Bukti Korespondensi

Paper18

Comprehensive Characterization Of Novel Cellulose Fiber From Paederia Foetida and Its Modification For Sustainable Composites Application

Pengusul
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Comprehensive Characterization of Novel Cellulose Fiber from Paederia Foetida and its Modification for Sustainable Composites Application

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From:

Prof. Nasmi Herlina Sari,
Professor, Mechanical Engineering Department, Faculty of Engineering, University of Mataram, Mataram, West Nusa Tenggara 83125, Indonesia

To:
Prof. Chi-Wang Li
The Editor-in-Chief,
Journal of Applied Science and Engineering

Dear Sir,

Sub: Submission of manuscript to Journal of Applied Science and Engineering

Warm Greetings!

I am submitting a manuscript entitled **"Comprehensive Characterization of Novel Cellulose Fiber from *Paederia foetida* and its Modification for Sustainable Composites Application "** for publication in your esteemed journal. This work has not been published elsewhere and also it has not been simultaneously submitted for publication elsewhere.

Highlights of this Investigation:

- NaOH treatment was found to be more effective in removing lignin and hemicellulose components, as well as increasing cellulose compounds in the fiber.
- The tensile strength increased significantly by 43.27% and 22.37%, respectively, as well as the thermal resistance
- The highest crystallinity index was obtained from KOH-treated PFs of 79.685%, while the highest tensile strength was from NaOH-treated of 2450.9 MPa.

Here are some reviewers that we recommend to review the manuscript above:

1. Prof Mounir El Achaby (Mounir.ELACHABY@um6p.ma)
2. Prof S.M. Sapuan (sapuan@upm.edu.my)
3. Prof Hairul Abral (abral@ft.unand.ac.id)
4. Dr S. Indran (indransdesign@gmail.com)

We look forward to hearing a positive response from you.

Thank you,

Date: 20.09.2022

Place: Mataram, Indonesia

Best regards,

Prof . Nasmi Herlina Sari

Respected Editors & Reviewers

We are thankful for the valuable comments provided from your side. These comments were very helpful in enhancing the quality of the manuscript. We have given a point by point reply for the comments given by the reviewers in the following sections. The comments are considered with prime importance and manuscript is revised accordingly. So kindly consider the manuscript for further processing.

Reviewer: 1

In general, the paper is well written and it is interesting but some relevant information should be revised before considering it for publication. I recommend this manuscript to minor revision.

1. Abstract need to be modified by adding few information on the expected application of this current work. Author is also advised to add few more key results obtained from this work.

Thank you for your valuable comments. We have incorporated in the revised manuscript.

2. Most of the references cited in this manuscript are quite back dated. I am strongly suggesting the author to add more recent references dated between 2017 to 2022.

Thank you for your valuable comments. We have incorporated in the revised manuscript.

1. Ismail, S.O., Akpan, E., Dhakal, H.N. 2022. Review on natural plant fibres and their hybrid composites for structural applications: Recent trends and future perspectives, Composites Part C, 9, 100322, https://doi.org/10.1016/j.jcomc.2022.100322.
2. Zhang, Q., Ma, Y., Qi, Z., Jia, C., Yao, Y., Zhang, D. 2022. Optimisation on uniformity and compressibility of rapeseed straw cellulose fiber mixtures for straw/mineral hybrid natural fiber composite, Industrial Crops and Products, 189, 115852, https://doi.org/10.1016/j.indcrop.2022.115852.

Reviewer: 2

Comments to the Author

1. Part of the findings are important as Paederia Foetida is one of the important sources of lignocellulose component and has never been extracted for its fibre. However, it is not sufficient and scientifically-sound if simply say that no study has been done. Instead, the authors should provide technical reasons or research gap that can justify the selection of this plant. Perhaps authors can refer this paper on how new resources of biomass being discussed as stated by Mat Nasir et al on The Effect of Alkaline Treatment onto Physical, Thermal, Mechanical and Chemical Properties of Lemba Leaves Fibres as New Resources of Biomass, Pertanika Journal of Science & Technology, 2020, 28, 1531. Authors should highlight the technical importance of PF fibre in the abstract as well as in the introduction part so that it can strengthen the novelty of this paper. Especially, the use of alkaline treatment (NaOH and KOH) is not new. It would be good if the authors can state the reason, why still focus on this traditional method.

Thank you for your valuable comments. We have incorporated in the revised manuscript. We have added the journal in this manuscript revision as suggested by the reviewers, namely: "Nur Aina Farhana Mat Nasir, et al. 2022. The Effect of Alkaline Treatment onto Physical, Thermal, Mechanical and Chemical Properties of Lemba Leaves Fibres as New Resources of Biomass. "

2. Based on abstract and introduction part, the author mentioned on the fibre treatment using NaOH and KOH. However, from method part, I come to know that authors also left fibre to be rotten in water. Since this step was carried out for 10 days, it looks to me that this step is significant, as compared to 2 hr alkaline treatment. But you did not mention it at all in abstract or introduction part. By any chance, the results that labelled as raw fibre, are you referring those results to the sample after water immersion? If so, please correct it. If not, you need to indicate the results for rotten fibre and discuss it also. This is especially because if you perform alkaline treatment at a low concentration of NaOH, low temperature and for a short period of time, it is not sufficient to obtain cellulose with higher purity. I have seen in several papers in order to purify cellulose, other authors studied at higher alkaline concentration, temperature and time. (Bazargan et al. https://doi.org/10.1016/j.biombioe.2014.08.018, Lai et al,

https://doi.org/10.1002/tqem.21825). Need to compare the results obtain with fibre of similar range.

Thank you for your valuable comments. We have incorporated in the revised manuscript and We have added the journals suggested by the reviewer in this revised manuscript as suggested by the reviewer, namely: Alireza Bazargan, Tesfalet Gebreegziabher, Chi-Wai Hui, Gordon McKay, The effect of alkali treatment on rice husk moisture content and drying kinetics, Biomass and Bioenergy, Volume 70, 2014, Pages 468-475, ISSN 0961-9534, https://doi.org/10.1016/j.biombioe.2014.08.018.

Reviewer: 3

There is no 'response to reviewer' from the authors, but some parts of the current version have been revised. The abstract should be revised; some sentences are redundant.

Thank you for your valuable comments. We have incorporated in the revised manuscript

Comment: Eq. (7): please give details/reference of using ideal gas law to calculate volume of gas, is it acceptable/reasonable? This is an important issue because the authors use the values to determine process efficiency.

- The details or reference of using ideal gas law are not presented.

Thank you for your valuable comments. In the manuscript, we did not calculate the gas volume of the fiber sample, so we did not discuss the efficiency of the process.

Comment: How to obtain DTG curve of Fig 2 (b), what is the equation to calculate dTG/dt? If it is the gas temperature dynamics, why dTG/dt are negative values when the temperature increase?

#Figure 2b shows the alkaline chemical process of fiber samples starting from photos of raw fiber, fiber in alkaline solution and fiber after being soaked in chemical solution.

TG analysis was utilized to study the thermal durability of the PFs by monitoring weight change over time at a steady temperature range of 25°C to 600°C using a thermogravimetric analyzer (NETZSCH STA 2500).

Comment: Fig. 1 should be improved to clearly illustrate the process diagram. The authors may consider showing some pictures of the real process.

- A picture was added, but the components of the process still are not clear.

Thank you for your valuable comments. We have corrected Figure 1 in accordance with the reviewer's suggestions and We have incorporated it in the revised manuscript.

Comment: - Fig. 4 (c) shows that the gas product from MOP 600 W of 500 ºC is less than that of 450 ºC but their total energy data from Table 1 are 141.59 and 130.45 kJ, please add more explanation for this issue?

Thank you for your insightful comments. However, as the reviewer suggested, there is no 4c image in this manuscript. We do not calculate the total energy of the sample in the manuscript. The density, moisture content, and diameter data for raw and alkali-treated PFs are shown in table 1.

Reviewer: 4

The manuscript explains about the characteristics of NaOH and KOH pretreated PF and its comparison with the untreated PF. However, the paper requires major revision before acceptance.

1) In INTRODUCTION, Line no: 61- what are urinary bullets and stomatic?

Thank you for your valuable comments. We have deleted “urinary bullets and stomatic in the manuscripts.

2) FIGURE 1: Improve the quality of the figure.

Thank you for your valuable comments. We have repaired Fig 1.

3) In RESULTS AND DISCUSSION, Line 216-217, please provide reference.

Thank you for your valuable comments. We have incorporated in the revised manuscript.

4) In FIGURE 4: What is %T? Please mention it

\# Thank you for your valuable comments. We have repaired Fig 4 and we have incorporated in the revised manuscript.

5) In FIGURE 4, it would be more better, if you could mark the peaks with wave numbers.

\# Thank you for your valuable comments. We have described each fibers' peak location and chemical assignment group and we have incorporated in the revised manuscript.

6) In TABLE 3 what is silikate? Did you mean silicate?

\# Thank you for your valuable comments. We have replaced silicate with "silica" and we have incorporated in the revised manuscript.

7) FIGURE 5, in X- axis, please change deg as degree Celsius or its symbol.

\# # Thank you for your valuable comments. and we have incorporated in the revised manuscript (Please seen in Fig 5)

8) Please check the legends in FIGURE 5. In FIGURE 5, peak is high for Raw and KOH pretreated PF, but in line 262-263, CrI is less for Raw PF and more for NaOH pretreated PF.

\# Thank you for your valuable comments. and we have incorporated in the revised manuscript (Please seen in in line 262-263)

9) In SECTION 3.7, the authors have explained the changes happening in each temperature range. Please connect it with your result or graph.
Explain why weight loss is more in raw PF.

\# Thank you for your valuable comments. Figure 5 also shows that the weight loss of raw fiber is more than that of fiber after NaOH and KOH treatment due to the high water and hemicellulose content of the raw fiber (see table 1). (Please see in line 328-331).

10) In CONCLUSION, Crystallinity index of KOH is mentioned as 79.685%, but in LINE 263, it is 50.93%, please correct it.

Thank you for your valuable comments. and we have incorporated in the revised manuscript (Please seen in conclusion (line 338)

11) Add about the future perspective or application of this study in conclusion.

Thank you for your valuable comments. and we have incorporated in the revised manuscript. The mechanical properties of these PFs have the potential to replace glass fibers as composite raw materials, especially for construction applications. (Please seen in conclusion (line 338),

Reviewer: 5

The manuscript titled "Comprehensive Characterization of Novel Cellulose Fiber from Paederia Foetida and its Modification for Sustainable Composites Application" can be accepted for publication after all the below mentioned comments are addressed.

- Rewrite the species name genus name in Italics format in title and throughout the manuscript (titile, line 51, line 112)

Thank you for your valuable comments. and we have incorporated in the revised manuscript

- Same abbreviations are given for Paederia Foetida stem (PFs) and Paederia Foetida fiber. make it separate abbreviation throughout the manuscript.

Thank you for your valuable comments. and we have incorporated in the revised manuscript

- Line 28: instead of chemical, mechanical and physical properties keywords add glass fiber and coconut fibers. Also make it alphabetical order

Thank you for your valuable comments. and we have incorporated in the revised manuscript

- Check the grammar and spelling mistakes throughout the manuscript

Thank you for your valuable comments. and we have incorporated in the revised manuscript

- Line 121 & 122: In equations add superscript and subscript appropriately.
Thank you for your valuable comments. and we have incorporated in the revised manuscript

-Line 128: spelling mistake "slices"
Thank you for your valuable comments. and we have incorporated in the revised manuscript

- Line 193: Mention the full form of TGA first then followed by abbreviation
Thank you for your valuable comments. and we have incorporated in the revised manuscript

Abstract

Fiber derived from the plant *Paederia Foetida* stems (PF) is a novel natural fiber with the capacity to replace glass fibers in composite reinforcement. The technique of modifying the fiber surface with NaOH and KOH is simple and straightforward, generating properties that differ from the raw fiber. Therefore, this study aims to investigate the effects of NaOH and KOH treatments on the surface of *Paederia foetida* fiber (PFs) in relation to the physical, crystal structure, functional groups, tensile strength, thermal and morphological properties compared to raw PFs. The raw fiber was obtained by soaking the PFs rods in water for 10 days, followed by surface treatment with a solution of NaOH and KOH. The results showed that compared to raw fiber, after NaOH dan KOH treated PFs, the diameter and moisture content of the PFs decreased, while the tensile strength increased significantly by 43.27%, and good the thermal stability. The highest crystallinity index was obtained from KOH-treated PFs of 79.685%. According to the functional groups in the FTIR observations, NaOH treatment was found to be more effective in removing lignin and hemicellulose components, as well as increasing cellulose compounds in the fiber. The surface morphology was found to be rougher with the loss of impurities after NaOH treatment. Based on the results, raw, NaOH and KOH-treated PFs have the potential to be used as reinforcement for lightweight composite and alternative materials to replace glass fiber and coconut fiber in construction applications.

Keywords: chemical, KOH, mechanical properties, NaOH, *Paederia foetida* fiber (PFs), physical properties.

1. Introduction

Nature has provided various renewable and environmentally friendly resources that have many potentials uses for textile fibers and composite materials. Natural fibers can be produced from any part of the plant and used in a wide range of applications, including fibers and textiles for composite applications [1,2,3,4,5]. In recent decades, new natural fibers with their best properties have been investigated and characterized such as corn husk fiber [1], *Juncus* plant [2], *Hibiscus tiliceaus* fiber [6], Sterculia *foetida* fruit shell fiber [5,7], *Musaceae* and *Saccharum officinarum* Cellulose Fibers [8], etc. Furthermore, the lignocellulosic natural fiber is known to have strength and stiffness properties comparable to glass fiber, as well as low density, and non-abrasive [9,1,10]. These unique traits and properties have created opportunities for the investigation and development of various natural fibers.

One source of natural fiber that has not been utilized optimally is *Paederia foetida* (PF) which grows convoluted vines, as well as forms shrubs and annuals. Furthermore, PF leaves belong to the *Rubiceae tribe*, have soft stems, and are used for climbing tree, they are only half a centimeter in diameter, but can reach 10 m in length. Several investigations have been conducted on the PF plant in relation to the use of drugs in the health sector, such as anti-rheumatic, analgesics, mucolytics, appetite enhancers, anti-biotics, anti-inflammatory, cough medicines or anti-tussive, and diarrhea medication [11]. However, there is no information related to the use of fiber from the PF plant as a filler for composite materials. The abundant and eco-friendly fiber sources from this plant need to be developed to achieve the excellent mechanical, physical, and thermal durability, as well as for wider applications.

Several investigations on the reinforcing of natural fibers in polymer composite materials to obtain desired qualities have been conducted. However, the developed composites often experience mechanical failures such as fiber pullout and debonding with the matrix. This failure is presumably due to the poor adhesion between hydrophobic (polymer) and hydrophilic (fibers), culminating in poor mechanical properties [12,13]. Fortunately, Natural fibers' hydrophilic qualities can be lowered chemically by treating them with NaOH, silane, KOH, and peroxide [14, 6, 8]. Sari et al. [1] reported that the tensile strength of a single cornhusk fiber was enhanced to 368.25 MPa after being modified with NaOH from 0.5 to 8% for 2 hours. Furthermore, Shanmugasundaram & Ramkumar [15] stated that the cellulose content of betel leaf fiber of 57.49% can be increased to 68.54% after treatment with 5% NaOH. Sari and Padang, [6] also reported that the tensile strength of *T. hibiscus* fiber after treatment with 8% KOH reached 5144.9 MPa, while the surface of the fiber became cleaner, rougher, and fibrillar. Khan et al. [16] mentioned that banana fiber improved the mechanical properties after the treatment with NaOH solution. It was also discovered that the epoxy composite with banana fiber reinforcement treated with 4.5% NaOH had the greatest improvement in both compressive and tensile strength, with 24.2% and 34% increases, respectively. The optimal epoxy composite strength was obtained at 25.4 MPa or increased by 38% after banana fiber was treated with 6.5% NaOH. Mat Nasir et al [17] also reported that after being treated with 6-10 wt% sodium hydroxide (NaOH) solution for 24 hours, Lemba Leaves Fibers (LeLeF) have fiber diameters in the range of 191.37 μm - 36.81 μm, and tensile properties in the range of 14.45 - 511.10 MPa, where the resulting properties are better than raw fiber. The mechanical properties and thermal stability of the fiber after NaOH treatment are superior to raw catfish fiber. Maity et al. [18] investigated the characteristics and uses of jute and nonwoven-based composites. Meanwhile, Liu et al. [19] gathered *Sterculia* and *Foetida* plant stalk debris and slices it long and short, with an average length of around 38 mm and 16 mm, to make environmentally sustainable light translucent concrete. Furthermore, alkaline treatment has been shown to increase the moisture retention of biomass while decreasing the activation energy of rice husks. While it is known that sample pre-treatment increases the drying rate and effective diffusivity of rice husks [20]. This previous study shows that the properties of new natural fibers need to be investigated and developed. The unique properties of different, abundant, and environmentally friendly natural fibers need to be considered to encounter composite reinforcing materials. The modification of natural fibers also needs to be investigated to ensure the best properties which can provide added value to natural fibers.

Therefore, the purpose of this study was to provide detailed information on the extraction and evaluation of new natural fibers from the fiber stems of the plant *Paederia foetida* (PFs). The characteristics of the fiber were investigated through changes in morphology, tensile strength, thermal resistance, crystallinity index, and functional groups. The properties of the raw materials used will be compared to those of the fiber post-treatment with NaOH and KOH.

2. Experimental program

2.1 Materials

Paederia foetida plant in **Fig. 1a** was collected from Batukliang, Lombok, Indonesia, the stems were separated from the leaves manually, then sliced to lengths of 150 mm (**Fig. 1b**). Furthermore, a solution of 5% sodium hydroxide (5% NaOH) and 5% potassium hydroxide (5% KOH) was used as alkali treatment. It was obtained from the Biochemistry Laboratory, Faculty of Food Technology and Agricultural Products, University of Mataram.

2.2. Extraction of *Paederia foetida* fiber

The PF were soaked for 10 days in fresh water (**Fig. 1c**) to allow them to rot, then, the fibers from the rods were removed using a plastic comb to maintain a uniform diameter, subsequently, they were air-dried and prepared to be tested (Fig. 1d).

Fig. 1. *Paederia foetida*, PF, (a) plant, (b) selection of PF stems, (c) Soaking PF in water, (d) Extracting fiber from PF stems.

2.3 Alkali treatment of PFs

The PFs were immersed in 5% NaOH and 5% KOH alkaline solutions for 2 h at room temperature of 28 ^{o}C. Next, they were washed with distilled water four times to remove the remaining alkaline solution, followed by drying in the sun. The below is the reaction scheme [1,8].

Fiber – OH + NaOH → Fiber – O⁻ Na+ + H_2O (1)

Fiber – OH + KOH → Fiber – O⁻ K+ + H_2O

(2)

Following that, the PFs were placed in a dry plastic container with a humidity of 20%; **Fig. 2** depicts the alkaline treatment method for PFs. Raw fibers as well as those treated with NaOH and KOH are shown in **Fig. 3.**

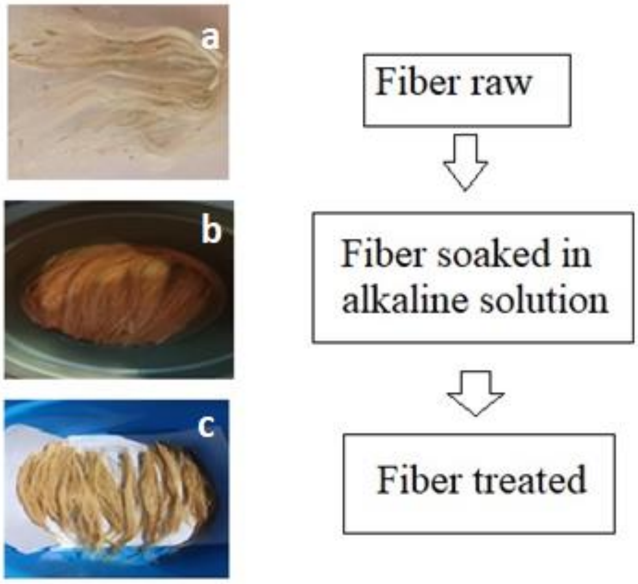


Fig. 2. Alkaline treatment process of PFs (a) fiber raw, (b) alkalization, and (c) dry PFs.

2.4 Characterization

2.4.1 Fiber density and diameter

A single PF sample was cut into small splices and placed on a microscope slide, then the raw and alkali-treated fiber diameters were measured using optical microscopy. A randomly selected 10 samples were measured at five locations along the length, and the average diameter value was reported.

Fig. 3. PFs (a) raw, (b) KOH treated dan (c) NaOH treated.

Furthermore, the buoyancy force was calculated by weighing PFs first in air and then in distilled water. The weighing was done using an analytical balance with a resolution of 0.001 g and was adapted for suspension weighing using a stainless-steel wire with a diameter of approximately 0.4 mm. By dividing the liquid density by the buoyancy force, sample volume was calculated. The density f was calculated by dividing the sample weight in air by the volume of the sample [1, 14].

$$\rho_f = \frac{(m_3 - m_1)}{(m_3 - m_1) - (m_4 - m_2)} \rho_w \quad (3)$$

where ρ_f is the fiber density (g/cm^3), and m_1 and m_2 are the weights of suspension wire in air and liquid, respectively. m_3 and m_4 represent the weight of the wire plus the sample weight in air and liquid (g), respectively. ρ_w = 0.998 g/cm^3 (standard density of distilled water). All densities were measured using a Metller Toledo measurement kit at 23±0.2 ^{o}C [14].

2.4.2 Moisture content

Water absorption has an impact on the physical properties of composites as well as the PFs-matrix interface [21]. The lowered hydrophilic hydroxyl groups increased the moisture absorption characteristics of the fiber. The moisture content of PFs was evaluated by cutting them into 5-10 mm lengths and drying them in an oven at 103^{o}C for 4 hours before placing them in a desiccator for 24 hours. The calculation of moisture contents is analogous, normal weight loss methods were used. Weighed PFs were dried in an oven at 105 °C for 4 hours, then chilled in a desiccator for 10-15 minutes and left until the weight of fibers reached constant. The moisture content in PFs was determined by using formula 2 [2,6].

$$\%Mc = \frac{W_{fb} - W_{fa}}{W_{fb}} \ x \ 100 \tag{4}$$

where Mc is the moisture content, W_{fb} (g) is the weight before putting in oven, and W_{fa} is the weight after putting in oven.

2.4.3 FTIR

To detect the distinctive functional groups, a Perkin Elmer Spectrum FTIR spectrometer (model Frontier) was employed by delivering infrared light via the sample fibers and co-adding 32 scans at 8 cm^{-1} resolutions within the 4000-450 cm^{-1} range with a resolution of 4 cm^{-1} [14]. Following that, the FTIR spectra of raw and processed PFs were studied.

2.4.4 Chemical Content.

The chemical composition has an effect on fiber characteristics. TAPPI Standard T 264 technique [14] was used to assess the raw as well as NaOH, and KOH treated fibers.

2.4.5 XRD

The crystallization of PFS fibers was examined using XRD analysis before and after alkali treatment, while the PHILIPS X-ray diffractometer was used to evaluate the powdered cured fiber (model PW3050). With a target monochromatic radiation of Cu K, the diffractometer was set to run at 30 mA current, 40 kV voltage, and 30 mA current. Equation 5 was used to calculate the cured PFs's crystallinity index [3].

$$CI = \frac{\mathrm{H}_{(0\,0\,2)} - \mathrm{H}_{(1\,0\,1)}}{\mathrm{H}_{(1\,0\,1)}} \tag{5}$$

where *CI*: is the crystallinity index of the PFs, $H_{(0\ 0\ 2)}$ denotes the maximum height of the crystalline fraction at 2θ (22.07° – 22.31°), and $H_{(1\ 0\ 1)}$ is the smaller peak of the amorphous fraction at 2θ = 18.23°.

2.4.6 Single PFs Tensile Test

The tensile strength of PFs raw, NaOH treated, and KOH treated PFs was tested using a universal testing machine (UTM) brand INSTRON 1390. The test was carried out in line with ASTM D3379-7. To determine the accuracy of the PFs test results, at room temperature, ten samples of each raw and alkali-treated fiber were tested at a loading rate of 2.5 mm/min. The average tensile strength of a single fiber is computed by using Equation 6 [9].

$$\sigma_f = \frac{4F}{\pi d^2} \quad (6)$$

where σ_f denotes tensile strength, *F* denotes the maximum tensile force required to break the PFs, and *d* denotes average diameter.

2.4.7 SEM morphology

The surface morphology or bonding of shattered materials used in tensile tests was characterized using SEM with a JEOL Model JSM - 840A. To investigate the bonding and interior structural changes, as well as to improve sample conductivity, the surfaces were coated with a thin layer of gold sputtering using a JEOL sputter ion coater, while the morphology was investigated using SEM at 10 kV.

2.4.8 Thermogravimetric analysis (TGA)

TG analysis was utilized to study the thermal durability of the PFs by monitoring weight change over time at a steady temperature range of 25^{o}C to 600^{o}C using a thermogravimetric analyzer (NETZSCH STA 2500).

3. Result discussions

3.1 Fiber density and diameter analysis

Table 1 shows that the diameter was reduced by 25% and 18.27%, respectively after the treatment with 5% NaOH or 5% KOH compared to raw PFs. This is due to impurities being removed from PFs surfaces. Raw PFs were observed to have a lower density than treated fibers. The elimination of lower density sections such as lignin and hemicellulose caused the densities to rise. A higher density suggests that the fibers contain more cellulose. These PFs have a higher density than 8% treated corn husk fiber (0.61 g/cm^3) [3] but lower than glass (2.54 g/cm^3) [22].

Table 1. Raw and alkali-treated PFs' density, moisture content, and diameter.

Sample	Density (g/cm^3)	Moisture content (%)	Diameter (mm)
raw	0.9 ± 0.071	7.18 ± 0.34	0.104 ± 0.03
Treated (5% NaOH)	1.16 ± 0.082	6.87 ± 0.27	0.085 ± 0.021
Treated (5% KOH)	1.09 ± 0.09	6.46 ± 0.38	0.078 ± 0.012

3.2. Moisture content analysis

The NaOH and KOH treatments reduced the moisture content of the PFs, as demonstrated in **Table 1**. The moisture absorption of raw PFs namely 7.18 % is higher than that of NaOH and KOH treated fibers of 6.87 % and 6.46 %, respectively. The structure's hydrogen bond is broken down by alkali treatment, which improves matrix interlocking by reducing the hydrophilic hydroxyl groups in the fiber [7,27].

3.3 FTIR analysis

The chemical treatment was utilized to clear the PFs surface of hemicellulose, lignin, and other impurities, whilst FTIR was used to analyze the effects of alkali treatment on fiber chemical processes. **Fig. 4** depicts the complete infrared pattern of PFs samples. A spectrum recorded between 4000 cm^{-1} to 400 cm^{-1} corresponds to hydrogen bonding alcohols or OH molecules extending from cellulose, hemicellulose, and lignin. The C–H bond, which is common in alkane groups, was found at the broadband of 3400–2850 cm^{-1}. The absorption was observed at a wave number of 3440 cm^{-1} when the fiber was treated with NaOH and KOH, which is connected to the C-H strain vibration in cellulose and hemicellulose [14, 23]. Peaks at 1727 cm-1 were attributed to the C=O bending vibration of OH groups in hemicellulose compounds for raw fibers, as seen in **Fig. 4**. In the case of PFs treated with 5% NaOH and 5% KOH, the strength of their peaks was not apparent. The presence of hemicellulose compounds in the fibers was reduced after alkali treatment. The positions of the peaks, as well as the chemical assignment groups of the PFs, are shown in **Table 2**.

The most prevalent chemical compositions of fibers that affect chemical, physical, and tensile strength features are cellulose, lignin, hemicellulose, and wax. **Table 3** illustrates the chemical compositions of raw, as well as NaOH, and KOH-treated PFs. The cellulosic fiber swelled due to alkali treatments, this also removed hemicellulose, lignin, and other impurities from the surface of the PFs. A higher cellulose percentage improves mechanical properties, while a high hemicellulose content reduces fiber strength by promoting cellulosic microfibril disintegration [14].

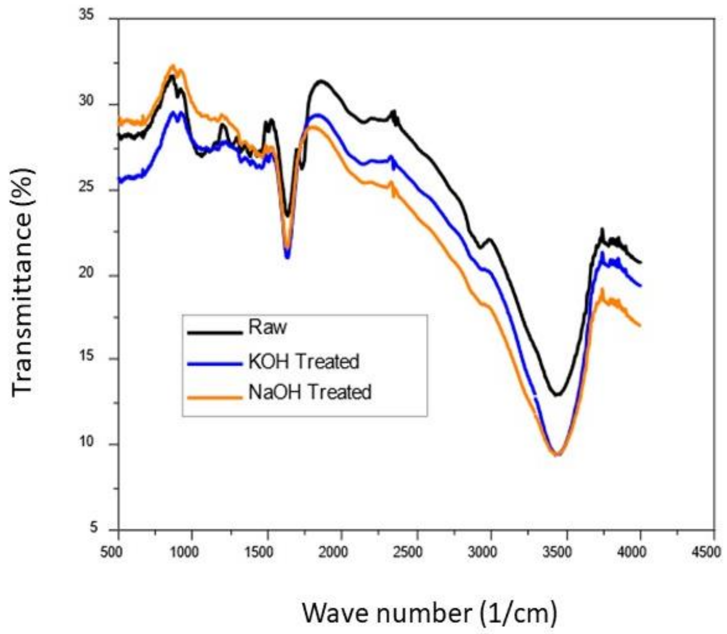


Fig.4. FTIR of PFs before and after NaOH and KOH treatments.

Table 2. PFS fibers' peak location and chemical assignment group

Wavenumber (cm^{-1})			Assignments
raw	NaOH treated	KOH treated	
3473	3440	3440	Stretching -cellulose's O-F vibration and hydrogen bonding of hydroxyl groups.
2958	2941	2957	C-H stretching vibration of cellulose and hemicellulose components on cellulose and hemicellulose/alkyl C-H stretching
2359	2309	2343	C-C alkynes group
1727	-	-	CO stretching of Hemicellulose C=CO stretching of carboxylic acid and ester group of hemicellulose
1645	1628	1628	CO stretching of lignin
1529	1512	1462	Lignin/aromatic C=C stretching in lignin
1063	-	-	Lignin, hemicellulose, cellulose C-O stretching vibration on cellulose.
847	897	847	C–H bond of aromatic hydrogen from lignin compound
465	414	531	COOH bending band and the CH2 symmetric bending

3.4 Chemical Properties analysis of PFs

The elimination of the majority of the hemicellulose components has an effect on the PFs properties, and morphology. The amount of lignin in the NaOH and KOH-treated fiber was reduced by 19.09% and 16.687%, respectively compared to the PFs raw. Furthermore, **Table 3** shows that treated PFs had significantly higher cellulose content than the raw. This wide range is presumably because PFs in nature contain more cellulose than hemicellulose, lignin, and other compounds. As indicated in **Table 3**, treatment with NaOH and KOH increases the content of cellulose.

Table 3 Chemical composition of raw, PFs treated NaOH, and PFs treated KOH.

Sample Codes	Composition			
	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Silika (%)
PFs Raw	48.21	24.51	15.84	0.76
Treated (5% NaOH)	52.83	19.83	14.78	0.68
Treated (5% KOH)	50.62	20.42	14.83	0.71

3.4 XRD analysis

Fig. 5 shows the XRD spectra of raw, as well as the alkali-treated PFs, the curve for the samples presents two primary sharp peaks with an intense angle at $2\theta = 15.5^{o}$ and 22.53^{o}, respectively, which are part of the crystallography (1 0 1) and (0 0 2). According to Sari et al. [3], a sharp peak angle of 22.53^{o} indicates the presence of type I_{β} cellulose, but a low peak angle suggests the presence of amorphous compounds from PFs such as wax, lignin, and hemicellulose. The percentage crystallization index of each fiber sample was calculated using equation 5, as illustrated in **Fig. 6**. In addition, after NaOH and KOH treated, the crystallinity index of raw PFs namely 50.93% increased to 51.34 % (fiber treated NaOH) and 55.21% (fiber treated KOH) respectively. Alkaline solutions dissolve wax, hemicellulose, and impurities, as well as non-crystalline components on the fiber surface, culminating in a rise in crystallinity index [17, 24, 23, 25].

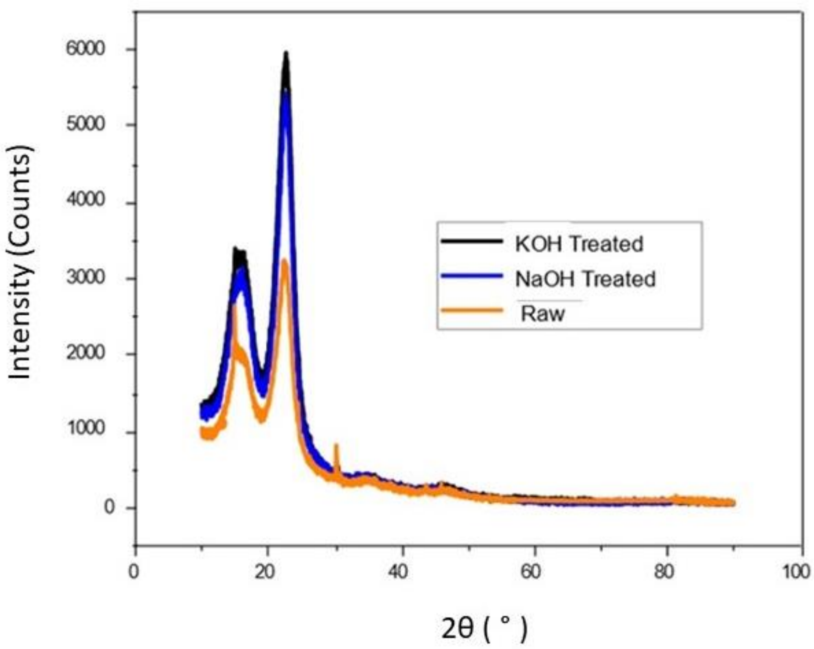


Fig.5. XRD spectra of raw and alkali treated PFs

KOH-treated PFs samples had a higher crystallinity index than pineapple leaves (54% [26], Sisalana Agave (78%) [27]. The increased crystallinity of chemically treated fibers coincides with their higher tensile strength [28]. This suggests that raw PFs, NaOH-treated PFs, and KOH-treated PFs have the potential to be employed as fillers in composite materials.

3.5 Tensile Strength of PFs

As according **Table 4**, the tensile force of PFs treated with NaOH and KOH was increased by 43.27% and 14.18%, respectively, when compared to the PFs raw. The greatest tensile force divided by the surface area of the fibers generates the strength, which is represented in cN/tex. Single PFs treated with NaOH have a stronger toughness than raw or those treated with KOH (**Fig. 7**). Higher concentrations of NaOH and KOH have been shown to lower fiber toughness [29,30].

Table 4. Mechanical properties of raw, NaOH and KOH treated PFs

Sample codes	Tensile Strength (MPa)	Elongation (%)	Modulus of Elasticity (MPa)
Raw	1710.7 ± 76	3.518 ± 0.93	48547 ± 1927
NaOH treated	2450.9 ± 84	4.238 ± 1.08	59405 ± 2085
KOH treated	1953.28 ± 69	4.117 ± 0.9	56304 ± 2108

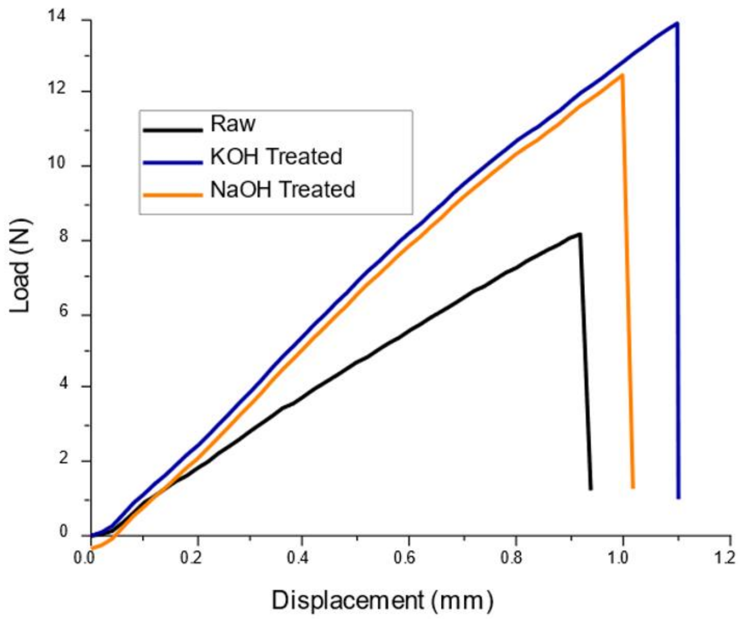


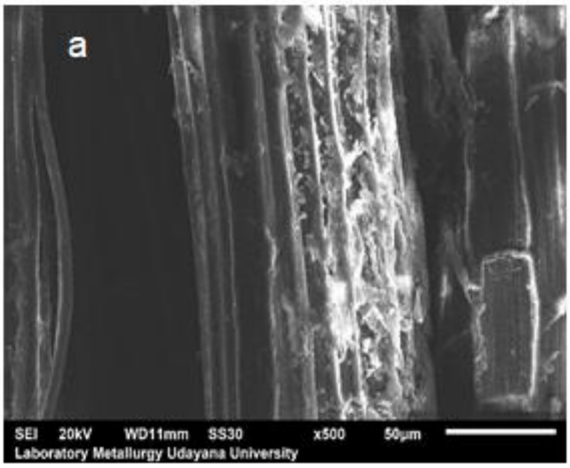
Fig.7. Displacement-load of raw and treated PFs

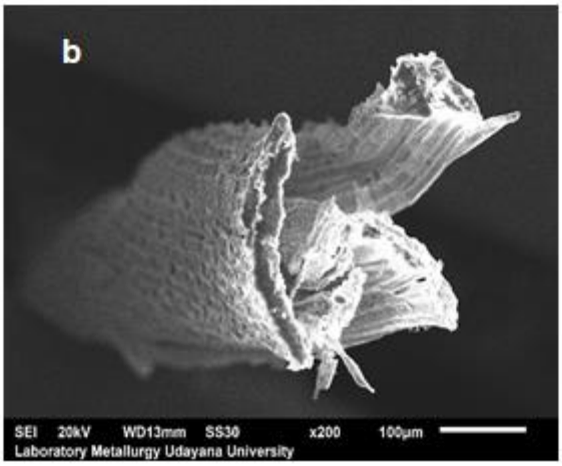
After the alkali treatment, the tensile characteristics of the PFs improved because the cellulose content increased and the fiber diameter reduced as the hemicellulose and lignin components were lost. With NaOH and KOH treatment, the modulus of elasticity increased by 22.37% and 15.98%, respectively (**Table 4**). The tensile strength of these PFs is higher than that of coco fiber, with a value of 280.94 MPa [31], and bamboo at 360 MPa [32]. This means that PFs can replace coco and bamboo fiber as a filler in polymer composites.

3.6 SEM analysis

As depicted in **Fig. 8a**, the raw and alkali-treated surfaces of PFs were covered with fiber impurities such as wax, lignin, or hemicellulose. Meanwhile, due to impurity deposits which preserve the cellulose as well as the presence of a swallow groove and several lumens in the

PFs bundle, the fiber cross-sectional surface in Fig. 8b indicates that the fiber surface is still smooth. After the PFs were treated with NaOH as shown in **Fig. 8c-d** and Fig. **8e-f**, the impurities were removed from the surface, thereby making the samples more fibrillar with smaller diameters compared to the raw (**Fig. 8(c-d**) or KOH-treated (**Fig. 8(e-f**), As a result, the tensile strength increased. The surface of the NaOH-treated PFs is rougher than that of the raw and KOH-treated PFs. This will allow the PFs treated with NaOH to create interlocking mechanics with a better matrix as a polymer composite reinforcement. Finally, the composite with NaOH-treated fiber reinforcement outperformed the KOH in terms of mechanical parameters.





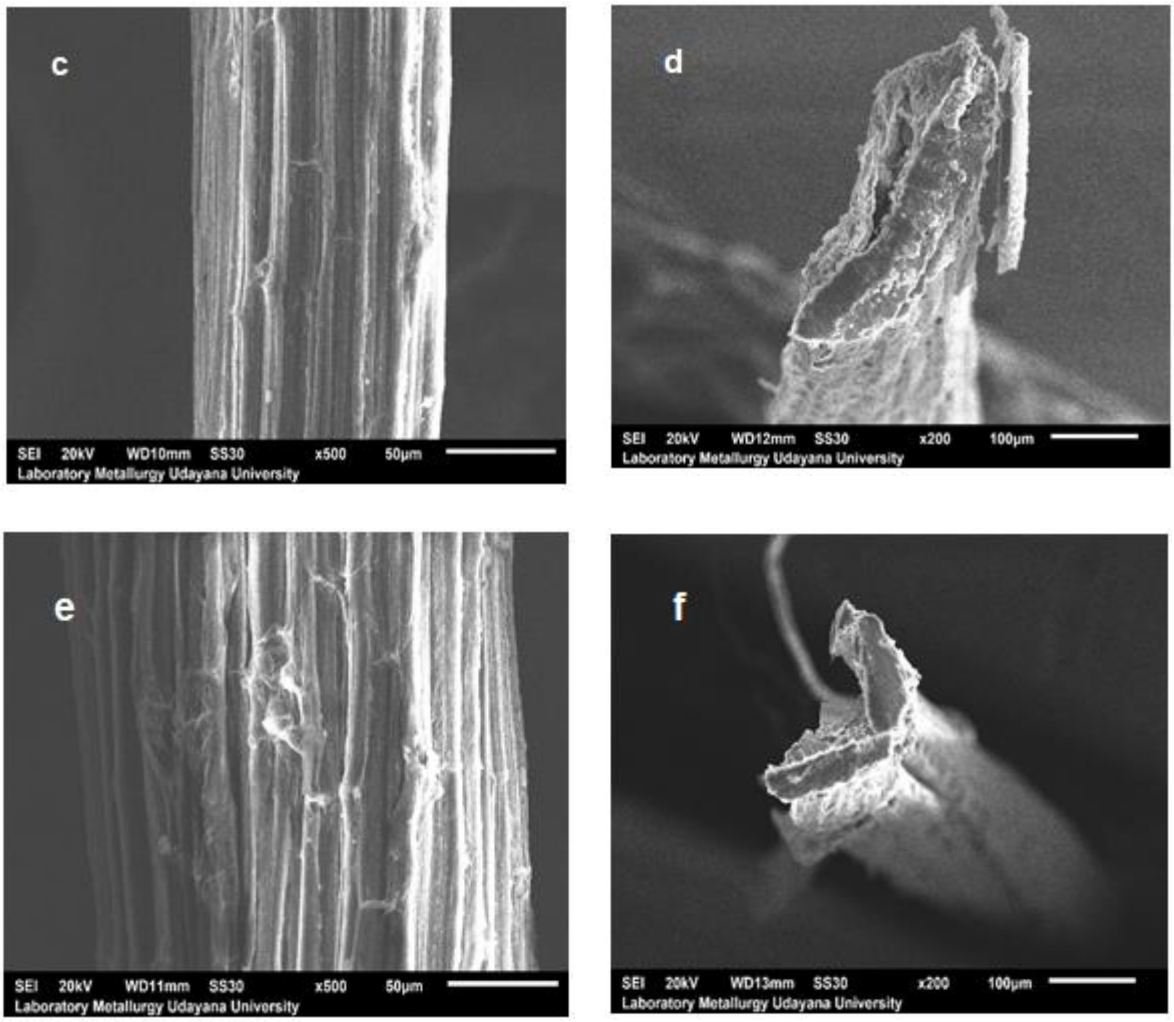


Fig.8. SEM images of PFs, (a-b) raw, (c-d) NaOH treated and (e-f) KOH treated.

3.7 TGA analysis

Fig. 9 depicts a three-stage process of changing fiber mass as temperature rises from 28 to 600^{o} Celsius. The decomposition temperature of PFs treated with NaOH and KOH is greater than the raw types.

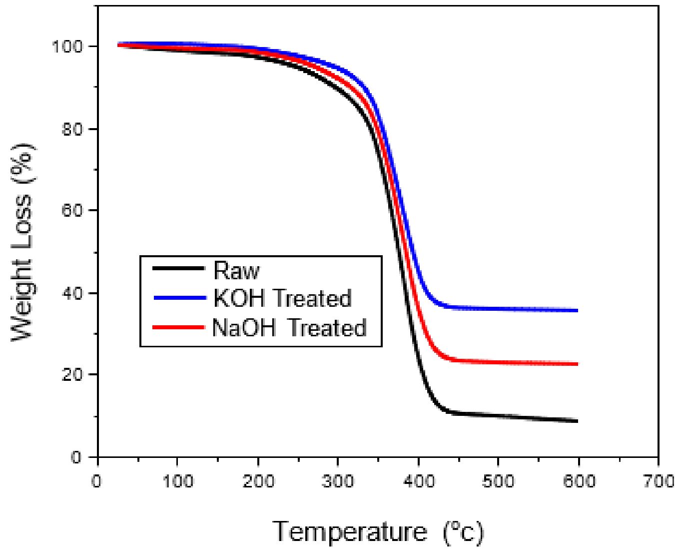


Fig. 9. TGA of *Paederia Foetida* fiber

Fig. 9 shows that only a tiny change occurred in fiber mass at the first stage, which is between 30 and 150 °C. The process of water or moisture evaporation from the fiber occurs at this step, while the main fiber decomposition occurs at temperatures ranging from 200 °C to 420 °C [33-35]. Hemicellulose compounds decompose at 220 °C to 320 °C, followed by cellulose decomposition. Depending on the degree of composition, the amorphous cellulose portion decomposes first, while the high crystalline type is relatively stable and requires high temperatures of approximately 300 °C. The decomposition of lignin follows cellulose degradation and it decomposes slowly from an initial temperature of roughly 150 °C to 900 °C [35-37]. This is because lignin is an extremely hard and inflexible component. Figure 5 also shows that the weight loss of raw fiber is greater than that of fiber after NaOH and KOH treatment because the raw fiber's water content and high hemicellulose content evaporate (see table 1). Furthermore, the leftover heating composition, including ash and other inorganic components, undergoes the final breakdown [36,38].

4. Conclusions

As a lightweight and ecologically acceptable composite reinforcement, the characteristics of PFs have been explored by comparing the types of alkaline treatments KOH and NaOH. The color shift was brighter and cleaner due to the alkaline treatment, while the dissolving of wax, lignin, and hemicellulose of fibers reduced moisture content and fiber diameter. Additionally, the density of PFs tends to rise as the fiber's cellulose concentration increases. After NaOH and KOH treatment, PFS's tensile strength and crystallinity index increased from 1710.7 MPa to 1953.28 MPa and 51.34 % and 55.21%, respectively. The thermal stability also improved, while the shape of the fiber appeared rougher due to the component disintegration. These PFs' mechanical properties have the potential to replace glass fibers and coconut fiber as composite raw materials, particularly in construction applications.

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Comprehensive Characterization Of Novel Cellulose Fiber From *Paederia Foetida* and Its Modification For Sustainable Composites Application

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Fiber derived from the plant *Paederia Foetida* stems (PF) is a novel natural fiber with the capacity to replace glass fibers in composite reinforcement. The technique of modifying the fiber surface with NaOH and KOH is simple and straightforward, generating properties that differ from the raw fiber. Therefore, this study aims to investigate the effects of NaOH and KOH treatments on the surface of *Paederia foetida* fiber (PFs) in relation to the physical, crystal structure, functional groups, tensile strength, thermal and morphological properties compared to raw PFs. The raw fiber was obtained by soaking the PFs rods in water for 10 days, followed by surface treatment with a solution of NaOH and KOH. The results showed that compared to raw fiber, after NaOH dan KOH treated PFs, the diameter and moisture content of the PFs decreased, while the tensile strength increased significantly by 43.27%, and good thermal stability. The highest crystallinity index was obtained from KOH-treated PFs of 79.685%. According to the functional groups in the FTIR observations, NaOH treatment was found to be more effective in removing lignin and hemicellulose components, as well as increasing cellulose compounds in the fiber. The surface morphology was found to be rougher with the loss of impurities after NaOH treatment. Based on the results, raw, NaOH and KOH-treated PFs have the potential to be used as reinforcement for lightweight composite and alternative materials to replace glass fiber and coconut fiber in construction applications.

Keywords: Chemical; KOH; mechanical properties; NaOH; *Paederia foetida* fiber (PFs); physical properties