



Fabrication and Characterization of Pineapple Leaf Fiber (PALF) as Candidate of Composite Reinforcing Material

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Abstract

Pineapple leaf is a waste product of pineapple cultivation which may leads to environmental issues. Pineapple leaf fibre (PALF) is an important natural fibre with high cellulose content that exhibits high mechanical properties which is high specific strength and stiffness which may vary for each cultivar. Besides, isolation of cellulose from lignocellulosic fibre become more crucial. In this study, isolation of cellulose were undergo by alkaline treatment In that order, concentration of NaOH 0.5% at 121oC for 60 min and bleaching teatment using NaClO 10% at 121oC for 30. The PALF and treated fiber were characterized using Tensilon, Scanning Electron Microscopy, Fourier Transmission Infrared Spectroscopy. Based on results, 5% NaOH (60 min) and bleaching process by using 10% NaClO (30 min) in autoclave succeed to remove pektin, lignin, and unwanted components. The surface morphology showed high surface area, however still remaining small amaount of pektin and lignin was confirmed from FTIR and SEM images.

Keywords: Characterization, Pineapple leaf fiber, Composite

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INTRODUCTION

Pineapple leaf fiber (PALF) is one type of fiber obtained from pineapple leaves by combination of mechanical and chemical process. Pineapple plants (*Ananas comosus*) are fruit plants that have been widely known by the public. Pineapple plants can grow well especially in tropical countries. The availability of pineapple leaves which is quite abundant in Indonesia has not been utilized optimally by farmers due to the lack of knowledge about post-harvest pineapple leaf processing techniques to make products with high economic value (Soeprijanto et al., 2021). One alternative for utilizing this waste is to process it into natural fiber by utilizing leaf fibers into staple fiber (long fibers). Staple fiber has several broad application in the automotive, textile, composite material and other fields.

PALF consist of about 80% cellulose, 6-12 % hemicellulose, 5-12% lignin (Fan et al., 2022; Gaba et al., 2021). Cellulose is the largest component in PALF, exist in two distinct forms; the crystalline and amorphous phases. In the crystallin phase, there are bundle of microfibrils, made of assemblies of (1-4) β -D-glucan chains that are strongly hydrogen-bonded (Cozier, 2014). The amorphous phase consist of randomly arranged cellulose and hemicellose that contribute insignificantly to the structural and mechanical stifness of fiber. previous study reported that removal of amorphous phase through hydrolsis leading a higher degree of ordered crystalline regions with ultimate goal of enhancing the cellulose isolation (Gaba et al., 2021).

Previous study shown that the chemical treatment of natural fibers can not only enhance their surface morphology but improve the mechanical properties of the fiber (Gholampour & Ozbakkaloglu, 2020). The PALF chemical treatment are developed by the process of steam treatment correlated with alkali treatment. Steam treatment is hydrothermal process of biomass treatment, involving of the application of steam high pressure, and which can be performed by batch or continuous reactor. The biomass is exposed to saturated steam (180-230oC) for 20-40 min. Generally the steaming method results in hydrolysis of glycosidic bond in the hemicellulose and, to lesser extent in the cellulose (Cherian et al., 2011). The microfiber from PALF has a high surface area and porosity, morphological properties of the fiber, better mechanical properties compare to macrofiber, and has an ability of heat resistant (Barhoum et al., 2019). It would makes this natural fiber have wide applications, one of which is as a reinforce for composite.

Composite is a material formed from a combination of two or more materials with different mechanical properties of the forming materials, where one material is a filler (matrix) and the other is a reinforcing phase (reinforcement) (Lalmangaihzuali et al., 2019). Natural fibers are crucial materials in developing eco-friendly, biodegradable composites to address pressing environmental issues. These composites offer a promising solution due to their excellent mechanical properties, low density, affordability, high toughness, reasonable specific strength, recyclability, biodegradability, renewability (Gnanasekaran et al., 2021). Therefore PALF is suitable for reinforcing candidate material.

Recent studies have been explored the development of eco-friendly composites reinforced with pineapple leaf fiber (PALF). The mechanical properties of PALF-reinforced polyethylene composites, emphasizing factors such as fiber size, loading percentage, and orientation. It notes that PALF-LDPE composites are eco-friendly and demonstrate superior performance compared to other cellulose-fiber-reinforced LDPE systems (Sethupathi, Murugan, Khumalo, Mandla, Skosana, Sifiso, Muniyasamy, 2023). (Duangsuwan et al., 2023) reported that low-carbon composites using biobased poly(butylene succinate) (PBS) reinforced with well-aligned PALF. The study highlights the potential of PALF as a reinforcing fiber to improve composite mechanical properties while reducing environmental impact and production costs. While green composite used of recycled plastic bags combined with pineapple fibers to create green composites. The research emphasizes the mechanical properties of these composites, suggesting potential applications in various industries (Gandara M, Zanini NC, Mulinari DR, Saron C, 2022). The recent advancements in PALF-reinforced polymer composites, discussing their properties and potential applications in sustainable materials. These studies underscore the growing interest in utilizing PALF for developing sustainable, eco-friendly composite materials with enhanced mechanical properties (Xiao Hanyue, Mohamed Thariq Hameed Sultan, Muhammad Imran Najeeb, 2024).

The pineapple leaf fiber (PALF) as a sustainable and eco-friendly natural fiber for composite reinforcement by isolating microscale cellulose from PALF, the study leverages its superior characteristic to develop advanced composites. The aim of this study, to produce microscale cellulose produce as candidate of reinforcing material for composite. The resulting microcellulose were characterized by TENSILON to identify the tensile strength, Scanning Electron Microscopy (SEM), and Fourier Transmission Infrared Spectroscopy (FTIR).

METHOD

Materials

The pineapple fiber were purchased from farmer in Subang, Indonesia. The reagents used for chemical process were NaOH (pa) and Sodium Hypochlorite. Distilled water used for all the process. The apparatus for pretreatment of pineapple fiber was an Autoclave. The characterization were carried out using the Transmission Electron Microscope (JEOL JCM-

7000 Neoscope), Fourier Transmission Infrared (Perkin Elmer L1600300 Spectrum Two Lita), and Tensilon (RTG-1310).

Chemical Treatment

Pineapple leave fiber were treated by adding 5% NaOH at 121°C for 60 min in autoclave. The bleaching process was carried out by adding NaClO 10% at 121°C for 30 min in autoclave then washed until neutral pH and dried at room temperature. The dried were cut into small pieces using cutter and mechanical blender. The treated fiber will undergo further characterization.

Characterization

Chacaracterization of pineapple fiber, fisrt by using Tensilon to measure the tensile strength, second by using Fourier Transform Spectroscopy, The powder sample were pelleted and mixed with KBR and recorded the spectrum range 400 cm^{-1} to 4000 cm^{-1} . and the last is Scanning Electron Microscopy used to analzed the morphology of fiber.

RESULTS AND DISCUSSION

Effect of Alkaline Treatment

PALF was extracted through the mechanical and chemical treatment, each affecting the fiber's surface characteristics and mechanical performance. The chemical treatment of PALF was undergo under alkaline treatment by degumming and bleaching process. degumming process is carried out by soaking the PALF in a solution of Sodium hydroxide (NaOH) 5% at 121°C for 60 min in autoclave (Fig 2). This process aims to remove hydrophilic components such as pectin, lignin, hemicellulose and other unwanted contents from the surface of fiber. The mechanism of alkaline treatment is correlated to saponification of intermolecular ester bond cross-linking lignin and part of hemicellulose. The reduction of this cross-linking would increase the porosity of PALF, the used of 5% of NaOH was the suitable concentration to the alkaline treatment (Amirulhakim et al., 2021) for removing these components.



Figure 1. The Untreated PALF (a) Treated PALF (b)

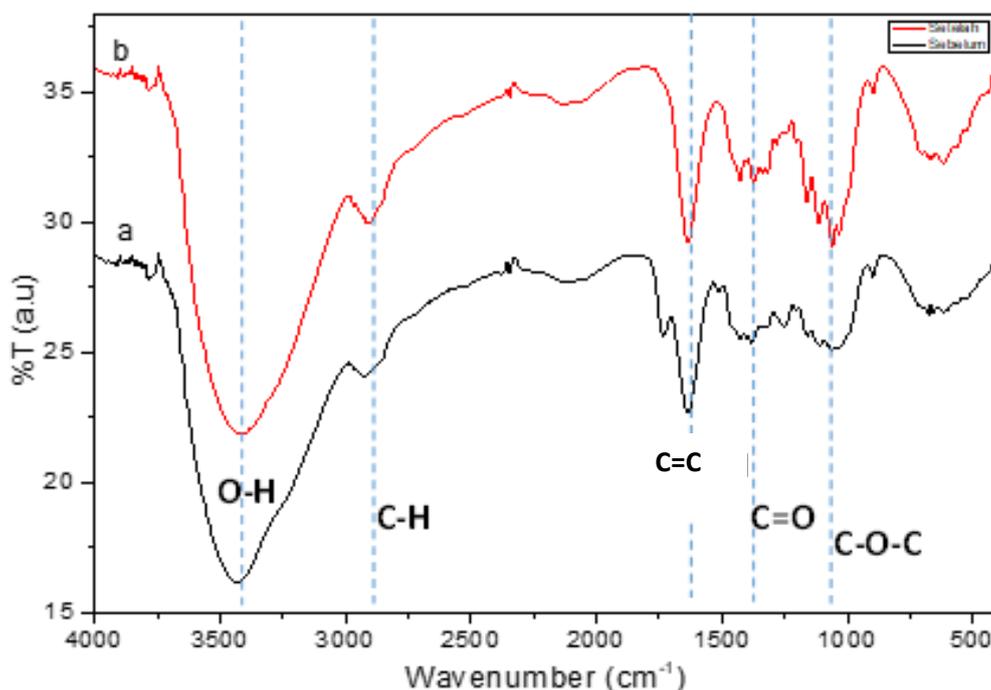
Furthermore, the bleaching process or known as oxidative delignification by using Sodium Hypoklorite 10% NaClO. The PALF was soaked in 10% NaClO (sodium hypochlorite) solution then placing it in an autoclave for 30 minutes at a temperature of 121°C and the final stage is washing and drying. Sodium hypochlorite (NaClO) is used for bleaching. The indication of this process visually is the color alteration from brown to white (Fig 2). The mechanism reaction is Sodium Hypoklorit may react with the side chain of carbonyl and carbon-carbon double bond lignin to further oxidative degradation, which leading to lignin removal and unwanted components on the surface (Septevani et al., 2018)(Gnanasekaran et al., 2021). This is further supported by the pH value being close to 7, indicating that sodium hydroxide was released along with the impurities (Table 1)

Table 1. pH of PALF After Degumming and Bleaching Process

No	Sample	pH
1	Degumming	7.4
2	Bleaching	7.1

PALF Analysis by Using FTIR

The FTIR analysis used to identify the functional group of treated and untreated PALF. Based on the IR spectra (Fig. 2). The FTIR results of PALF examine the changes in the chemical bonds of PALF before and after surface modification. Identified functional groups in the chemical structure included alkanes, alcohols, aromatics, and esters. The infrared absorption peaks at 1370 cm^{-1} and 1730 cm^{-1} are linked to the benzene ring (C=C) in lignin and the acetyl group (C=O) in hemicellulose, respectively. Additionally, the sharp peak at 1070 cm^{-1} is attributed to the pyranose ring structure (C-O-C) or the β -glycosidic bonds in celluloses. In untreated PALF, these three peaks were clearly visible. However, the peaks at 1370 cm^{-1} and 1730 cm^{-1} diminished significantly in treated fibers, while the peak at 1070 cm^{-1} remained prominent. The strong 1070 cm^{-1} peak observed treated fibers confirms that the used of NaOH for 60 minutes, retained a higher cellulose content compared to untreated PALF. This indicates that the process effectively removed lignin and hemicellulose (Gnanasekaran et al., 2021). Infrared absorption stretching in the range of $3000\text{--}3700\text{ cm}^{-1}$ was observed, corresponding to the hydroxyl (O-H) groups of carboxylic acids and alcohols present in lignin (Yudhanto et al., 2021).

**Figure 2.** FTIR Spectra of Treated and Untreated PALF

However, while the content of lignin and hemicellulose was nearly eliminated, the cellulose content may have degraded, as indicated by the intensity of certain peaks. The C-H peak intensity was low in the range of $2800\text{--}2900\text{ cm}^{-1}$, suggesting the loss of alkyl groups due to cellulose degradation. Additionally, the low intensity of the C=O stretching in carboxyl groups ($1730\text{--}1750\text{ cm}^{-1}$) indicates partial degradation of cellulose. This peak, often associated with hemicellulose, can diminish, signaling degradation or removal. The intensity ratio between the peaks at 1375 cm^{-1} and 1335 cm^{-1} may also reflect a loss of crystallinity in cellulose, which is typically associated with its degradation.

Scanning Electron Microscopy (SEM)

In the SEM analysis of pineapple leaf fiber (PALF), both long fibers and microfibrils display distinct characteristics that provide insight into their potential applications, particularly in composite materials. The SEM images (Fig 2) of long PALF show a relatively smooth and elongated structure, which is typical of natural fibers. These fibers retain their bundle form, but the presence of impurities such as lignin, pectin, and hemicellulose can be observed along the surface. These impurities contribute to a rough texture and might hinder the fiber's ability to bond with matrices in composite materials. However, the long fibers are beneficial in terms of mechanical reinforcement due to their large length, which can enhance the strength and stiffness of composites when properly treated and incorporated (Gaba et al., 2021).

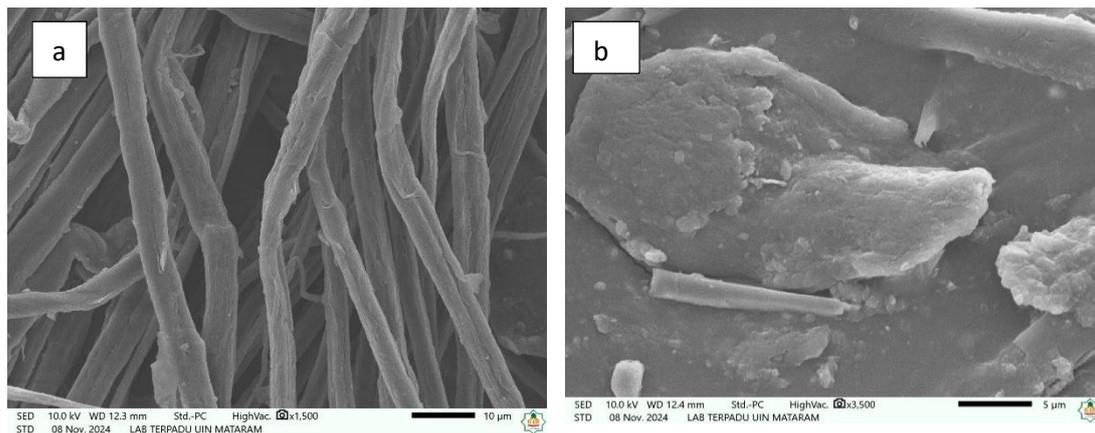


Figure 3. SEM Images of Treated PALF

While the treated PALF, undergoes disintegration, resulting in isolated microfibrils with a much finer structure compared to the long fibers. SEM images of microfibrils typically reveal some areas exhibit the accumulation of lignin, pectin, and other coating materials like hemicellulose and wax, forming a thin layer on the rough surface of PALF surface texture, which suggests that the treatment effectively removed much of the impurities and surface coatings. The reduced presence of lignin, waxes, and pectin makes the microfibrils more hydrophilic and increases their surface area, promoting better adhesion when integrated into composite matrices. The high surface area is crucial for reinforcing composite materials, as it facilitates stronger bonding with the polymer matrix (Gnanasekaran et al., 2021).

However, even after treatment, some microfibrils retain small amounts of residual impurities, which could influence the fiber-matrix interaction. Despite this, the SEM results indicate that these microfibrils possess desirable characteristics, such as increased surface area and reduced impurity content, making them suitable for use as reinforcing materials in composites. The presence of rough areas and thin layers of residual coatings in some regions also suggests that further refinement could enhance the overall performance of PALF in composites, particularly by improving its interfacial bonding with other materials (Gholampour & Ozbakkaloglu, 2020).

Tensile Strength Analysis

Tensile strength tests are crucial in assessing the mechanical properties of PALF, as they provide insight into its ability to reinforce composite materials. The tensile strength of PALF is influenced by several factors, including the extraction method, fiber diameter, moisture content, and surface treatment. Natural fibers like PALF exhibit significant variability in their mechanical properties due to these factors, and thus, the tensile strength may differ based on geographic location, processing methods, and environmental conditions (Karolina et al., 2020). In general, PALF has been found to exhibit a relatively high tensile strength compared to other natural fibers like jute and hemp. This study found that untreated PALF exhibited a maximum

stress of 4982 MPa, while treated PALF had a maximum stress of 428 MPa. Although the stress decreased, the value is still acceptable for use as a reinforcing material (Rizal & Hamdan, 2021). The treatment process likely caused the observed degradation, leading to a reduction in cellulose crystallinity (Amirulhakim et al., 2021). Surface treatment, such as alkali treatment (e.g., sodium hydroxide), is commonly applied to PALF to enhance its interfacial bonding with polymer matrices in composite materials. Alkali treatment helps remove impurities such as lignin and hemicellulose, improving the fiber's surface roughness and increasing its compatibility with the matrix, thereby improving the tensile strength of PALF-reinforced composites (Yudhanto et al., 2021)(Purkuncoro, 2017).

Table 2. Summary of Tensile Strength Test Data for Pineapple Leaf Fiber Samples

Pineapple Leaf Fiber Samples	Maximum Load (N)	Maximum Stress (MPa)	Maximum Elongation (mm)	Break Point Strain (%GL)
Untreated PALF	4.2612	4982.1	0.7795	3.0689
Treated PALF	14.703	428.29	0.9193	4.0118

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CONCLUSION

Based on the results of this research, it can be concluded that alkaline treatment with 5% NaOH and bleaching with NaClO effectively reduce the content of hemicellulose, lignin, pectin, and other impurities while increasing the cellulose content in the fiber. The tensile strength of untreated pineapple leaf fiber (PALF) is higher than that of treated PALF, as the extended drying process in treated fibers led to cellulose degradation. Additionally, the morphology of pineapple leaf fiber subjected to alkali treatment and bleaching revealed a significant reduction in amorphous structures such as hemicellulose, lignin, pectin, and other impurities, while retaining a substantial amount of cellulose content in the fiber.

RECOMMENDATION

Research related to the characterization of pineapple leaf fiber as a composite reinforcing material can include analysis methods using XRD to measure the crystallinity and scale up the material size into nano.

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