

Evaluation of Moisture Content, Chemical, and Functional Groups of *Paederia Foetida* Fibers: Effects of Time Soaking in Chemical Solutions

Nasmi Herlina Sari¹(⊠), Edi Syafri², Suteja¹, Muhammad Usman Jayadi¹, Emmy Dyah Sulistyowati¹, Paryanto dwi Setyawan¹, Sanjay Mavinkere Rangappa³, and Suchart Siengchin³

> ¹ University of Mataram, Mataram, West Nusa Tenggara, Indonesia n.herlinasari@unram.ac.id

² Department of Agricultural Technology, Politeknik Pertanian Negeri Payakumbuh, Kabupaten Lima Puluh Kota, Indonesia

³ Natural Composites Research Group Lab, Department of Materials and Production Engineering, The Sirindhorn International Thai-German Graduate School of Engineering (TGGS), King Mongkut's University of Technology North Bangkok (KMUTNB), Bangkok, Thailand

Abstract. Due to their abundance and environmental friendliness, *Paederia foetida* stem fiber (PFs) are an interesting material to develop as a substitute for glass fiber. The goal of this research is to provide a thorough understanding of the physical, chemical, and functional properties of PFs. The PFs were treated for 1 h, 2 h, and 4 h with 95% ethanol and 25% NaClO₂, respectively, before being rinsed and dried in a 105 °C oven. The density, moisture content, functional group (FTIR), and chemical composition of PFs were all tested to determine their properties. The results showed that PFs raw has moisture content and density of $12 \pm 0.78\%$ and 1.02 ± 0.012 g/cm³, respectively. After submerged in chemical solution for 4 h, the PFs had the lowest moisture content and density, but cellulose content to increased (67%), compare with other fibers; due to lignin and hemicellulose content reduced. The properties of PFs fiber suggest that it could be used instead of glass fiber.

Keywords: Paederia foetida L fibers (PFs) \cdot NaClO2 \cdot physical properties \cdot FTIR \cdot Chemical properties

1 Introduction

Researchers and industrialists are currently interested in the development of natural biopolymers from lignocellulosic materials due to their superior performance, sustainability, and abundant sources [1, 2]. Plants, particularly those in the fiber section [1], such as Sugar palm fiber [3], Acacia nilotica L., can be used to produce lignocellulose.

[4, 5], Phaseolus vulgaris [6, 7], Momordica Charantia [8, 9], Agave Gigantea [10]. This material has the potential to replace synthetic fibers, particularly as a composite filler [9, 10]. Unfortunately, the hydrophilic nature of this fiber makes it less desirable because it weakens the interface bond between the fiber and the matrix, affecting the mechanical properties of the resulting composite. Researchers have used chemical technology to reduce the moisture content of the fiber while increasing its cellulose content. Sari et al. [11] found that soaking the fiber in 1-8% NaOH for 2 h increased the cellulose content from 47.59%–62.87% while decreasing the hemicellulose and lignin content of 27.28 percent and 25.5% respectively. After the fiber was treated with NaOH, the functional groups of the fiber changed as well. Then, Sari et al. [12] reported that treating T. hibiscus fiber with NaOH and KOH reduces the water content, density, and chemical composition of the fiber. Soaking Agave Gigantia fibers in NaOH solution followed by soaking the fibers in 25% sodium chlorite (NaClO₂) has also been reported to reduce moisture properties, density, and crystallinity index whereas changing the functional groups of the fibers [8]. Furthermore, 4 mL of acetic acid and 8g NaClO₂ and mercerization 5 NaOH were found to be effective in removing 25% and 38% of the water content from raw sugar palm fibers, respectively [2]. Previous research has shown that chemical treatment is one of the simplest techniques for improving fiber properties and decreasing fiber hydrophilicity.

Paederia foetida (PF) is a creeping plant that grows rapidly and forms a canopy, making it a weed in agricultural areas. There is a very strong fiber in the stem of the PF plant that has the potential to be a source of cellulose with high economic value. To the best of our knowledge, no information exists about the properties of fiber from *Paederia foetida* stem.

Therefore, the purpose of this research is to provide a thorough understanding of the chemical properties, water content, density, and functional groups of the stem fiber of Paederia foetida (PFs). Chemical treatment of PFs with ethanol, followed by 25% sodium chlorite for 1 h, 2 h, and 4 h, respectively at room temperature (24 °C). The chemical composition, density, water content, and functional groups of the fiber were tested and compared to untreated fibers to characterize their chemical properties, density, water content, and functional groups.

2 Methods

2.1 Materials

Fresh PFs-cured plants were obtained from the Setanggor area of West Nusa Tenggara, Indonesia (Fig. 1a). The chemical solution used was ethanol (95%) and NaClO₂ (25%) supplied by (Sigma-Aldrich).

2.2 Extraction of PFs

Freshly cured PFs plants (Fig. 1a) were cleaned of leaves and cut 10 cm in length. The PF stems were cleaned with plain water, and the PF bundles were manually removed with a plastic comb. The fresh PFs fibers are then sun dried for three days at a relative humidity of 10 to 15%. The PFs were then cut into 10–15 mm lengths, ground with a grinding machine, and sieved through an 80 mesh sieve.

Samples	
A-PFs/1	Soaked in ethanol and NaClO ₂ for 1 h, respectively.
B-PFs/2	Soaked in ethanol and NaClO ₂ for 2 h, respectively.
C-PFs/4	Soaked in ethanol and NaClO ₂ for 4 h, respectively.

Table 1. Pronunciation



Fig. 1. (a) PF plant, (b) PF stem, (c). The PF fiber is 2 cm, (d). Cured fiber after grinding, (e-f). Immersion of PFs in ethanol and NaClO₂, (g). PFs treated.

2.3 PFs Treatment

1 L of ethanol solution was poured into a measuring cup containing 10 g of PFs and left for 1 h, 2 h, and 4 h, respectively. The fibers were rinsed with mineral water to remove any remaining ethanol solution from the PFs before being aerated. Furthermore, the PFs were immersed in a 25% NaClO₂ solution for 1 h, 2 h, and 4 h, respectively, then being rinsed with mineral water, aerated, and heated in a 105 °C oven for 60 min. Finally, as shown in Table 1, three different samples were ready for characterization.

2.4 Characterization

Density. An AccuPyc 1340 pycnometer was used to determine density via gas incursion under a flow of helium gas. In order to reduce the amount of moisture in the fibers, samples were dried in an oven at 105 °C for 24 h. Before being placed in the pycnometer, the sample was placed in a desiccator to eliminate any leftover water. At a temperature of 27 °C, 4 test samples from each individual treatment were used, and the average result

was obtained. Equation (1) is used to calculate the sample's density value [13].

$$\rho\left(\frac{g}{cm}^3\right) = \frac{Mass}{vol.} \tag{1}$$

Moisture Content. For the analysis of the water content of A-PFs/1, B-PFs/2, and C-PFs/4, five samples were prepared. All accordance with the requirement a 24-h heating process in an oven set to 105 C. To determine the sample's water content, its weight was measured before, *Wa*, and after, *Wb* heating [11, 14, 15]. Equation is used to determine the water content (1):

$$Moisture\ content(\%) = \frac{W_b - W_a}{W_b} \times 100\tag{2}$$

Fourier Transform Infrared (FTIR) Spectroscopy. To identify potential changes in functional groups present in samples after various treatments, FTIR is utilized. By using Perkin Elmer FTIR, spectra of PFs samples were collected at each treatment stage (Frontier Spectrum Instrument, USA). The range of 4000 to 500 cm-1 was used to gather the sample FT-IR spectra (10 10 3 mm). The sample containing the KBr matrix was pelletized before the spectra were obtained at a scan rate of 4 cm1 per minute and a wavelength range of 4000 to 500 cm1.

Chemical Composition. Due to changes in immersion duration, the standard TAPPI T203 om 93 method was used to determine the value of the cellulose, hemicellulose, and lignin content of micro PFs. Soxhlet extraction with acetone and alcohol for eight hours. Additionally, the lignin content was calculated using T222 om-88, and the hemicellulose content was calculated using Gatenholm and Tenkanen's technique (2003).

3 Results and Discussion

3.1 Density

As a result of the treatment, the densities of A-PFs/1, B-PFs/2, and C-PFs/4 decreased from 1.67 ± 0.021 g/cm³, 1.25 ± 0.015 g/cm³, and 1.02 ± 0.012 g/cm³, respectively. This downward tendency can be brought on by the fiber's loss of lignin and hemicellulose. When immersed in a chemical solution for a prolonged period of time, the concentration of lignin and hemicellulose gradually decreases.

The various fiber densities have dropped, as seen in Table 2. In ethanol and NaClO₂, the loss of amorphous non-cellulose molecules causes cavities to form in the fiber, which in turn causes the fiber to inflate and eventually separate into its constituent parts. According to Ilyas et al. [2], an increase in volume with a drop in weight may be the source of the decreased density value. Additionally, it was discovered that the density values of these three samples were lower than those of common man-made fibers such aramid (1.4 g/cm³), carbon (1.7 g/cm³), glass fiber (2.5 g/cm³), and sugar palm fiber (1.30 0.0023 g/cm³) [16].

3.2 Moisture Content

Table 2 displays the moisture content of samples A-PFs/1, B-PFs/2, and C-PFs/4. Table 1 shows that samples A-PFs/1, B-PFs/2, and C-PFs/4 had moisture contents of 12.4 ± 0.021 wt%, 10.12 ± 0.015 wt%, and 5.4 ± 0.012 wt%, respectively. This drop is assumed to be caused by a significant number of alkaline labile bonds between lignin, hemicellulose, and polysaccharides that break when the fiber is submerged in a chemical solution for 4 h, leaving only cellulose stable to dissolve in an acid solution [2].

3.3 FTIR Analysis

The FTIR spectra curves for A-PFs/1, B-PFs/2, and C-PFs/4 are shown in Fig. 2. The wavelengths for all samples, which are 3432.01 cm⁻¹, 2926.9 cm⁻¹, 2347.33 cm⁻¹, 1628.56 cm⁻¹, 1424.53 cm⁻¹, 1372.09 cm⁻¹, 1161.02 cm⁻¹, 1055.84 cm⁻¹, 897.49 cm⁻¹, and 665.37 cm⁻¹, respectively, have been clearly characterized from Fig. 2. The presence of the prominent lignocellulosic components, such as cellulose,

Table 2. Moisture Content and Density of Different PFs Samples.

Samples codes	Moisture Content (%)	Density (g/cm ³)
A-PFs/1	12.5 ± 0.78	1.67 ± 0.021
B-PFs/2	10.12 ± 1.02	1.25 ± 0.015
C-PFs/4	5.4 ± 0.89	1.02 ± 0.012



Fig. 2. FTIR of A-PFs/1, B-PFs/2, and C-PFs/4 samples

Samples			Assignments
A-PFs/1	B-PFs/2	C-PFs/4	
Wave number (cm ⁻¹)		-1)	
3432.01	3434.91	3436.95	O-H stretching vibrations of α -cellulose and hydrogen bond of the hydroxyl groups/ Cellulose I β
2926,9	-	-	Alkyl C-H stretching/ Cellulose and hemicellulose components / C-H stretching vibration in cellulose and Hemicellulose
2347.33	2090.84	2347.7	C = C alkynes group
1628.56	1638.87	1638.72	Water/adsorbed water
1424.53	1424.77	1424.25	Cellulose/CH ₂ bending
1372.09			Cellulose/C-H bending
1161.02	1162.35	1161.35	Cellulose, hemicellulose, lignin/C-O stretching vibration in cellulose
1055.84	1057.26	1056.05	C-O stretching modes of the hydroxyl and ether groups in the cellulose
897.49	-	-	Cellulose/β-Glucosidic linkage in cellulose
665.37	663.99	665.64	C-OH out of plane bending

Table 3. Peak Positions And Assignments Of Chemical Groups Of A-PFs/1, B-PFs/2, Dan C-PFs/4 Samples.

hemicellulose, lignin, and other impurities, is indicated by the intense peaks (see Table 3).

3.4 Chemical Composition

Table 3 lists the chemical compositions of samples A-PFs/1, B-PFs/2, and C-PFs/4. It was discovered that the cellulose content of B-PFs/2 rose from the preceding 51.27% (A-PFs/1) by 24.93%. After being immersed in a chemical solution for 4 h, the fiber breaks the hydrogen bonds, causing the hemicellulose and lignin molecules to dissolve [8].

The cellulose content of the C-PFs/4 sample was found to be higher than that of the other samples studied and higher than that of Sugar Palm (56.67%), but still lower than that of Agave Gigantea fiber (89.39%). The chemical composition of the developed fibers is shown in the Table 4.

Samples	Chemical con		Reff.	
	Lignin	Hemicellulose	Cellulose	-
A-PFs/1	9.78	29.59	51.26	This Study
B-PFs/2	10.91	22.38	64.04	This Study
C-PFs/4	18.82	9.81	67.43	This Study
Sugar Palm	0.27	19.8	56.67	[19]
Agave Gigantia	0.53	3.73	89.39	[8]

 Table 4. Chemical Composition of A-PFs/1, B-PFs/2, and C-PFs/4 Samples.

4 Conclusion

Experiments were used in this study. Paederia foetida L's density, moisture content, functional groups, and chemical content have been investigated. The findings indicated that while the moisture percentage of the fiber reduced after it was immersed in the ethanol/NaClO₂ solution for a longer period of time (4 h), the fiber's density and cellulose content increased as a result of reduction of lignin and hemicellulose. Furthermore, the wavelength absorption band of the fiber functional groups changed, indicating that lignin and hemicellulose were removed from the fiber after being soaked in chemical solutions.

References

- Manimaran, P., Senthamaraikannan, P., Sanjay, M. R., Marichelvam, M. K., Jawaid, M.: Fiber characterization of furcraea foetida natural fiber as potential reinforcement of biocomposite. Carbohydrate Polymers 181, 650–58 (2018). doi: https://doi.org/10.1016/j.car bpol.2017.11.099.
- Ilyas, R. A., Sapuan, S. M., Sanyang, M. L., Ishak, M. R., Zainudin, E. S.: Nanocrystalline cellulose as reinforcement for polymeric matrix nanocomposites and its potential applications: a review. Curr Anal Chem 14, 203–25 (2018). https://doi.org/10.2174/157341101366617100 3155624.
- Ilyas, R. A., Sapuan, S. M., Atiqah, A., Ibrahim, R., Abral, H., Ishak, M. R., Zainudin, E. S., Nurazzi, N. M., Atikah, M. S. N., Ansari, M. N. M., Asyraf, M. R. M., Supian, A. B. M., Ya, H.: Sugar palm (Arenga pinnata [Wurmb.] Merr) starch films containing sugar palm nanofibrillated cellulose as reinforcement: Water barrier properties. Polymer Composites 1–9 (2019). DOI: https://doi.org/10.1002/pc.25379.
- Kumar, R., Sivaganesan, S., Senthamaraikannan, P., Saravanakumar, S. S., Anish Khan, S. A. A. D., Loganathan, L.: Characterization of new cellulosic fiber from the bark of acacia nilotica l. plant. Journal of Natural Fibers 19, 199–208 (2020).
- Babu, B. G., Princewinston, D., Saravanakumar, S. S., Anish Khan, P. V. A. B., Indran, S., Divya, D.: Investigation on the physicochemical and mechanical properties of novel alkalitreated phaseolus vulgaris fibers. Journal of Natural Fibers 1–12 (2020).
- Vijay, R., Jafrey Daniel James Dhilip, S. G., Harikrishnan, S., Chandru, B., Amarnath, M., Khan, A.: Characterization of natural cellulose fiber from the barks of vachellia farnesiana. Journal of Natural Fibers 19, 1343–1352 (2020).

- Anish, K., Vijay Raghunathan, D. L. S., Sanjay, M. R., Siengchin, S., Jawaid, M., Alamry, K. A., Asiri, A. M.: Extraction and characterization of cellulose fibers from the stem of momordica charantia. Journal of Natural Fibers 19, 2232–2242 (2020).
- Syafri, E., Jamaluddin, J., Sari, N. H., Mahardika, M., Amanda, P., Ilyas, R. A.: Isolation and characterization of cellulose nanofibers from Agave gigantea by chemical-mechanical treatment. International Journal of Biological Macromolecules 200, 25–33 (2022). https:// doi.org/10.1016/j.ijbiomac.2021.12.111.
- Mahardika, M., Abral, H., Kasim, A., Arief, S., Hafizulhaq, F., Asrofi, M.: Properties of cellulose nanofiber/bengkoang starch bionanocomposites: Effect of fiber loading. Lwt 108554 (2019).
- Mochamad, A., Abral, H., Yogi Kurnia, S. M. S., Kim, H.-J.: Effect of duration of sonication during gelatinization on properties of tapioca starch water hyacinth fiber biocomposite. International Journal of Biological Macromolecules 108, 167–76 (2018). doi: https://doi.org/ 10.1016/j.ijbiomac.2017.11.165.
- Sari, N. H., Wardana, I. N. G., Irawan, Y. S., Siswanto, E.: Characterization of the Chemical, Physical, and Mechanical Properties of NaOH-treated Natural Cellulosic Fibers from Corn Husks. Journal of Natural Fibers (2017). https://doi.org/10.1080/15440478.2017.1349707.
- Sari, N. H., Rahman, A., Syafri, E.: Characterization of Musaceae and Saccharum Officinarum Cellulose Fibers for Composite Application. International Journal of Nanoelectronics and Materials Volume 12 (2), 193–204 (2019).
- Sari, N. H., Suteja, S., Ilyas, R. A., Syafri, E., Indran, S.: Characterization of the density and mechanical properties of corn husk fiber reinforced polyester composites after exposure to ultraviolet light. Functional Composites Structure 3 034001 (2021). https://doi.org/10.1088/ 2631-6331/ac0ed3.
- Ilyas, R. A., Sapuan, S. M., Ishak, M. R., Zainudin, E. S.: Effect of Delignification on the Physical, Thermal, Chemical, and Structural Properties of Sugar Palm Fibre. BioResources, 12 (4), 8734–8754 (2017).
- Jumaidin, R., Sapuan, S. M., Jawaid, M., Ishak, M. R., Sahari, J.: Characteristics of Eucheuma cottonii waste from East Malaysia: Physical, Thermal, and Chemical composition. European Journal of Phycology 0 (0), 1–8 (2016). https://doi.org/10.1080/09670262.2016.1248498.
- Mohanty, A. K., Misra, M., Hinrichsen, G.: Biofibres, biodegradable polymers and biocomposites: An overview. Macromolecular Materials and Engineering 276–277, 1–24 (2000). https://doi.org/10.1002/(SICI)1439-2054(20000301)276:13.0.CO;2-W
- Razali, N., Salit, M. S., Jawaid, M., Ishak, M., Lazim, Y.: A study on chemical composition, physical, tensile, morphological, and thermal properties of roselle fibre: Effect of fibre maturity. BioResources 10 (1), 1803–1823 (2015).
- Akil, H. M., Omar, M. F., Mazuki, A. A. M., Safiee, S. S., Ishak, Z. A. M., Bakar, A.: Kenaf fiber reinforced composites: A review. Materials and Design 32 (8–9), 4107–4121 (2011). DOI: https://doi.org/10.1016/j.matdes.2011.04.008.
- Ilyas, R. A., Sapuan, S. M., Ishak, M. R.: Isolation and characterization of nanocrystalline cellulose from sugar palm fibres (Arenga Pinnata). Carbohydrate Polymers (2017). https:// doi.org/10.1016/j.carbpol.2017.11.045.

Open Access This chapter is licensed under the terms of the Creative Commons Attribution-NonCommercial 4.0 International License (http://creativecommons.org/licenses/by-nc/4.0/), which permits any noncommercial use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons license and indicate if changes were made.

The images or other third party material in this chapter are included in the chapter's Creative Commons license, unless indicated otherwise in a credit line to the material. If material is not included in the chapter's Creative Commons license and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder.

