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Comprehensive Extraction and Multiscale Characterization of a Novel Lignocellulosic Fiber From *Microcos paniculata* Bark

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ABSTRACT

Natural fibers are gaining popularity as eco-friendly and biodegradable reinforcements in polymer composites. This project is aimed at extracting and analyzing organic cellulose fibers from the barks of *Microcos paniculata* plants, employing a 9-day water retting process followed by alkali treatment with 5% (*w/w*) NaOH. Both alkali-treated and untreated fibers were analyzed using advanced techniques to assess their key properties. The chemical composition of the untreated fibers, determined by the TAPPI method, was found to consist of 55% cellulose, 20% hemicellulose, and 18% lignin, with the cellulose content increasing to 64% after alkali treatment. Surface morphology analysis using scanning electron microscopy revealed a smoother surface for untreated fibers and a rougher surface for alkali-treated fibers. The density and moisture regain of the fibers were measured as 1.28 g/cm³ and 12.61%, respectively. Fourier transform infrared spectroscopy confirmed the presence of functional groups, which correspond to the cellulosic polymers, and indicated the removal of natural impurities after alkali treatment. The crystallinity index and tenacity of the alkali-treated fibers increased from 63% to 69% and from 34.85 to 38.59 g/tex, respectively, compared to the untreated fibers. Thermogravimetric analysis demonstrated excellent thermal stability, with the fibers exhibiting temperature resistance up to ~240°C and 24.09% char formation at 993°C. These results suggest that the isolated natural cellulose fibers from *M. paniculata* have significant potential as a durable reinforcement material in polymer composites.

1 | Introduction

Growing demand for eco-friendly materials in the industrial sector has caused a sharp turn toward nature-derived fibers from synthetic polymers. Global warming, depleting resources, and growing environmental issues, coupled with customer focus, have prompted manufacturing industries to go in search of

new-generation materials that are able to substitute traditional nonrenewable reinforcing materials with eco-friendly and biodegradable materials [1]. This reflects global recognition of the importance of environmental preservation, leading to a growing emphasis on the development and adoption of biodegradable and bio-based materials [2]. Material scientists are thus trying their level best to scout for new materials according to the principles of

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sustainability and a circular economy [3]. Other than this, the new trends have been fueled by adverse environmental impacts from the extensive usage of fossil fuel-based products, such as effects on human health, escalated landfill waste, greenhouse gases, and pollution. Therefore, to offset the environmental impact of petroleum-derived products, there is an increasing shift toward the use of biodegradable and recyclable green materials [4]. As a result, the adoption of green materials is becoming progressively popular. Natural fibers provide an appropriate alternative in the form of renewable, biodegradable, and recyclable raw materials that can be used as reinforcement agents in the production of green composites. Natural fibers possess numerous advantages over man-made fibers, including ease of processing, cost-effectiveness, renewability, minimal contribution toward global warming, and biodegradability. In addition, they have a promising future in plastics, composites, automotive uses, packaging, and other applications due to their excellent properties, such as high mechanical strength, biodegradability, renewability, nontoxicity, and good thermal and sound insulation. Cellulosic fibers, natural, renewable biopolymers abundantly found in plants, represent an environmentally friendly alternative [3–5].

Cellulosic fibers help lower costs compared to synthetic fibers when used in the production of eco-friendly materials like green composites. Green composites usually consist of natural fibers obtained from plant origin with various chemical constituents such as cellulose, hemicellulose, lignin, wax, and ash [6, 7]. These materials are gaining prominence in material technology due to their desirable properties, including lightweight, high strength-to-weight ratio, biodegradability, simple manufacturing, and improved mechanical properties compared to traditional synthetic fiber-reinforced composites [8]. Green composites are now applied in many industries, including construction, aerospace, sporting equipment, household consumer products, motor vehicle components, and the paper industry. The growing emphasis on sustainability in manufacturing has sparked intense interest in the utilization of fibers from nature as reinforcing materials in polymer composites and other technologies from both industry and academics [9, 10]. As a result, there is currently an ongoing search for novel disposable and environmentally friendly materials to replace existing synthetic fibers that provide benefits such as versatility, biodegradability, minimal density, reduced hazard qualities, and cost. Natural fibers are derived from various parts of plants, including bark, leaves, stems, buds, and roots. Seed fibers (e.g., cotton, coir, and kapok), bast fibers (e.g., jute, flax, hemp, ramie, kenaf, banana, and bamboo), and leaf fibers (e.g., sisal, abaca, and pineapple) are the most common commercial cellulosic fibers [11].

Microcos paniculata is a woody shrub or small tree of the Malvaceae family (hitherto in Tiliaceae), endemic to Southeast Asian tropical and subtropical areas such as India, Bangladesh, Myanmar, southern China, and Southeast Asia. It is popularly referred to as “Asar” or “Pichondi” in Bengali [12]. *Microcos paniculata* is an evergreen species that typically grows up to 2–5 m in height and thrives across diverse ecological conditions ranging from lowland forests to deteriorated lands. The plant features alternate, simple leaves with serrate edges, small yellow panicle flowers, and drupe fruits. Although it is highly valued in conventional medicine, its ecological flexibility and widespread growth throughout South and Southeast Asia make it significant from the perspective of

conservation and potential agricultural applications [13]. *Microcos paniculata* possesses a unique ability to shed its outer bark naturally without harming the tree, similar to paper birch and eucalyptus trees. Bark regrowth is facilitated by the living cambium layer, which creates new protective tissue while it sheds. In healthy, mature trees, initial bark regeneration begins within 6–12 months, while complete restoration normally takes one to two growing seasons, depending on environmental conditions [12, 13]. Natural exfoliation occurs in patches as outer dead layers are shed, but inner layers remain intact to protect vascular tissues. The period following the monsoon (October–November), when the plant has finished its active development phase and the ambient moisture levels are reasonable, is when *M. paniculata* bark can be gathered. Sustainable harvesting is possible if less than 50% of the bark circumference is removed at once and the cambium layer remains undamaged—a practice that could make this species valuable for renewable fiber production [14].

Water retting refers to a microbial degradation process in which bast fibers from plants such as jute, flax, and hemp are soaked in water, typically at 20°C–35°C [15]. During retting, both anaerobic and aerobic bacteria (*Clostridium* and *Bacillus*) and various fungi break down pectin, hemicellulose, and lignin enzymatically, thereby loosening the woody core from bundles of fibers [16, 17]. The retting time (7–21 days) is regulated by the condition and temperature of the water and the amount of microbial life, with an optimum pH of 6.5–7.5. Perfect retting enables maximum strand separation in decortication, producing uniform, high-tenacity fiber with a minimum of damage [18]. Overretting promotes cellulose disintegration and loss of fiber strength, while underretting results in poor separation. Despite concerns such as unpleasant smells and water contamination caused by decomposing organic matter, water retting is still essential for the production of long, spinnable fibers with good mechanical properties, making it the primary choice for ecological textile processing. Although mechanical and chemical methods of fiber extraction exist, they often compromise fiber quality and reduce environmental sustainability [19, 20].

For natural fibers to function effectively as reinforcements in composite materials, strong adhesion between the natural fiber and the matrix is essential. However, the inherently hydrophilic nature of natural fibers disrupts this adhesion, resulting in weak interfacial interactions with the typically hydrophobic polymer matrix. To address this challenge, researchers have developed various surface modification techniques to enhance the compatibility and bonding of natural fibers within composite systems [21–23]. Among these methods, chemical treatments such as alkali treatment, maleation, silanization, coupling agents, acetylation, acrylation, and benzoylation have been the most prevalent due to their efficiency and fast capability to alter the surface properties of the fibers. Of these, alkali treatment has gained significant attention due to its low cost and extensive use in natural fiber treatment [24, 25]. The primary goal of alkali treatment is to remove superficial impurities and noncellulosic components like hemicellulose via a swelling reaction that distinguishes the fibers into fibrils and enhances a densely packed cellulose chain by alleviating internal fatigue without devaluing the cellulose. Ultimately, the treatment enhances the fibers' mechanical performance, surface effectiveness, and active area on the surface [26]. Fibrillation of fibers induced by the alkali treatment greatly improves the fiber–matrix adhesion in polymer

composites by increasing the effective surface area for resin wetting [27]. Treatment with alkaline substances also activates fibers by breaking hydrogen bonds and increasing the number of free hydroxyl groups [26].

The *M. paniculata* plant presents a promising source for cellulosic fibers, exhibiting properties comparable to conventional cellulosic fibers and suitable for various commercial and polymer composite applications. Despite its potential, this evergreen plant has yet to be harvested, characterized, and utilized for fiber extraction in composite development. Therefore, this study introduces a novel approach focused on the extraction and characterization of stem fibers from *M. paniculata*, which, to date, have not been previously investigated. This study is aimed at evaluating the potential of isolated *M. paniculata* fibers as reinforcement in composite materials based on their physical and chemical properties. Water retting was employed to extract the bast fibers, followed by sodium hydroxide treatment to enhance their properties. The fibers underwent comprehensive characterization, including assessments of their physical, chemical, morphological, thermal, and mechanical properties using advanced analytical techniques such as scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), Fourier-transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and thermogravimetric analysis (TGA). Furthermore, the properties of these fibers were compared with those of other known botanical cellulosic fibers.

2 | Materials and Methods

2.1 | Materials

The bark of *M. paniculata* was collected from a healthy, matured, 9-year-old wild plant that was grown in the village of North Guthuma, Parshuran, Feni, Bangladesh. Initially, 2 kg of raw bark was harvested for the extraction process. Due to the plant's natural ability to shed and regenerate bark, this partial harvesting did not harm the tree. This is commonly known as sustainable strip harvesting. Sodium hydroxide (NaOH) granules, acetic acid (99.5% purity, used for neutralizing pH), ethanol (C₂H₅OH, 99% purity), toluene (C₆H₅CH₃, 98% purity), and sodium chlorite (NaClO₂) flakes were purchased from a local chemical store at Hatkhola, Dhaka, Bangladesh. All the chemicals were laboratory grade and used without further purification.

2.2 | Fiber Extraction Process

After collecting raw barks from the plant, they were chopped into small pieces at a length of approximately 6 in. They were then soaked in water at a material-to-liquor ratio (*M:L*) of 1:25. The retting process was carried out on a trial-and-error basis, and the optimum retting time was found to be 9 days. At this stage, the fibers could be easily separated by manual stripping and appeared in a meshy structural form. The extracted fibers were then dried in natural sunlight until fully dried. A portion of the dried fibers was treated with 5% (*w/w*) NaOH (*M:L* of 1:20) at 80°C for 30–40 min using a temperature-controlled hot plate to determine its effect on the fiber surface. After that, fibers were thoroughly washed with water and neutralized with

5% (*v/v*) acetic acid until the pH became neutral. The yield percentage was also calculated from the raw bark to the final extracted fibers. Figure 1 shows the overall fiber extraction process. For maintaining a proper sample size, visual inspection, measurement, and mechanical cutting are the most compatible. Cutting would ensure that the fiber lengths are maintained at an optimal value, thus preserving the high aspect ratio, which is crucial for efficient stress transfer. After that sieving method, the fiber sizes would be made uniform.

2.3 | Fiber Characterizations

2.3.1 | Surface Morphology and Chemical Composition

To examine the microstructure of both raw and alkali-treated fiber surfaces, the samples were analyzed using a Quanta 200 FEG SEM at the Institute of Materials Science, Kaunas University of Technology, Kaunas, Lithuania. Prior to imaging, the samples were properly conditioned and thoroughly dried to remove any residual moisture, ensuring accurate results during SEM analysis. The ImageJ software was used to determine the surface roughness of the treated and untreated fibers from the SEM image.

The elemental composition of the fibers was determined by a Bruker EDS (X-ray 4030) at the Institute of Materials Science, Kaunas University of Technology, Kaunas, Lithuania. The EDS generates 133 eV of energy and can identify chemical elements ranging from boron (5) to americium (95) at high X-ray photon detection rates.

The cellulose, hemicellulose, lignin, extractive, and ash contents of *M. paniculata* fibers were measured by TAPPI standard test procedures. Extractive percentage was determined using Soxhlet extraction with a solution of ethanol and toluene as the solvent following TAPPI T204 om-88 [28]. The fibers were then treated with a NaOH solution to determine the α -cellulose content in the holocellulose, which was generated using a NaClO₂ solution, as per the TAPPI T203 om-93 method [29]. The Klason method (TAPPI T211 om-83) was employed to determine the lignin content [30]. All compositional analyses were conducted in triplicate, and the mean values are reported.

2.3.2 | Physical Characterization

Prior to extraction, the raw bark samples were weighed, and the yield percentages of both raw and alkali-treated fibers were calculated by comparing their respective weights with the initial bark mass using the following equations (Equations (1) and (2)):

$$\text{Raw fiber yield (\%)} = \frac{MP_f}{MP_i} \times 100, \quad (1)$$

$$\text{Alkali-treated fiber yield (\%)} = \frac{MP_a}{MP_i} \times 100, \quad (2)$$

where MP_i is the initial mass of raw barks, MP_f is the mass of dried raw fibers, and MP_a is the mass of dried alkali-treated fibers.

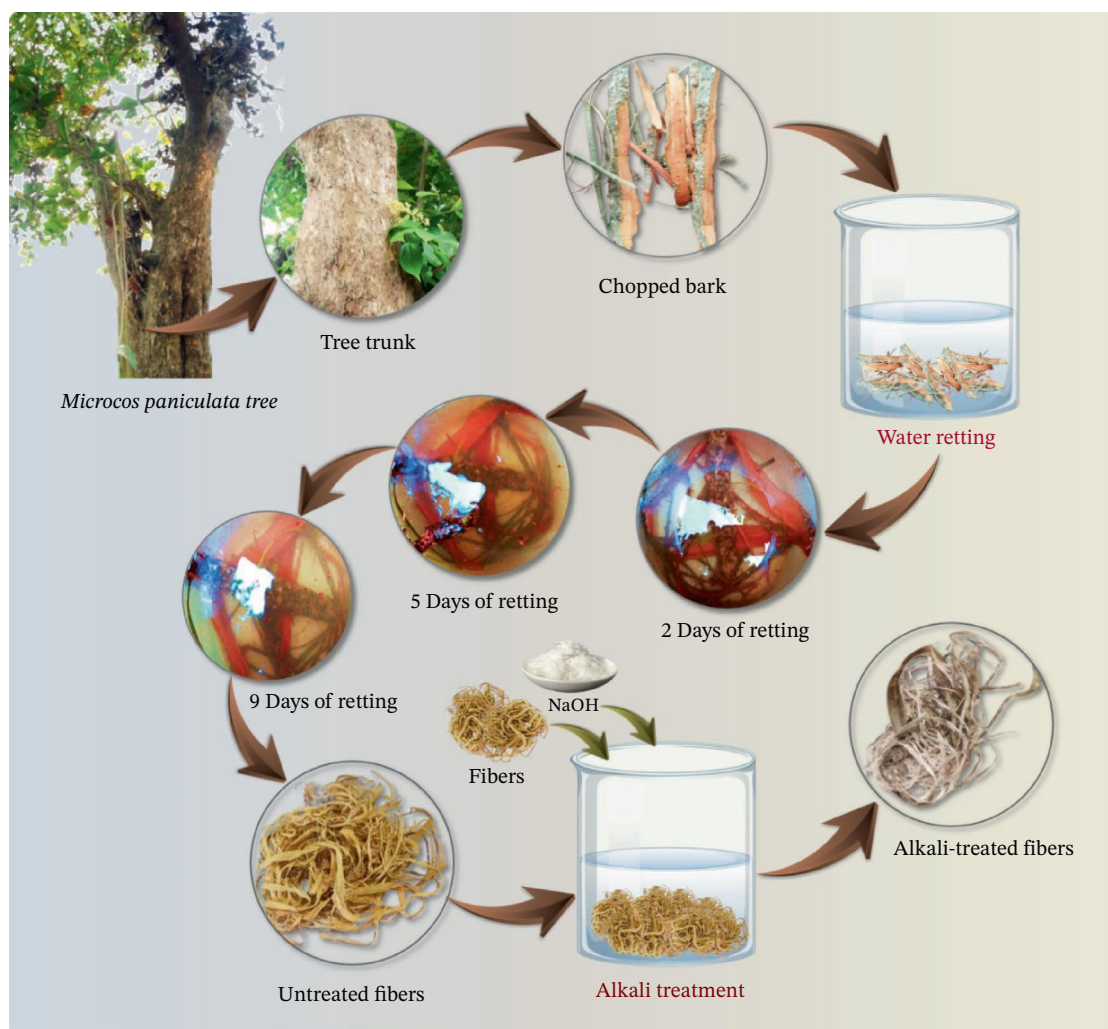


FIGURE 1 | Extraction process of *M. paniculata* fiber.

The moisture regain of *M. paniculata* fiber was measured by the weight loss method in accordance with ASTM D 2654 [31]. The fibers were conditioned at a room temperature of $27^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Moisture regain was calculated using Equation (3):

$$\text{Moisture regain (\%)} = \frac{W_w}{W_D} \times 100, \quad (3)$$

where W_w is the weight of water in the samples and W_D is the oven-dry weight of the samples.

The bulk density of the *M. paniculata* was measured using a pycnometer and the water submersed method in accordance with the ASTM D891-18 [32]. The density was determined using Equation (4):

$$\rho_{M.paniculata} = \frac{m_2 - m_1}{(m_3 - m_1) - (m_4 - m_2)} \rho_{\text{water}}, \quad (4)$$

where ρ_{water} represents the density of water (0.997 g/cm^3 at 25°C), $\rho_{M.paniculata}$ represents the density of fiber in grams per cubic centimeter (g/cm^3), m_1 denotes the mass of the empty pycnometer, m_2 denotes the mass of the pycnometer with fibers, m_3 denotes

the mass of the pycnometer filled with water, and m_4 denotes the mass of the pycnometer filled with both fiber and water.

2.3.3 | Structural and Chemical Characterization

The crystal structure of fiber samples was determined using a D8 Discover X-ray diffractometer (Bruker AXS GmbH) at the Institute of Materials Science, Kaunas University of Technology, Kaunas, Lithuania. The diffractometer operated at 40 kV and 40 mA with a $\text{Cu K}\alpha$ ($\lambda = 1.5418 \text{ \AA}$) radiation source and a parallel beam geometry with a 60-mm Göbel mirror. Diffraction patterns were captured using a fast-counting LynxEye detector with a 2.475° opening angle and a 6-mm slit size. Peak intensities were scanned from 10° to 70° (coupled 2θ - θ scans) with a 0.02° step size. The crystalline and amorphous regions were measured using the DIFFRAC.EVA Version 3.0 (Bruker) software, following the Nara and Komiya technique [33]. The degree of crystallinity was computed by dividing the crystalline area (as determined by the difference between the total and amorphous areas) by the diffractogram's total area.

A FTIR spectrometer (VERTEX 70) with ATR (attenuated total reflectance) from Bruker, Germany, was used at the Institute of Materials Science, Kaunas University of Technology, Kaunas,

Lithuania, to analyze specific functional groups as well as the chemical structure of fibers and nonwovens. They also assess both the presence and lack of functional groups prior to and during chemical treatments. The FTIR-ATR spectrometer can record spectra in the 500–4000 cm^{-1} range at a spatial resolution of 1 cm^{-1} .

2.3.4 | Mechanical and Thermal Characterization

The tenacity of the fiber bundles was measured using a Stelometer in accordance with ASTM D1445 [34]. The *M. paniculata* fibers were hand-sorted and placed between two small clamps with gauge lengths of 3.2 mm. Tension was applied to the fiber clamp at the top of the pendulum until the fibers ruptured. The technique provides data on both the breaking strength and extensibility at break. Figure 2 shows the visual representation of the fiber setup for this test. The tenacity of the fibers was measured using Equation (5). The sample mass was determined using a JWS-A mini electric pocket scale (Yongkang, China):

$$\text{Tenacity} = \frac{\text{breaking force (kg)} \times 15}{\text{sample mass (mg)}}. \quad (5)$$

The flammability and burning behavior of *M. paniculata* fibers were evaluated according to ISO 15025. According to this method, a flame was applied to the fiber sample for 10 s to assess its tendency to ignite and burn. The behavior of fibers under flame, odor while burning, and the residue after burning was observed from this test.

The thermal behavior of the *M. paniculata* fiber was analyzed using a simultaneous thermal analyzer (STA) L82 from LINSEIS, capable of recording both TGA and differential scanning calorimetry (DSC) data concurrently. Approximately 5 mg

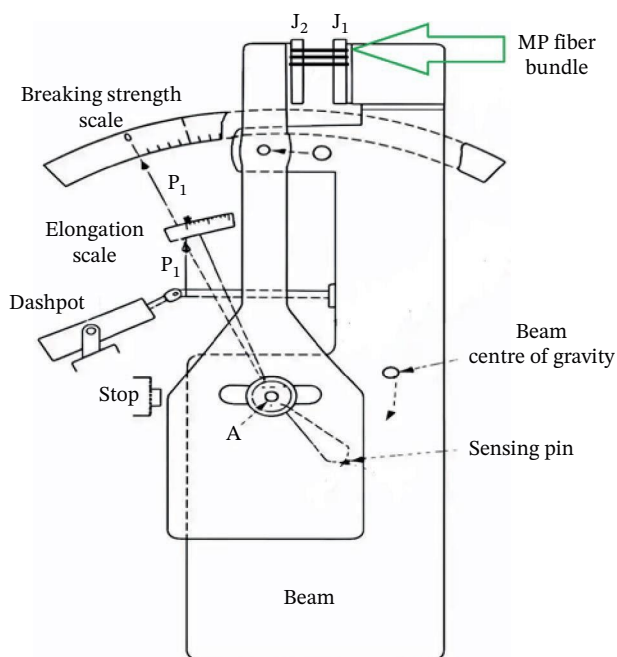


FIGURE 2 | Visual representation of the Stelometer setup.

of dried fiber sample was placed in an alumina crucible and heated from 30°C to 1000°C at a constant heating rate of 10°C/min under a nitrogen atmosphere. The nitrogen flow rate was maintained at 100 mL/min throughout the experiment. The TGA curve recorded the mass loss behavior of the sample, while the DSC curve provided information on heat flow associated with thermal transitions and decomposition events.

3 | Results and Discussion

3.1 | Surface Morphology Analysis by SEM

Surface morphology of the untreated and alkali-treated fibers was analyzed by the SEM micrographs as presented in Figures 3a, 3b, 3c, and 3d. Figure 3a reveals that the individual fibers were attached together by lignin as they formed a meshy structure. At 250× magnification, two microfibrils were observed with diameters of 12.53 and 10.82 μm , indicating high fiber fineness comparable to that of other textile fibers. Figure 3b,c illustrates the surface morphology of untreated and alkali-treated fibers, respectively, at 1000× magnification. The untreated fiber surface appears relatively smooth due to the presence of natural impurities such as lignin, waxes, fatty acids, and surface protrusions. In contrast, the alkali-treated fiber exhibits a rougher surface, confirming the effective removal of these impurities. This increased roughness is further highlighted in Figure 3f, which appears noticeably rougher than the untreated fiber surface shown in Figure 3e.

Additionally, the high-magnification SEM image at 4000× in Figure 3d provides a closer view of the alkali-treated fiber, revealing a more irregular and textured surface, indicative of enhanced surface roughness after treatment. Alkali treatment significantly modifies the surface morphology of natural lignocellulosic fibers. In *M. paniculata* fibers, this results in increased surface roughness, porosity, and exposure of microfibrils and cellular structures with small voids. These surface characteristics are useful to enhance the mechanical interlocking and interfacial bonding between the fiber and polymer matrix. The presence of surface fissures and voids may contribute to a larger effective contact area, facilitating better adhesion. Each fiber unit cell is composed of tiny cellulose particles embedded within a matrix of lignin and hemicellulose, which might support the structural integrity and binding potential of the fiber [35–38]. Therefore, this rough microstructure plays a crucial role in locking the polymer matrix and ensuring strong fiber–matrix bonding, making the treated fibers highly suitable for use in green composite applications.

3.2 | Elemental and Chemical Composition of the MP Fibers

EDS is a technique used to determine the elemental composition of a material by detecting X-rays emitted when a sample is bombarded with high-energy electrons. It is commonly employed in combination with SEM. In this study, EDS analysis was conducted on both untreated and alkali-treated *M. paniculata* fibers to assess their elemental composition, as shown in Figure 4a. The figure displays the EDS spectra along with

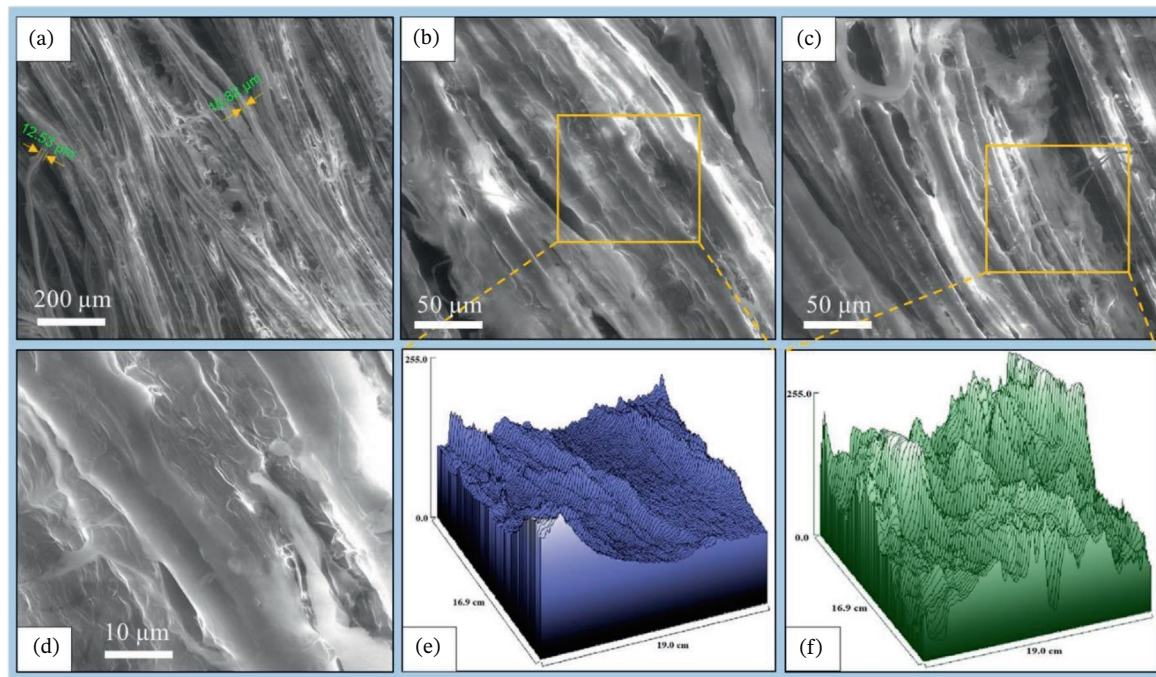


FIGURE 3 | SEM micrographs of (a) untreated fibers at 250 \times magnification, (b) untreated fibers at 1000 \times magnification, (c) alkali-treated fibers at 1000 \times magnification, and (d) alkali-treated fibers at 4000 \times magnification; surface roughness of (e) untreated fibers and (f) alkali-treated fibers.

the weight (%) and atomic (%) proportions of the detected elements. It is important to note that the intensity of EDS signals depends on several factors, such as atomic number, absorption, and fluorescence, and does not directly correlate with the exact weight or atomic percentages of elements. For instance, the peak intensities of light elements like carbon (C) and oxygen (O) may not reflect their actual abundance when mixed with heavier elements. Therefore, the EDS spectrum primarily serves to identify the types of elements present rather than to quantify them precisely.

As shown in Figure 4, a high content of C and O suggests a significant presence of cellulose in the fiber. In both untreated and treated samples, some minor elements such as calcium (Ca), magnesium (Mg), copper (Cu), aluminum (Al), and sulfur (S) were also detected negligibly. These elements likely originate from natural impurities such as oils, waxes, and pectins that form part of the primary cell wall structure of raw fibers. The 5% alkali treatment effectively reduced these impurities, which was confirmed by the EDS results. Notably, the C content increased by approximately 6% in the alkali-treated fibers. This may be attributed to the removal of inorganic compounds and the re-configuration of polymer chains to accommodate hydroxyl ions, resulting in a relative increase in C concentration. Overall, *M. paniculata* fibers were primarily composed of C and O, as evidenced by the characteristic peaks for cellulose, along with minor peaks for S, Al, and Ca.

Chemical composition of natural fibers can vary significantly depending on the plant species, the part of the plant used, and various external factors such as climate, soil conditions, plant maturity, extraction methods, and geographical origin. In this study, *M. paniculata* fibers were found to contain 55.13% α -cellulose, 19.65% hemicellulose, 20.19% lignin, and 5.1% extractives.

Figure 4b presents a pie chart showing the proportion of these components.

After alkali treatment, the composition of the fibers changed notably, with α -cellulose increasing to 63.25%, hemicellulose decreasing to 17.29%, lignin to 16.18%, and extractives to 2.11%, as illustrated in Figure 4c. This enhancement of cellulose content is attributed to the removal of noncellulosic components such as hemicellulose, lignin, pectins, waxes, and other impurities by NaOH. While cellulose is crystalline and resistant to mild alkali, noncellulosic components are typically amorphous and more easily dissolved [39]. Alkali treatment breaks ester and ether bonds in hemicellulose and lignin, effectively cleaning the fiber structure and increasing the relative cellulose content [40]. These results align with previous findings and explain the compositional changes observed. Table 1 presents a comparative overview of the chemical composition of *M. paniculata* fiber alongside values reported for other natural fibers.

The alkali-treated *M. paniculata* fiber contains approximately 64% cellulose, which is slightly lower than that of flax (61.4%–71%), hemp (68%–72%), and ramie (72%–76%) but comparable to *Cissus populnea* (61.8%) and jute (61%). However, it has a higher cellulose content than fibers such as *Triumfetta cordifolia* (38.8%), kenaf (53.14%), and *Carica papaya* (58.71%). An increased cellulose content on the fiber surface enhances mechanical strength, making the fibers more suitable for composite applications. Cellulose contributes significantly to fiber strength through hydrogen bonding and other molecular interactions, meaning that higher cellulose content typically results in greater strength and stability [36]. These characteristics suggest that *M. paniculata* fibers are promising candidates for reinforcing polymer composites.

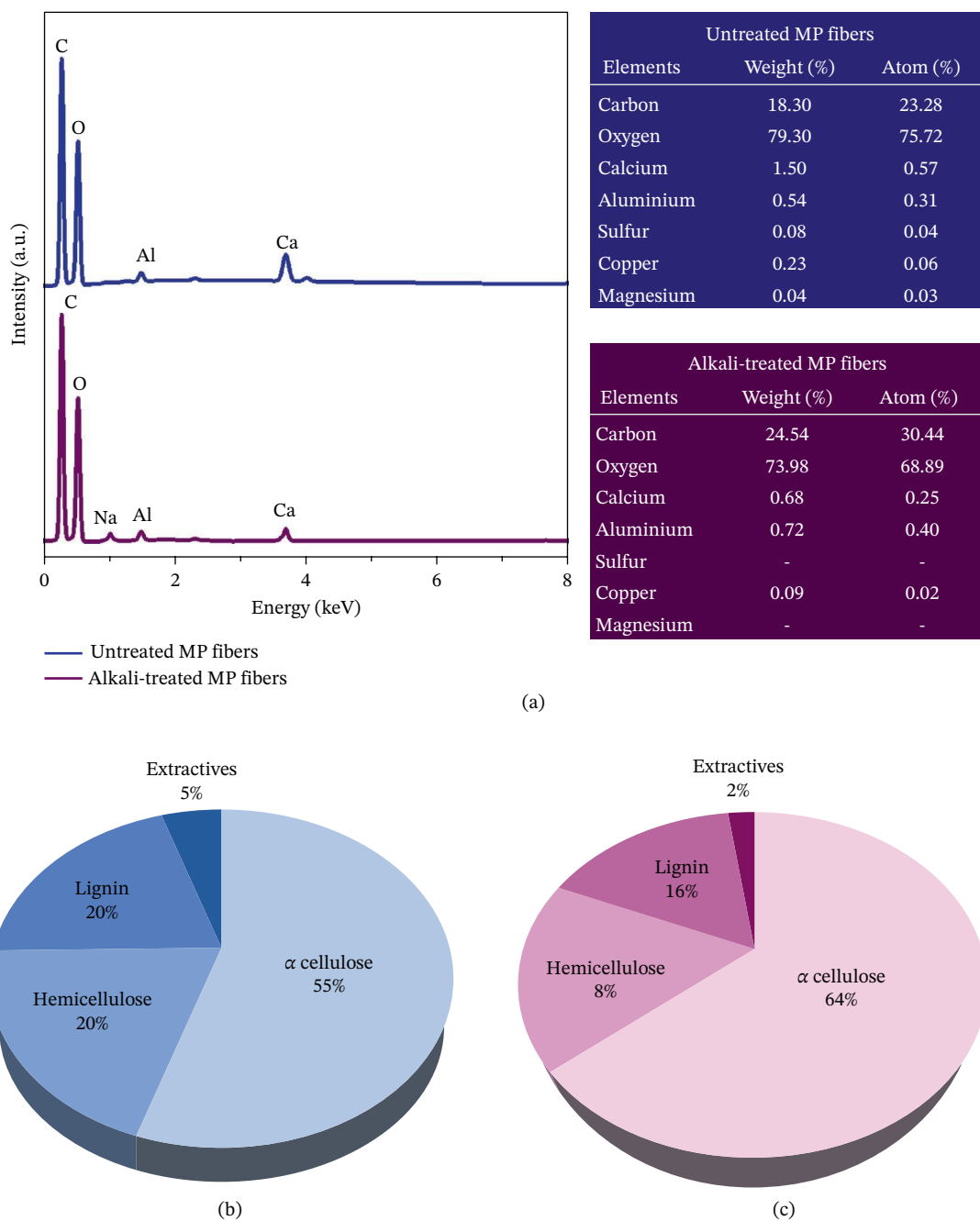


FIGURE 4 | (a) EDS spectra and elemental composition of untreated and alkali-treated MP fibers, (b) chemical composition of untreated MP fibers, and (c) chemical composition of alkali-treated MP fibers.

3.3 | Physical Properties of the MP Fibers

The percentage yield in textile fiber extraction typically ranges from 2.5% to 55%, depending on the fiber type, plant variety, and extraction method. In this study, the yield for raw and alkali-treated *M. paniculata* fibers was found to be 30% and 24.8%, respectively.

The moisture regain of a fiber significantly influences its structural and performance characteristics. Moisture management plays a critical role, as it affects key textile properties such as elasticity, friction, thickness, and tenacity. To minimize moisture loss during processing, controlling ambient humidity is

essential [53]. In composite applications, managing moisture regain is particularly important because natural fibers are inherently hydrophilic due to their cellulose content, which causes them to absorb environmental moisture. This water absorption can impact the fiber–matrix interface and overall composite performance [54]. Table 2 compares the moisture regain values of various natural fibers.

In this study, the average moisture regain values for raw and alkali-treated *M. paniculata* fibers were 12.61% and 13.58%, respectively, based on five individual fiber samples (2g each). The coefficients of variation (CVs%) were 5.13 and 3.75, respectively, indicating good consistency and statistical reliability of the results.

TABLE 1 | Chemical composition of *M. paniculata* fibers compared with the values of other natural fibers reported in recent studies.

Studied fibers	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Extractive (%)	Reference
<i>M. paniculata</i> (untreated)	55	20	20	5	This study
<i>M. paniculata</i> (alkali-treated)	64	18	16	2	This study
Palmyra seed sprouts	64	8	24	—	[41]
<i>Mikania micrantha</i>	56.42	21.42	15.78	—	[42]
Jute	64.4	12	11.8	—	[43]
Napier grass	45.66	33.67	20.6	—	[44]
Sugar cane straw	33.5	—	25.8	—	[45]
Flax	62–71	16–18	3–4.5	—	[46]
Hemp	67–75	16–18	3–5	—	[47]
Water hyacinth	67.5	6.6	22.3	—	[48]
Sisal	60–67	10–15	8–14	1.2–2	[49]
<i>Grewia ferruginea</i>	60.4–72.6	18.5	13.55	—	[11]
Banana	63–64	19	5	13	[50]
<i>Triumfetta cordifolia</i>	38.8	16.9	6	1	[51]
<i>Amaranthus viridis</i>	49.54	24.82	18.96	9.68	[52]

TABLE 2 | The moisture regain and density of *M. paniculata* fibers compared to other natural fibers.

Fiber name	Moisture regain (%)	Density (g/cm ³)	Reference
<i>M. paniculata</i> (untreated)	12.61 ± 0.65	1.28 ± 0.019	Present work
<i>M. paniculata</i> (alkali-treated)	13.58 ± 0.51	1.31 ± 0.026	Present work
Palmyra seed sprouts	14.38	1.33 ± 0.037	[41]
<i>Mikania micrantha</i>	9.17	—	[42]
Jute	12.5	1.45	[43]
Ramie	12	1.49 ± 0.11	[38, 55]
Sisal	11	1.45	[49, 56]
Banana	12.3	1.5	[50, 57]
Flax	2–6	1.5	[46]
Hemp	12	1.393	[47, 58]
<i>Amaranthus viridis</i>	22.49	1.43	[52]

The relatively high moisture regain of *M. paniculata* fibers suggests potential for use in clothing and home textiles. However, its moisture regain is also comparable to other natural fibers such as jute, ramie, and hemp, making it similarly suitable for composite applications. The increase in moisture regain following alkali treatment is attributed to the removal of surface impurities, which enhanced the hydrophilicity of the fiber.

The density of a fiber is determined by its molecular structure and the intermolecular communications within it. It is influenced by both the fiber's internal arrangement and its chemical composition [59]. Natural fibers such as wool and cotton exhibit a wide range of densities due to their complex cellular structures,

whereas synthetic fibers can be engineered to achieve specific densities through controlled polymerization processes. Physical properties such as tenacity, flexibility, and resilience are all affected by fiber density [60]. Higher density fibers typically offer greater strength and resistance to degradation, as their molecular structures are more tightly packed.

A pycnometer analysis of freshly isolated *M. paniculata* fibers revealed an average density of 1.288 g/cm³, with a CV% of 1.5 across five samples. In comparison, the alkali-treated *M. paniculata* fibers showed a slightly higher average density of 1.31 g/cm³, with a CV% of 1.99. This increase in density can be attributed to the removal of noncellulosic materials during alkali treatment, which retains the

denser, crystalline cellulose. As a result, the proportion of tightly packed cellulose structures increases, leading to a marginal rise in bulk density. The experimental data support this explanation. The density of *M. paniculata* fibers is lower than that of other natural fibers, such as cotton (1.5g/cm³), jute (1.45g/cm³), PSSF (1.33g/cm³), and flax (1.5g/cm³), as shown in Table 2 [41, 61, 62]. This suggests that *M. paniculata* fibers may be a suitable candidate for producing lightweight fiber-reinforced composites.

3.4 | Structural Properties Analysis by XRD

XRD patterns of untreated and alkali-treated *M. paniculata* fibers are presented in the aforementioned Figure 5a. Both the samples exhibit characteristic diffraction peaks at approximately $2\theta = 15.9^\circ$ and 22.6° , which correspond to the (110)/(110) and (200) crystallographic planes of Cellulose I, respectively. Both XRD peaks confirm the semicrystalline nature of cellulose in *M. paniculata* fibers, where the prominent and sharp (200) diffraction peak observed at approximately $2\theta \approx 22.6^\circ$ represents the well-oriented crystalline regions and a broader peak at approximately $2\theta \approx 15.9^\circ$ represents the amorphous regions of the fiber. The alkali-treated fiber exhibited a notably sharper and more intense (200) peak compared to the untreated fiber, suggesting an increase in crystallinity due to the removal of amorphous constituents such as lignin, hemicellulose, and pectin [37]. The untreated sample also showed minor peaks that disappeared after alkali treatment, indicating the elimination of some disordered or extraneous phases. The crystallinity index (CI) of the untreated *M. paniculata* fiber was calculated to be 63%, while the alkali-treated fiber showed an increased CI of 69%. This improvement can be attributed to the selective removal of amorphous components by NaOH, which hydrolyzes ester and ether linkages without disrupting the highly ordered cellulose crystalline regions [40]. Furthermore, the alkali treatment may cause the fiber to swell, promoting better alignment of cellulose chains and enhancing crystalline domain formation [39]. This structural reorganization not only increases crystallinity but also contributes to improved mechanical properties. Table 3 presents a comparative overview of the CI of *M. paniculata* fibers in relation to other natural fibers.

3.5 | Chemical Properties Analysis by FTIR

Figure 5b shows the FTIR spectrum of untreated and alkali-treated *M. paniculata* fibers. The characterization of this cellulosic fiber via FTIR revealed critical insights about the chemical composition and structural changes induced by alkali treatment. For untreated fibers, significant absorption peaks were identified, including broad peaks at approximately 3457 and 3350 cm⁻¹, which correspond to O–H stretching vibrations associated with hydroxyl functional groups [41]. Additionally, the peaks at around 2897 cm⁻¹ correlate with asymmetric C–H stretching, confirming the presence of methyl and methylene groups in cellulose [64]. The presence of a small peak at 1740 cm⁻¹ may correspond to the C=O bending of acetyl and ester groups in hemicellulose and lignin. The additional significant peak at 1602 cm⁻¹ is associated with aromatic compounds, and at 1456 cm⁻¹, it is related to the C–H bending of lignin [65]. The peak at 1039 cm⁻¹ is attributed to C–O stretching, and at 769 cm⁻¹, it relates to C–H bending modes, typical in the cellulose framework [66].

Upon alkali treatment, a marked alteration in the FTIR spectra was observed, evidenced by the shift in the broad peaks to 3473 and 3047 cm⁻¹. This indicates a modification in hydrogen bonding within the cellulose structure, which is suggestively related to a reduction in hydroxyl groups due to the removal of hemicellulose and lignin [67]. The prominent peak shift to 1687 cm⁻¹, slightly lower than the untreated peak at 1602 cm⁻¹, denotes a potential alteration in aromatic bond characteristics in posttreatment. The increased intensity of the 1456 cm⁻¹ peak following alkali treatment may indicate enhanced cellulose crystallinity, aligning with studies that highlight the effectiveness of alkali treatments in promoting crystalline structures in cellulose [68]. Furthermore, the prominent peak at 1024 cm⁻¹ observed after alkali treatment, shifted and reduced from 1039 cm⁻¹ of untreated fibers, implies a reduction in available hydroxyl groups due to alkali-induced solubilization of hemicellulose [67]. The emergence of the new peak at 850 cm⁻¹ might indicate alterations in the glycosidic linkages within the cellulose after treatment [41]. This change illustrates the dynamics of fiber modification, which

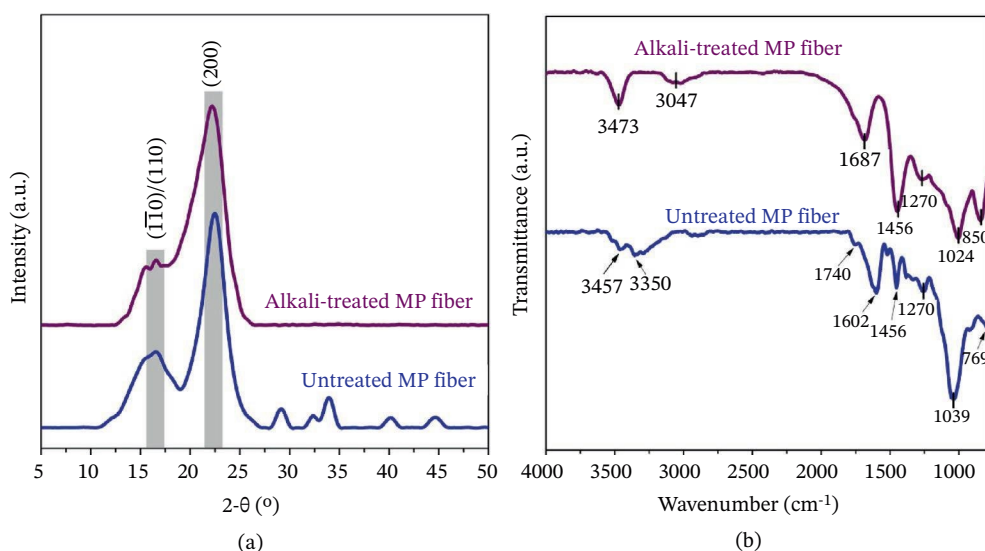


FIGURE 5 | (a) XRD and (b) FTIR spectra of untreated and alkali-treated MP fibers.

TABLE 3 | Crystallinity index, tenacity, and elongation of different natural fibers.

Fiber name	Crystallinity index (%)	Tenacity (g/tex)	Elongation (%)	Reference
<i>M. paniculata</i> (untreated)	63	34.85 ± 2.09	3.9 ± 0.079	This study
<i>M. paniculata</i> (alkali-treated)	69	38.59 ± 1.93	4.9 ± 0.065	This study
Palmyra seed sprouts	38	11.5	1.8	[41]
<i>Mikania micrantha</i>	72	36	1.8	[42]
Jute	71	20–30	1.8	[43]
Flax	70	27–40.9	1.8–3.5	[46]
Hemp	65	4.62	1.6	[47]
Water hyacinth	—	12.069	1.8	[48, 63]
Sisal	67	31.36	2–3	[49]
<i>Grewia ferruginea</i>	49	5.33 ± 0.66	3.6% ± 1.8%	[11]
Banana	39	22–38.5	1–6.54	[50]
<i>Amaranthus viridis</i>	40	10.23	1.35	[52]

enhances its structural properties for potential applications, aligning with discussions from various authors regarding the effects of chemical treatment on cellulose [69].

3.6 | Fiber Strength Analysis

The mechanical strength of textile fibers is one of their most critical properties, directly influencing their capacity to bear loads before failure, which is essential to consider when using fibers as reinforcement in composite materials. In this study, the Stelometer test was employed to assess the bundled fiber strength of *M. paniculata*. Five samples were measured, yielding an average tenacity of 34.85 g/tex with 3.9% elongation for raw fibers. In contrast, alkali-treated *M. paniculata* fibers exhibited a significantly improved tenacity of 38.59 g/tex alongside an elongation of 4.96%. The strength of fiber bundles was assessed through statistical estimation of the characteristic's dispersion. Untreated *M. paniculata* fibers had a standard deviation of roughly 2.09 g/tex and a CV of 6.0%; for elongation, the standard deviation and CV were 0.079 and 2.03%, demonstrating a moderate degree of heterogeneity typical of natural fibers. Conversely, the alkaline processing of the fibers resulted in reduced property variability, as evidenced by a standard deviation of 1.93 and 0.065 g/tex and a CV of 5.0% and 1.31% for fiber tenacity and elongation, which stemmed from the homogenization of fiber structure through the elimination of amorphous components.

Alkali treatment has a considerable impact on the tenacity of natural fibers by modifying their chemical composition and microstructure. Specifically, this treatment facilitates the removal of amorphous constituents such as hemicellulose, lignin, and surface impurities, resulting in an increase in the fiber's CI. As crystalline cellulose remains largely unaffected and constitutes a more substantial part of the fiber composition, the enhancement in crystallinity correlates with improved tenacity and stiffness [26, 70]. The mechanical strength of the reinforcing fiber

plays a vital role in determining the load capacity and stiffness of the composite material into which it is integrated. The effective transfer of stress from the matrix to the fiber is essential for maximizing performance. As a result, the enhancement of fiber strength through alkali treatment is likely to lead to improved overall mechanical properties, durability, and structural integrity of the resulting composites [26, 70, 71]. Thus, alkali-treated *M. paniculata* fibers present a promising avenue for enhancing composite properties during fabrication.

3.7 | Thermal Properties Analysis by Burn Test, TGA, and DSC

When *M. paniculata* fibers came into contact with flame, they ignited almost instantaneously, producing a vivid yellow flame indicative of a rapid combustion process. This burning generated a characteristic odor reminiscent of burning wood or leaves, a trait commonly associated with naturally occurring cellulose materials [72]. Following combustion, the fibers transformed into light-colored, fluffy grey ash, which was easily crushed between the fingers, suggesting its brittle nature. During the burning process, the smoke predominantly appeared white or grey, and significantly, the absence of noxious fumes indicated that no harmful byproducts were generated. These observations carry important implications for quality control in cellulose-based products, textile evaluation, forensic examinations, and authentication processes. Understanding the combustion characteristics of *M. paniculata* fibers can enhance product safety and inform the development of standards in the textile industry.

TGA is a technique used to evaluate the thermal stability of fibrous materials by analyzing their decomposition behavior under heat. This analysis is crucial for high-performance industrial applications, such as composites, where fibers must withstand elevated temperatures during manufacturing and end-use conditions. Under inert atmospheres, lignocellulosic

fibers typically degrade in three stages: drying (below 230°C), pyrolysis (230°C–425°C), and carbonization (over 500°C) [73].

As shown in Figure 6, the initial weight loss of approximately 6.86% occurred between room temperature and ~150°C–226°C, corresponding to moisture evaporation and the release of low molecular weight compounds [74]. The onset of thermal degradation began with the breakdown of hemicellulose and glycosidic linkages in amorphous cellulose. Although cellulose degradation can start around 250°C, it becomes more pronounced at higher temperatures due to its long-chain, semicrystalline structure. Active pyrolysis occurred between ~250°C and ~500°C, during which significant mass loss was observed. Hemicellulose, being amorphous, degrades earlier, whereas cellulose, with its crystalline microfibrillar structure, exhibits higher thermal resistance, maintaining relative stability up to around 545°C [75]. Lignin degradation occurred over a broad temperature range, starting around 498°C and continuing beyond 600°C. Due to its cross-linked aromatic structure and charring ability, lignin provides greater thermal stability compared to cellulose and hemicellulose. The final stage of decomposition, occurring between 400°C and 800°C, involved the oxidation of carbonaceous residues and other thermally stable components [76, 77]. The final residue (~25%) is considered char, representing the carbonaceous material and remaining nonvolatile fractions after pyrolysis. Table 4 compares the thermal degradation behavior of *M. paniculata* fibers with other natural fibers, highlighting similar decomposition patterns. These findings support the potential of *M. paniculata* as a viable reinforcement material for composite manufacturing.

While the fibers were heated, DSC was used to observe thermal transitions and reactions, particularly phase changes and decomposition events [79]. An initial endothermic peak was observed below 100°C, which corresponds to moisture evaporation, a typical feature of natural lignocellulosic fibers. This event indicates the physical release of absorbed and bound water. The DSC curve remained active from approximately 80°C to 450°C, showing a broad endothermic behavior, likely related to the thermal volatilization of hemicellulose and partial depolymerization of amorphous cellulose. These reactions consume heat and thus reduce the heat flow, indicating energy absorption associated

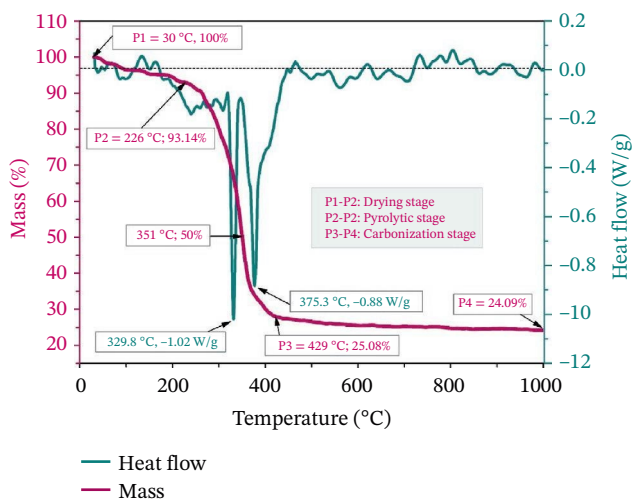


FIGURE 6 | TGA and DSC curve of *M. paniculata* fiber.

TABLE 4 | Comparison of different thermal weight loss conditions of cellulosic fibers.

Parameters	Fiber name							
	<i>M. paniculata</i>	PSSF	<i>Mikania micarnta</i>	<i>Prosopis juliflora</i>	<i>Azadirachta indica</i>	<i>Phaseolus vulgaris</i>	<i>Heteropogon contortus</i>	<i>Grewia ferruginea</i>
Primary weight loss condition (°C)	> 106	100	103	66	97	< 150	90	< 150
Primary weight loss (%)	6.86	7.27	7.44	8.12	9	2.43	4.04	18.25
Ultimate weight loss condition (°C)	993	474	477	351	788.6	328	337	700
Ultimate weight loss (%)	75.91	76.89	80.51	89	82.2	92.3	87.26	95.7
Reference	This work	[41]	[42]	[78]	[80]	[81]	[82]	[11]

with chemical bond breakdown and structural transitions. A noticeable exothermic event was initiated near 450°C, peaking around 1000°C, as evident from the pronounced rise in heat flow. This sharp exothermic peak may be attributed to oxidative degradation or carbonization, indicating the formation of char and possible aromatic compound breakdown. The large heat output suggests significant chemical changes—either decomposition of lignin or combustion of residual carbonaceous material, even under controlled atmosphere conditions. The steady decline in thermal activity beyond this point implies that the major thermal transformations were largely completed, and no further substantial reactions occurred at higher temperatures.

4 | Limitation of the Study

This study of MP fiber had several constraints. This particular study was limited to fibers sourced from plants collected within a particular geographical area and temporal window, thereby excluding potential variations arising from differences in collection periods or locations. Furthermore, the extraction and processing methodologies employed were not subject to optimization, which could have influenced fiber quality due to the severity of the retting and mechanical procedures. The research also omitted a detailed characterization of the fibers' chemical composition, as well as an evaluation of their physical properties and any existing defects. Consequently, to augment the comprehension and application of MP fiber, additional research is warranted. Such investigations should assess the impact of seasonal and regional variables on fiber characteristics, refine extraction and processing techniques to enhance quality and consistency, and examine the fibers' durability, biodegradability, and functional alterations.

5 | Conclusion

The extraction and characterization of novel natural cellulosic fibers from *M. paniculata* bark have demonstrated promising potential as reinforcement materials in polymer composites. Lignocellulosic fibers were isolated using chemical extraction methods and thoroughly evaluated for their physicochemical, mechanical, morphological, and thermal properties. The extracted fibers exhibited a high cellulose content and a CI of 63%, indicating a semicrystalline structure favorable for composite reinforcement. Alkali treatment notably enhanced the fiber quality by improving several critical properties. The treated fibers showed increased cellulose content, higher CI (rising to 69%), improved tenacity, and slightly higher density due to the removal of noncellulosic components such as lignin, hemicellulose, waxes, and surface impurities. This treatment also increased surface roughness, promoting better fiber–matrix adhesion in composite applications. Compared to traditional natural fibers such as cotton, jute, kenaf, and sisal, *M. paniculata* fibers, particularly after alkali treatment, exhibited superior performance when embedded in polymer matrices. Thermal analysis confirmed that the fibers can withstand temperatures up to ~240°C, making them suitable for processing with polymer matrices such as polyolefins and thermosets. Overall, the results suggest that *M. paniculata* fibers possess a favorable combination of strength, thermal stability, and composition,

making them suitable for use in green polymer composites, bio-based materials, lignocellulosic composites, cellulosic nanocomposites, and technical textile applications. It is clear that this fiber has comparable strength and thermal stability to ordinary natural fibers, making it appropriate for semistructural and insulating composite materials. Furthermore, the extraction method proposed in this paper supports sustainability by using low amounts of chemicals.

Author Contributions

Afsar Uddin: conceptualization, investigation, methodology, and resources. Shahidul Islam: conceptualization, investigation, methodology, and resources. Fahmida-E-Karim: conceptualization, data curation, formal analysis, and writing—original draft. Md. Redwanul Islam: conceptualization, data curation, formal analysis, and writing—original draft. K.Z.M. Abdul Motaleb: conceptualization, supervision, validation, visualization, and writing—review and editing. Arkid Chakma: investigation, methodology, and resources. Shahib Sharar Hossain: investigation, methodology, and resources. Md. Safwan Hossain: investigation, methodology, and resources. Md. Zahid Hasan: resources, data curation, formal analysis, and visualization. Asta Tamulevičienė: data curation, formal analysis, investigation, methodology, and writing—review and editing. Brigita Abakevičienė: data curation, formal analysis, investigation, methodology, and writing—review and editing.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding authors. The data are not publicly available due to privacy or ethical restrictions.

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