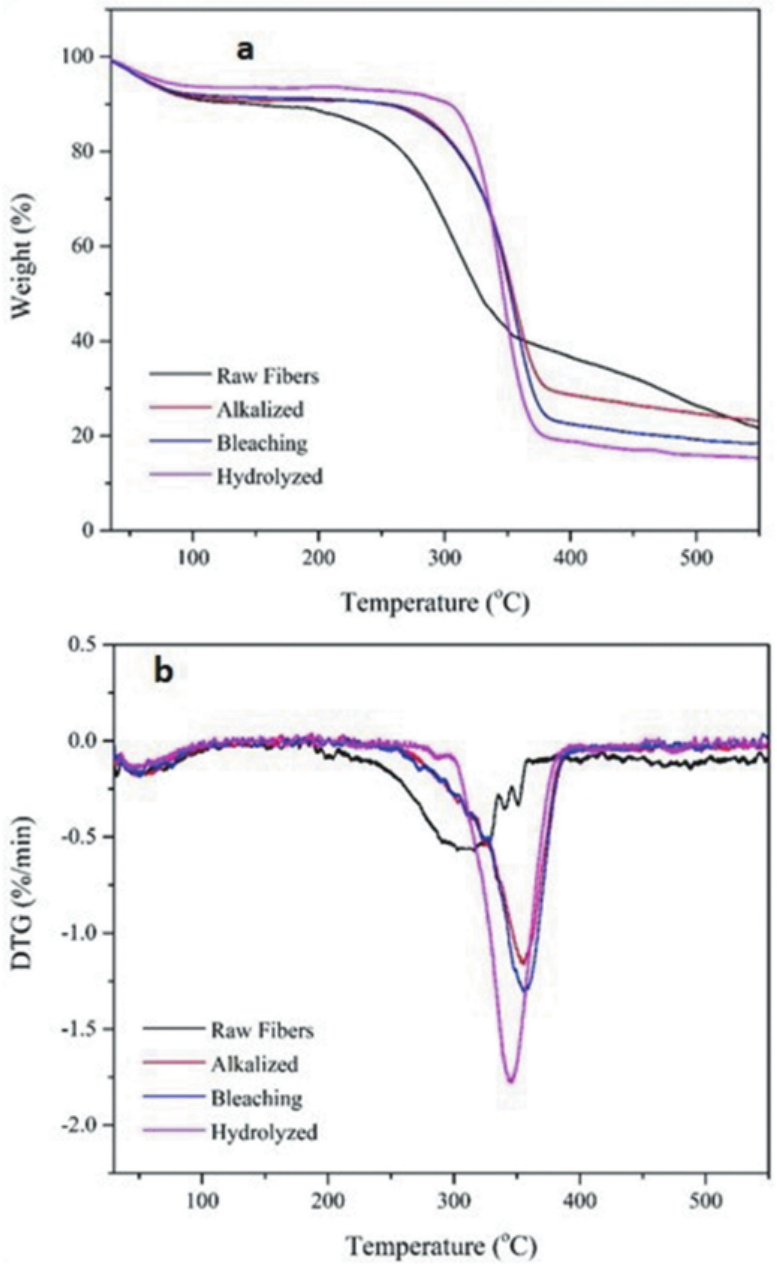

Figure 5. TGA curve (a), DTG curve (b) of raw fibers, alkalized, bleaching, and hydrolyzed.

X-ray diffraction (XRD) analysis

The XRD curve of the sample is shown in Figure 4. According to this analysis, the crystallinity index of the fiber can be calculated before and after chemical treatment. The X-ray diffraction pattern was similar to the previous study's, which showed the intensity diffraction peaks around 16.2°, 22.3°, and 34.6° for Pandan Duri fiber as a typical polymorph of cellulose I (Mahardika et al. 2018; Supian et al. 2020). The crystallinity index was calculated using the Segal equation (Segal et al. 1958). The 22.3° indicated the crystalline plus amorphous regions of cellulose and and 18°peaks is relative to the amorphous phase. The crystallinity index value is shown in Table 1. According to the measurement results, the crystallinity index was 39.49%. Meanwhile,

there was a significant increase in the value (71.31%) after acid hydrolysis treatment. This is due to the reduced non-cellulose amorphous structure, and the increased crystalline structure (Hairul et al. 2019a). These results were supported by testing the chemical composition of the fibers (Table 1) and previous studies (Ilyas, Sapuan, and Ishak 2018).

Thermogravimetric analysis (TGA)

The thermal stability was tested before and after chemical treatment by using thermogravimetric analysis. Figure 5a,b showed the thermogravimetric analysis (TGA) and derived thermogravimetric curves (DTG). There were three regions of temperature drop which occurred around 100°C, 250–400°C, and 400–550°C, respectively. The weight loss of about 100°C was probably due to the evaporation of water (Abral et al. 2021). This is indicated as a small trough on the left side of the DTG curve. All samples showed a large weight loss in the 250–400°C region as the cellulose was depolymerized and damaged (Anish et al. 2020). After the alkalization, bleaching, and hydrolyzation treatments, the degradation temperatures of raw fiber were 309, 354, 357, and 346°C, respectively, as shown in the DTG curve (Figure 5b). Therefore, it had thermal stability than chemically treated fiber.

Based on 400–550°C (region 3), all samples completely decomposed to ash (Vijay et al. 2020). However, raw fiber had more residue compared to the chemically treated counterpart due to the leftover non-cellulose content. A similar trend can be observed in previous studies (Babu et al. 2020; Mahardika et al. 2018). The most significant TGA result was the higher bleaching temperature of the fiber degradation (357°C) which showed superior thermal stability over the raw fiber (309°C).

Conclusion

This study aims to extract cellulose from pandan duri fiber through alkalization, bleaching, and acid hydrolysis chemical treatments. According to the SEM observation, after acid hydrolysis treatment, the fiber's surface was smooth and in the form of long fibrils compared to the raw fiber, which was in the form of bundles with a rough surface. The FTIR spectrum Intensity indicated the presence of cellulose-related functional groups in the structure. Furthermore, the XRD results showed that the highest crystallinity index (67.65%) was indicated by fiber treated with acid hydrolysis. Meanwhile, the bleached sample showed the most increased thermal stability with a maximum degradation temperature of 357°C. Therefore, the results showed the superior characteristics of pandan duri fiber in its cellulose content compared to others. From the results, it is noteworthy to mention that the cellulosic microfibers from Pandan Duri (Pandanus tectorius) can be used to produce products for different applications such as such as personal protective textiles, skin grafts, tissue engineering scaffolds, and wound dressings, etc., and also it can be a competitive nanofabrication filler.

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Author contribution

"All the authors contributed equally for data curation, formal analysis, methodology, conceptualization, project administration, resources, investigation, supervision and writing."

Animal research (ethics)

"This article does not contain any studies with human participants or animals performed by the authors."

Consent to publish (Ethics)

"The authors give the consent to publish this research work in the journal."

Consent to participate (Ethics)

"The authors give the consent to participate this research study."

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

"All data generated or analyzed during this study are included in this published article."

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