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Characteristics of bio-composites from Polyvinyl alcohol reinforced micro-cellulose fibers of *Paederia foetida*

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Abstract. *The use of renewable natural fibers as reinforcements in biodegradable matrices has been boosted by the development of sustainable polymer materials. The purpose of this work is to examine the properties of bio-composites made of polyvinyl alcohol (PVA) reinforced with micro-cellulose fibers that are isolated from the underused plant resource Paederia foetida (MSPf). After retting and alkaline and bleaching treatments, micro-cellulose was obtained and added to PVA at a weight percentage of 5% utilizing the solution casting technique. The biocomposites' mechanical, thermal, water absorption, and morphological characteristics were assessed. When MSPf was added, PVA's tensile strength rose from 20.69 MPa to 23.10 MPa, and its elastic modulus increased dramatically, suggesting better stiffness and efficient stress transfer. The improvement is ascribed to the hydroxyl groups of PVA and cellulose having strong hydrogen bonds and good interfacial adhesion. Due to limited polymer chain mobility, elongation at break decreased, but the composite showed better structural integrity. After 36 h, water absorption attained an equilibrium value of 1.461%, indicating the creation of a compact fiber-matrix network. When compared to clean PVA, thermogravimetric analysis showed better thermal stability, and SEM verified strong interfacial compatibility and homogeneous dispersion. These results show that micro-cellulose derived from Paederia foetida is a viable sustainable reinforcement that can improve the performance of bio-composites based on PVA.*

Keywords: *Biocomposites; micro-cellulose; Paederia foetida; tensile strength; thermal stability.*

Type of the Paper: Regular Article.

1. Introduction

The pursuit of sustainable and biodegradable materials has increased due to the growing environmental concerns surrounding petroleum-based polymers. Although conventional synthetic plastics have superior mechanical and chemical qualities, their non-biodegradable nature greatly contributes to long-term environmental contamination [1,2]. In response, bio-composites derived from renewable resources have emerged as promising alternatives, combining environmental compatibility with desirable functional performance [3–5].

Polyvinyl alcohol (PVA) is one of the biodegradable polymers that has garnered a lot of interest due to its exceptional chemical resistance, non-toxicity, biodegradability, and film-

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forming capabilities [6–8]. However, the relatively low mechanical strength and high moisture sensitivity of neat PVA limit its broader application. Reinforcement using natural fibers, particularly cellulose-based materials, is an effective strategy to improve its mechanical, thermal, and structural performance [9–11]. For the creation of bio-composite materials, micro-cellulose—which is created by combining mechanical and chemical processes—has garnered a lot of interest in the materials research community. Its favorable characteristics—such as low density, high aspect ratio, a high level of mechanical and large specific surface area—which facilitate efficient stress transfer inside polymer matrices are the reason for this interest [12–14].

Cellulose is a particularly appealing bio-reinforcement because of its high inherent mechanical strength, low density, biodegradability, and renewability [15,16]. Cellulose reinforcement can be broadly divided into three categories based on its size scale and processing method: macro-fibers, micro-cellulose, and nano-cellulose. Each of these categories has unique structure-property properties. Although nano-cellulose offers remarkably high surface area and reinforcing efficiency, the material is susceptible to agglomeration during processing, particularly in hydrophilic polymer matrices, and its separation processes are frequently expensive and energy-intensive [17,18]. On the other hand, macro-scale natural fibers are generally less effective at transferring stress within polymer matrices and have a smaller interfacial contact area, although being simpler to prepare. An intermediate option that strikes a balance between processing viability, dispersion stability, and reinforcing effectiveness is micro-cellulose. Its surface functionality and moderate aspect ratio enable efficient interaction with hydroxyl-rich polymers like PVA without the complications of nanoscale systems.

Interfacial interactions significantly control cellulose's reinforcing effectiveness in PVA systems. Both PVA and cellulose include a large number of –OH groups that promote hydrogen bonding, which may enhance stress transmission and limit the mobility of polymer chains [6]. In addition to affecting moisture uptake and thermal degradation behavior, such interactions may raise tensile strength and modulus. However, because of limited chain flexibility, improvements in stiffness are frequently accompanied with decreased elongation at break, underscoring the significance of comprehending structure–property trade-offs in these composites.

The rapidly growing and extensively accessible plant *Paederia foetida* is a promising but underutilized source of natural cellulose. The *Paederia foetida* plant's fibers offer a great deal of potential for use as a source of microcellulose to reinforce PVA biocomposites. This plant has a rather high cellulose content and tensile strength, which have been reported to be roughly 48.21–52.85% and 2450.9 ± 84 MPa, respectively, in addition to being readily accessible and frequently considered a weed [19]. The utilization of this underexploited biomass not only enhances material performance but also supports sustainable resource utilization. Previous work by Azlan et al. [20]

developed PVA-based biocomposites reinforced with corn husk waste, and pineapple leaf fiber using the solvent casting method. Their study showed that PALF@$BaCO_3$ filler significantly improved the tensile strength and Young's modulus of the composite, with optimum values of 18.157 N/mm^2 and 3.13052 N/mm^2, respectively, achieved at 40 wt% $BaCO_3$. However, increasing the PALF content beyond this level did not result in significant additional improvement in mechanical properties. Although cellulose-reinforced PVA composites have been the subject of several investigations, little research has been done on the structure-property connection of micro-cellulose that is specifically produced from *Paederia foetida* in PVA matrices. Tensile, water absorption, thermal degradation behavior, & morphological examination are all included in a systematic evaluation that has not received enough attention.

Therefore, the goal of this research is to create and describe bio-composites made of PVA reinforced with microcellulose fibers taken from *Paederia foetida*. The resulting composites' morphological, mechanical, thermal, and physical characteristics are assessed to see whether they have the potential to be used as ecologically friendly materials.

2. Materials and methods

2.1. Materials

Paederia foetida plant stems were gathered from the Indonesian riverbanks in Kuta Village, Central Lombok. Sigma-Aldrich provided the polyvinyl alcohol (PVA), which was utilized as the biocomposite matrix and had a 99% hydrolysis degree. Glacial acetic acid (CH_3COOH), sodium hydroxide (NaOH), and sodium chlorite ($NaClO_2$). Every chemical utilized was of analytical quality and didn't require any additional purification.

2.2. Extraction of Cellulose Fibers

After being chopped into 200 mm lengths, *Paederia foetida* stems were submerged in water for around 14 days to allow for bacterial retting. Fibers were easier to separate from the stem tissues because to this technique. The fibers were combed after retting to get more homogeneous fiber bundles and to get rid of any leftover bark and non-fibrous debris. After being completely cleaned with clean water, the extracted fibers were allowed to air dry until the moisture content was between 15 and 20 percent. Fig. 1 shows a schematic diagram of the cellulose fiber production process.

2.3. Isolation of Micro-Cellulose from Paederia foetida (MSPf)

The separation of microcellulose from *Paederia foetida* fibers is shown schematically in Fig. 1. A blender was used to grind the dried fibers after they were cut into 15 mm lengths. To achieve a consistent particle size, the resultant powder was sieved through a 100-mesh screen. Alkalization was the first step in the chemical treatment. A 5% (w/v) NaOH solution was applied to 25 g of fiber powder and heated to 80 °C for one h on a hot plate. Then, using a 5% (w/w) $NaClO_2$ solution, the bleaching process was conducted for an hour at 80 °C while being continuously stirred with a magnetic stirrer.

After each chemical treatment, the samples were washed with running water until neutral pH was achieved. The treated material was labeled as MSPf (micro-cellulose from *Paederia foetida*). Finally, the MSPf powder was dried in an oven until the moisture content reached approximately 8%.

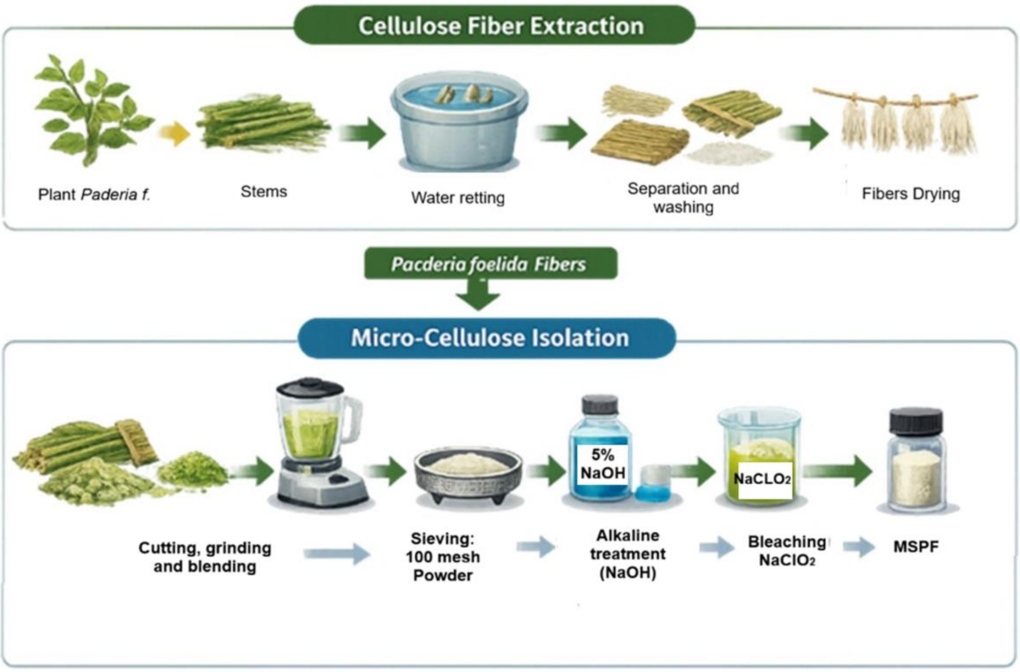

Fig. 1. Extraction and isolation process of micro-cellulose from *paederia foetida* (MSPf)

2.4. Fabrication of Biocomposites (Fig. 2)

To prepare the biocomposite, MSPf was dispersed at a weight percentage of 5% in relation to PVA. 50 ml of distilled water were used to dissolve and scatter 10 g of PVA and the equivalent amount of MSPf. The suspension was heated to 70 °C and agitated at 500 rpm with a magnetic stirrer until gelatinization took place, creating a uniform biocomposite gel. After that, the gel was cast onto a glass plate and degassed to reduce air bubbles and voids. The final biocomposite samples were produced by drying the cast films for 15 h at 50 °C in a vacuum drying oven.

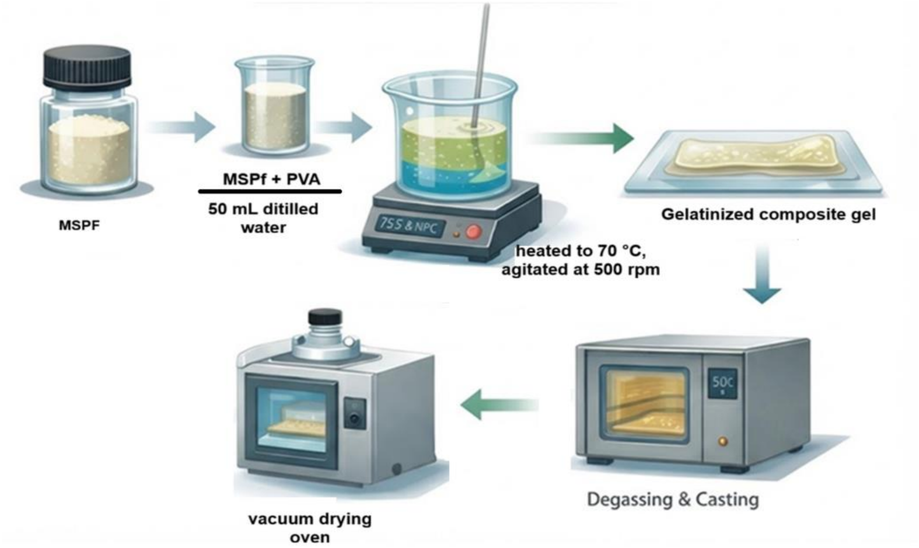

Fig. 2. Fabrication of biocomposites

2.5. Water absorption (WA)

WA of the biocomposite samples was evaluated in accordance with ASTM International standard ASTM D570 [21,22]. Before testing, the samples were dried for 24 h at 50 °C in an oven and then allowed to cool to room temperature in a desiccator. Next, a digital balance with a 0.001 g precision was used to record the initial dry weight (M_0) of each specimen. At room temperature (around 25 °C), the specimens were submerged in distilled water for a set amount of time. Immediately upon immersion, the samples were taken out, the surface water was gently removed with absorbent tissue, and the final weight (M_1) was measured.

The water absorption (WA) was calculated using Equation (1) [22,23]

$$W_a\ (\%) = \frac{M_1 - M_0}{M_0} \times 100 \qquad (1)$$

where M_1 is the weight of the specimen after immersion (g), and M_0 is the initial dry weight of the specimen before immersion (g). Three duplicates of each measurement were made, and the average results were reported.

2.6. Tensile Properties

The tensile properties of the biocomposites were evaluated in accordance with ASTM International standard ASTM D638 [24] using a universal testing machine (RTG-1310, Instron) equipped with a 5 kN load cell. The tests were conducted at a temperature of 26.7 °C and a relative humidity of 56%. The gauge length was set to 57 mm, and the crosshead speed was 2 mm/min [25].

2.7. Thermogravimetric Analysis (TGA)

A TGA-1 device (Mettler Toledo) was used to conduct thermogravimetric analysis in order to assess the biocomposites' thermal stability. With a flow rate of 50 mL/min and an heating rate of 20 °C/min, the measurements were performed in ramp mode between 50 and 1000 °C in a nitrogen environment [26,27].

2.8. Scanning Electron Microscopy (SEM)

The morphology of the biocomposites was examined using a Phenom Pharos Desktop scanning electron microscope (Thermo Fisher Scientific). The observations were conducted at an accelerating voltage of 5 kV. Prior to analysis, the sample surfaces were sputter-coated with a thin layer of gold to improve conductivity.

3. Results and Discussion

3.1 Tensile strength analysis

The clean PVA's stress-strain behavior is shown in Fig. 3a. The tensile strength of MSPf-reinforced PVA biocomposites is displayed in Fig. 3b. After MSPf was added, the tensile strength of neat PVA rose from 20.69 MPa to 23.10 MPa, demonstrating that the applied stress was successfully transferred from PVA to MSPf. Since MSPf and the PVA matrix have a strong interfacial connection, the tensile strength has improved. The hydroxyl (-OH) groups found in PVA and cellulose are plentiful and encourage the creation of intermolecular hydrogen bonds at the fiber-matrix interface. More effective load transmission under tensile stress is made possible by these hydrogen bonds, which also improve interfacial adhesion and lessen fiber pull-out.

Furthermore, MSPf's high aspect ratio and comparatively tiny diameter offer a large specific surface area, expanding the interfacial contact region and fortifying the interfacial bonding. By reducing stress concentration sites and encouraging uniform stress distribution, good MSPf dispersion throughout the matrix also aids in mechanical augmentation. According to these findings, micro-cellulose that has been separated from MSPfworks well as a reinforcing ingredient in PVA-based biocomposites. The tensile strength of the PVA/5%-MSPf biocomposite (23.10 MPa) is higher than that reported for PVA reinforced with 5% CMF from *Jatropha curcas* L. (22.34 MPa) [28], although it remains lower than that of PVA reinforced with 5% CMF from *Agave gigantea* (33.55 MPa) [8]. The differences in performance may be related to variations in fiber crystallinity, aspect ratio, surface chemistry, and dispersion quality among different cellulose sources.

The elongation at break decreased significantly with the addition of MSPf. As shown in Table 1, neat PVA exhibited a high elongation of 122.5% [8], which decreased to 28.35% after the incorporation of 5 wt% MSPf. This reduction happens as a result of the PVA polymer chains' limited mobility and extensibility due to the stiff cellulose microfibers. Chain slippage and plastic deformation under tensile loading are reduced by the more restricted network topology produced by the hydrogen bonding between MSPf and PVA.

The restricted molecular mobility also resulted in a substantial increase in stiffness. The elastic modulus increased from 2.88 MPa for neat PVA to 73.3 MPa for the PVA/5%-MSPf biocomposite. This value is considerably higher than that reported for PVA reinforced with 5% CMF from *Jatropha curcas* (11.48 MPa), indicating a strong reinforcing efficiency of MSPf. The high modulus reflects the formation of a rigid fiber–matrix network supported by strong interfacial hydrogen bonding and effective stress transfer. Via the combined effects of hydrogen bonding, enhanced interfacial adhesion, a high interfacial area, and limited polymer chain mobility, the mechanical performance shows that the addition of MSPf improves the strength and stiffness of PVA.

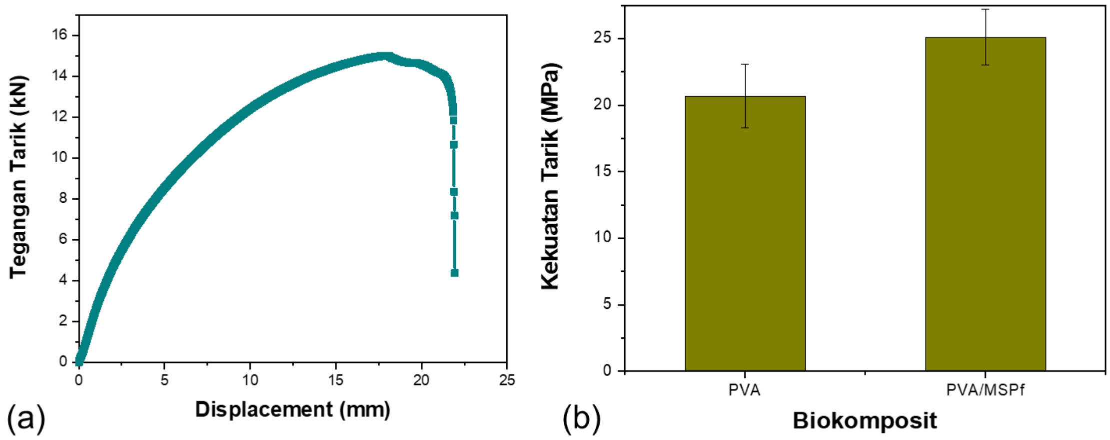

Fig. 3. Tensile strength of MSPf reinforced biocomposite PVA

Table 1. Comparison of biocomposites PVA/5%-MSPf and other biocomposites.

Sample	Tensile strength (MPa)	Elongation (%)	Modulus of Elasticity (MPa)	Reff.
PVA	20.69	122.5	2.88	[8]
PVA/5%-MSPf	23.1	28.35	73.3	Determination
PVA/5%-CMF Jatropha Curcas L (JC)	22.34	15	11.48	[28]
PVA/5%-CMF Agave Gigantea (AG)	33.55	98.65	708.64	[8]

3.2. Water Absorption

The MSPf-reinforced PVA biocomposite's water absorption % as a function of immersion time is displayed in Fig. 4. During the first 12 h of immersion, the created biocomposite showed a comparatively quick water uptake. Following this time frame, the absorption rate steadily dropped and got closer to equilibrium. After about 36 h, saturation was achieved, with a maximum absorption rate of 1.461%.

The presence of hydrophilic fillers, matrix composition, and immersion duration all affect water absorption in PVA-based biocomposites. The presence of hydroxyl (-OH) groups in PVA and cellulose facilitates water transport by forming hydrogen bonds with water molecules. Natural fillers like micro-cellulose usually make the composite more hydrophilic and offer more channels for moisture to enter through microvoids and interfacial areas [29–31]. However, the relatively low water absorption observed in this study indicates strong interfacial interaction between MSPf and the PVA matrix. The formation of intermolecular hydrogen bonding between the hydroxyl groups of PVA and cellulose reduces the number of free hydrophilic sites available to interact with water [7,20]. In addition, good dispersion and interfacial adhesion minimize interfacial gaps and voids, thereby limiting water diffusion into the composite structure. As a result, the composite exhibits improved resistance to moisture uptake despite the presence of a hydrophilic filler.

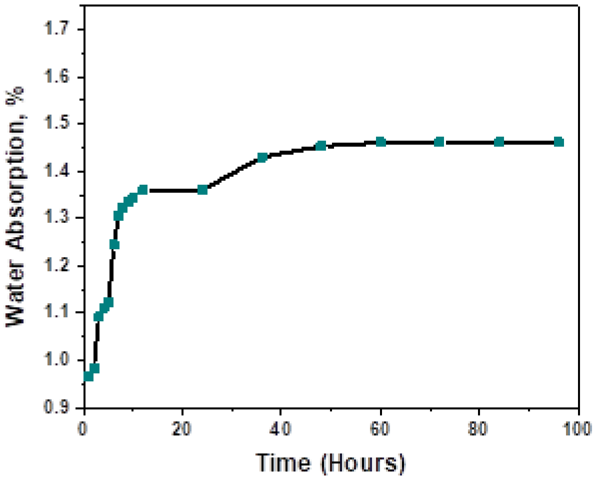

Fig. 4. Water Absorption of biocomposite PVA/MSPf.

This behavior is consistent with previous findings reported by Judawisastra et al. [32], who observed that the incorporation of PVA into starch-based bioplastics significantly reduced water absorption. The water uptake of pure tapioca starch films was extremely high (up to 495%), but it dropped to 271% and 251% when 17% and 29% PVA were added, respectively. The reduction was ascribed to the interaction between PVA and the natural filler's hydroxyl groups, which reduced the material's overall hydrophilicity. By limiting water diffusion and improving the material's moisture resistance, the PVA/MSPf biocomposite's low equilibrium water absorption points to the development of a compact network structure controlled by strong hydrogen bonds and efficient interfacial adhesion.

3.3. Thermogravimetric analysis (TGA)

Fig. 5 shows the TGA curve of the PVA biocomposite reinforced with MSPf. Three different phases of weight loss are visible in the thermal degradation behavior, suggesting a multi-step disintegration mechanism. A weight loss of roughly 2–3.5% was seen during the first stage, which took place in the temperature range of 80–100 °C. This area is linked to the release of bound water molecules and the evaporation of physically adsorbed moisture. Both PVA and cellulose include hydroxyl (–OH) groups, which encourage hydrogen bonding with water and retain moisture released at relatively moderate temperatures. A significant weight loss of up to 80% was noted during the second stage of degradation, which occurred between around 250 °C and 395 °C. This phase is equivalent to the thermal breakdown of the primary polymer structure, which includes side group removal, chain scission, and the breakdown of the PVA and cellulose backbones. In the PVA/MSPf biocomposite, the primary thermal degradation phase is represented by the simultaneous depolymerization of cellulose and the disintegration of PVA molecular chains.

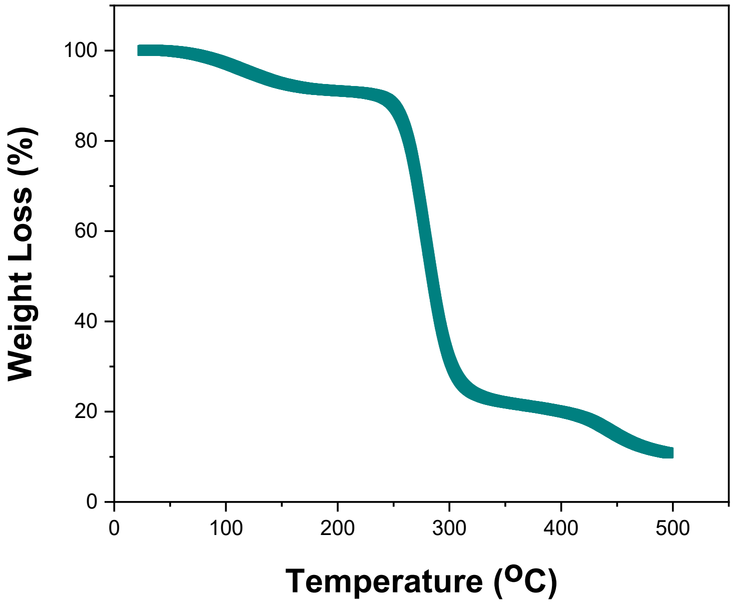

Fig. 5. TGA of biocomposite PVA/MSPf.

The third stage occurred in the temperature range of 400–500 °C, with total weight loss reaching approximately 89%. This stage is attributed to the final decomposition and carbonization process, involving the breakdown of residual carbonaceous structures and the cleavage of stable C–C bonds within the composite system. The thermal stability of the PVA/MSPf biocomposite is comparable to that reported for PVA-based composites reinforced with montmorillonite (MMT), which exhibit total weight loss of around 90% [33]. In general, neat PVA shows greater weight loss at lower temperatures compared to filled systems, indicating lower thermal resistance.

The PVA/MSPf biocomposite's enhanced heat stability can be attributed to the micro-cellulose's barrier and reinforcing properties. The hydroxyl groups in PVA and cellulose form strong intermolecular hydrogen bonds that limit polymer chain mobility and postpone heat breakdown. Furthermore, the more compact structure formed by the distributed MSPf slows the diffusion of volatile degradation products by acting as a barrier to mass and heat transfer. The composite is more resistant to thermal degradation as a result of this interfacial contact and structural confinement. Through the combined impacts of hydrogen bonding, molecular motion restriction, and the creation of a thermally resistant fiber-matrix network, the TGA data verify that the addition of MSPf enhances thermal stability.

According to the thermogravimetric measurement, the PVA/MSPf biocomposite showed good heat resistance before significant structural breakdown, with an onset degradation temperature (T_{on}set) of about 260 °C. The fast depolymerization of PVA chains and cellulose backbone cleavage coincided with the maximum degradation temperature (Tmax), which was around 315 °C. The residual char concentration stayed at about 12% at 600 °C, indicating the development of stable carbonaceous structures. Strong hydrogen bonds and interfacial contacts between PVA and MSPf, which limit polymer chain mobility and postpone heat breakdown, are responsible for the comparatively high char yield.

3.4. Morphological Analysis (SEM)

The MSPf/PVA biocomposite's cross-sectional SEM image is displayed in Fig. 6. The micrograph shows that the PVA matrix contains finely dispersed MSPf, which manifests as tiny, evenly spaced particles. There is no discernible agglomeration or voids, which suggests that MSPf and the PVA matrix are well-dispersed and compatible. As a hydrophilic polymer, PVA's compatibility with cellulose-based fillers is mostly determined by the hydroxyl (-OH) groups of both substances forming intermolecular hydrogen bonds. Interfacial adhesion is improved by these interactions, which makes it possible for the microcellulose to be firmly implanted in the matrix. Strong interfacial bonding facilitates efficient stress transfer and lessens the possibility of fiber pull-out, which is in line with the enhanced mechanical qualities noted.

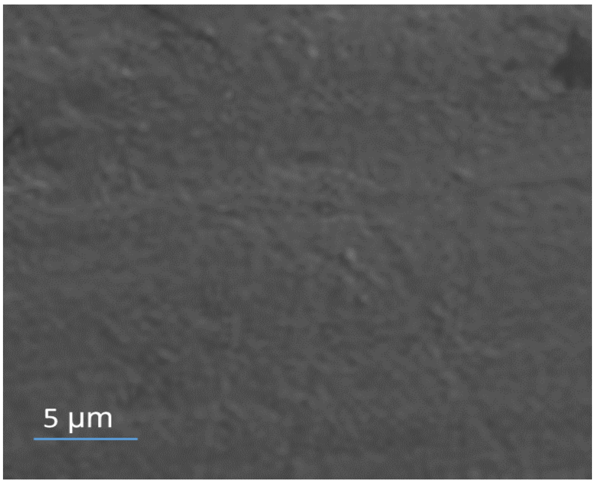

Fig. 6. SEM photos of biocomposite PVA/MSPf.

Additionally, the very compact and smooth fracture surface points to the development of a dense network structure of polymers and fibers. Stress concentration site development is reduced and particle aggregation is prevented at low filler content (≤10 wt%). Strong intermolecular interactions between fibers cause cellulose particles to aggregate at greater filler loadings, which can decrease compatibility and result in heterogeneous morphology. According to earlier research, pristine PVA has comparatively smooth fracture surfaces with tiny microcracks brought on by stress-induced crack propagation. Because micro-cellulose deflects cracks and dissipates energy at the fiber–matrix contact, it usually produces a rougher surface morphology [8]. This change in fracture behavior indicates improved interfacial interaction and enhanced resistance to crack propagation. The SEM observations (Fig. 6) confirm that the addition of MSPf leads to homogeneous dispersion, strong interfacial adhesion through hydrogen bonding, and the formation of a compact microstructure, all of which contribute to the improved mechanical and thermal performance of the PVA/MSPf biocomposite.

4. Conclusions

An underutilized natural resource, an MSPf/PVA biocomposite, was successfully developed in this work. A decrease in elongation at break was noted as a result of limited polymer chain mobility, however the addition of 5 (% weight) MSPf improved the PVA matrix's tensile strength and markedly raised its stiffness. Strong interfacial and intermolecular hydrogen bonds between the hydroxyl chains of PVA and cellulose were the main causes of the increases in mechanical performance. The biocomposite exhibited low water absorption (1.461% at equilibrium) and improved thermal stability, indicating the formation of a compact and well-integrated fiber–matrix network. SEM analysis confirmed homogeneous dispersion of MSPf and good interfacial compatibility without significant agglomeration, which contributed to the overall performance enhancement. This work is interesting because it uses micro-cellulose that was separated from *Paederia foetida* as strengthening agents for PVA, showcasing its potential as an efficient and sustainable filler for biodegradable composite applications. To increase the potential use of PVA/MSPf biocomposites in eco-friendly materials, future research should concentrate on filler loading optimization, surface modification of MSPf to further improve interfacial bonding, and assessing long-term characteristics like biodegradability, barrier performance, and durability.

Data availability statement

Data will be made available on request.

CRediT authorship contribution statement

Nasmi Herlina Sari: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology. **Muhammad Nabil Fadhlurrohman**: Project administration, Writing – original draft, Writing – review and editing. **Suteja**, **Syarif hidayatullah**: Resources, Software, Supervision, Validation, Visualization. **I Putu Lokantara**: Writing – review and editing

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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