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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

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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 [1–5]. 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 [1, 9, 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 trees. 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 antirheumatic, analgesics, mucolytics, appetite enhancers, antibiotics, 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 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 [6, 8, 14]. Sari et al. [3] 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).

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 oC. Next, they were washed with distilled water four times to remove the remaining alkaline solution, followed by drying in the sun.

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

Below is the reaction scheme [1, 8].

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + + \text{H}_2\text{O} \quad (1)$$

$$\text{Fiber} - \text{OH} + \text{KOH} \rightarrow \text{Fiber} - \text{O} - \text{K} + + \text{H}_2\text{O} \quad (2)$$

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.

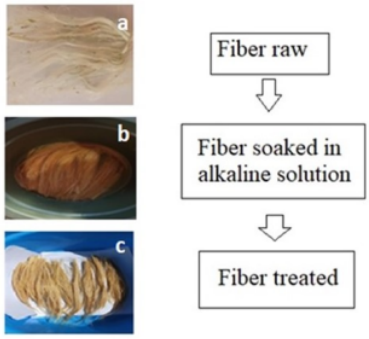

Fig. 2. Alkaline treatment process of PFs (a) fiber raw, (b) alkalization, and (c) dry PFs.

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.

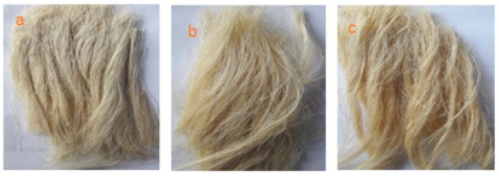

Fig. 3. PFs (a) raw, (b) KOH treated dan (c) NaOH treated.

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. $\rho_w = 0.998\ \text{g/cm}^3$ (standard density of distilled water). All densities were measured using a Metller Toledo measurement kit at $23 \pm 0.2^{\circ}\text{C}$[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 ^{o}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].

$$\%Mc = \frac{W_{fb} - W_{fa}}{W_{fb}} \times 100 \quad (4)$$

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 influences 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].

$$CI = \frac{H_{(002)} - H_{(101)}}{H_{(101)}} \quad (5)$$

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 $H_{(10}0_1)$ is the smaller peak of the amorphous fraction at $2\theta = 18.23°$.

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 °C to 600 °C using a thermogravimetric analyzer (NETZSCH STA 2500).

3. Result discussions

3.1. Fiber density and diameter analysis

Table 2 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].

3.2. Moisture content analysis

The NaOH and KOH treatments reduced the moisture content of the PFs, as demonstrated in Table 2. 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, 23].

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, 24]. 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 1.

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.

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.

3.5. XRD analysis

Fig. 5 shows the XRD spectra of raw, as well as the alkali treated PFs. The curve for the samples presents two pri-

Table 1. PFS fibers' peak location and chemical assignment group.

Wavenumber (cm^{-1}) Raw	NaOH treated	KOH Treated	Assignmennts
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 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

Table 2. 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

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

	Composition			
Sample Codes	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

mary sharp peaks with an intense angle at $2\theta = 15.5°$ and 22.53°, 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° 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, 24–26].

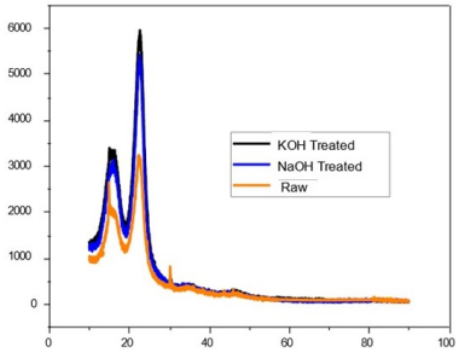

Fig. 5. XRD spectra of raw and alkali treated PFs.

KOH-treated PFs samples had a higher crystallinity index than pineapple leaves (54% [27], Sisalana Agave (78%) [23]. 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

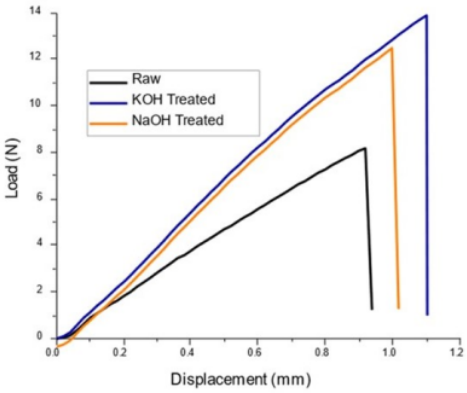

Fig. 6. Displacement-load of raw and treated PFs.

PFs have the potential to be employed as fillers in composite materials.

3.6. Tensile Strength of PFs

As according to 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].

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

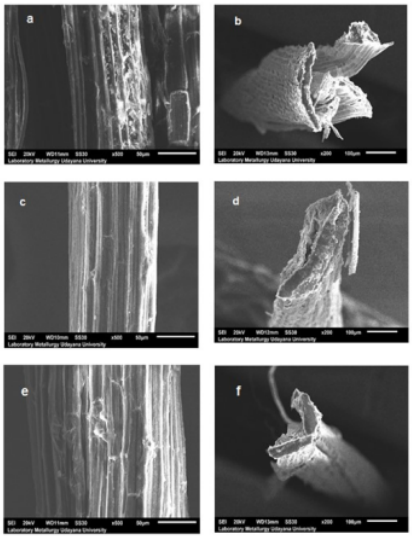

Fig. 7. SEM images of PFs, (a-b) raw, (c-d) NaOH treated and (e-f) KOH treated.

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

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.

3.7. 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.

3.8. TGA analysis

Fig. 8 depicts a three-stage process of changing fiber mass as temperature rises from 28 to 600 ^{o}C. The decomposition temperature of PFs treated with NaOH and KOH is greater than the raw types.

Fig. 8 shows that only a tiny change occurred in fiber mass at the first stage, which is between 30 and 150 ^{o}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 ^{o}C to 420 ^{o}C [33–35]. Hemicellulose compounds decompose at 220 ^{o}C to 320 ^{o}C, followed by cellulose decomposition. Depending on the degree of composition, the amorphous cellulose portion decomposes first, while the high crystalline type

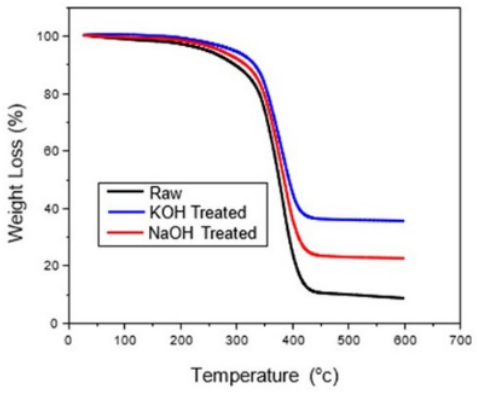

Fig. 8. TGA of *Paederia Foetida* fiber

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 2). 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.

5. Acknowledgment

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