

Valorization of *Moringa Oleifera* Pod Residues into Functional Lignocellulosic Fiber: A Chemical and Mechanical Performance Study

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ABSTRACT

This study explores the potential of *Moringa oleifera* pod fiber as a sustainable alternative to conventional fibers for reinforcement in various polymer matrix composites. The fibers were extracted and alkali treated to evaluate their suitability for textile and bio-based composite applications. The surface morphology, crystallinity, physical, mechanical, and thermal properties of the fibers were characterized. The fibers exhibited a favorable chemical composition, comprising 54.02% cellulose, 16.57% hemicellulose, and 17.81% lignin. The crystalline index (CI) was determined as 43.75%. The average diameter of the fibers tuft was $116.92 \pm 2.65 \mu\text{m}$, and the fiber density was 1.25 g/cm^3 . Additionally, the fibers dissolved in 70% sulfuric acid. Moreover, the fiber exhibited a maximum peak force of 2.02 N at an extension of approximately 15%. Furthermore, thermogravimetric analysis (TGA) indicated that the fiber was thermally stable up to about 250°C. A significant mass loss of 92.91% was observed between 85.1°C and 697.2°C, leaving a char residue of 7.09%. The findings suggest that *Moringa oleifera* pod fiber is a promising reinforcement material, comparable to other established natural fibers, for sustainable applications such as bio-composites, fiber blends, paper products, and eco-friendly packaging.

Keywords: Extraction, *Moringa Oleifera*, Lignocellulosic fiber, Chemical treatment

1. INTRODUCTION

The growing global demand has intensified research into natural fibers, particularly those derived from lignocellulosic biomass, as sustainable and environmentally friendly alternatives to traditional synthetic materials [1]. Natural plant fibers such as flax, jute, coir, banana, palm, and hemp have gained a considerable attention due to their abundance, environmental sustainability, biodegradability, renewability, low density, and cost-effectiveness, in contrast to synthetic fibers such as polyester, glass, carbon, and aramid, which are generally non-biodegradable and relatively expensive [2]. Consequently, numerous studies have focused on the extraction and characterization of lignocellulosic fibers from various plant sources, including bark, twigs, leaves, stems, and fruit bunches, to evaluate their potential applications in the textile, automotive, biomedical, and construction sectors [3].

Over the past decade, *Moringa oleifera* (MO) has attracted considerable research interest due to its

medicinal, agricultural, environmental, and industrial potential [4]. It is commonly known as the “horseradish tree” or “drumstick tree” which is widely cultivated and rapidly growing. It is native to Pakistan, India, Bangladesh and Afghanistan as its remarkable drought tolerance enables growth in arid regions and nutrient-deficient soils [5]. The *Moringa* plant is also referred to as the “Miracle tree” due to its rich medicinal properties and nutritional benefits [6]. Furthermore, *Moringa* plays a significant role in addressing global challenges related to health, and environmental sustainability through its applications in food and animal feed production, water purification, and various industrial sectors [7]. In addition, different parts of MO, including the leaves, seeds, bark, roots, sap, pods, and flowers, are rich in essential vitamins, minerals, and antioxidants, making them a valuable source of nutrition, particularly in regions affected by food insecurity [8]. The leaves of MO (MOL) are a nutrient dense plant material with considerable health promoting potential. Owing to their rich bioactive composition, MOL exhibits strong antibacterial and antioxidant activities and is

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widely utilized in functional foods and nutraceutical formulations. [9]. Several research utilizing MOL like natural feed supplement in poultry diets [10], medicinal uses [11] etc. Again, Moringa seeds are a rich source of nutrients, similar to other parts of the plant. They are especially thought to be a good source of protein and high-quality oil. The cosmetics industry extensively utilizes MO seed oil, which is also used for biodiesel production and as an edible cooking oil [12]. Traditionally, MO seed powder has been used as a natural coagulant for pollutant flocculation and drinking water purification [13].

Moreover, *Moringa oleifera* pods (MOP) are round or triangular in shape, and the seeds are enclosed in a covering that may be readily removed. The outer hard and rough pericarp of the fruit is typically discarded as waste after seed extraction. This material is rich in fiber and is often treated as agricultural waste by commercial industries once its economic value has been utilized [14]. The mature MOP used for oil extraction produce a large amount of fibrous residue that is unsuitable for human consumption [14]. Discarding this waste not only increases environmental burdens but also represents a loss of potentially valuable lignocellulosic material. Therefore, MOP fiber can be valorized by repurposing it as a sustainable reinforcing material or raw fiber. Converting this inedible agricultural waste into useful resources supports both waste valorization

and circular economy principles. The fibrous fraction can be investigated for use in bio-composites, eco-friendly textiles, paper goods, or biodegradable materials rather than being thrown away [15]. Biopolymer composites reinforced with MO fibers and a PLA matrix have been developed for potential applications in the automotive, aerospace, and packaging industries [16]. In addition, fly ash-modified MO fiber-reinforced epoxy composites have been fabricated and evaluated for sustainable engineering applications [17]. These applications are expected to support sustainable industrial development and improve overall resource efficiency. The global market for Moringa-based products reached USD 7.1 billion in 2021 and is expected to expand at a compound annual growth rate (CAGR) of 8.9 % between 2023 and 2032 [18]. If this underutilized biomass is turned into useful products by developing MOP fiber, it could create significant economic opportunities worldwide. Several studies have investigated different parts of the Moringa tree for various applications; however, the characterization of its fibers after chemical treatment remains limited in the literature. In order to determine the untapped potential of *Moringa oleifera* pod fiber as a novel lignocellulosic material for reinforcing composite materials and other cutting-edge sustainable applications, this study specifically aims to characterize the morphological, chemical, mechanical, and thermal properties of the fiber.

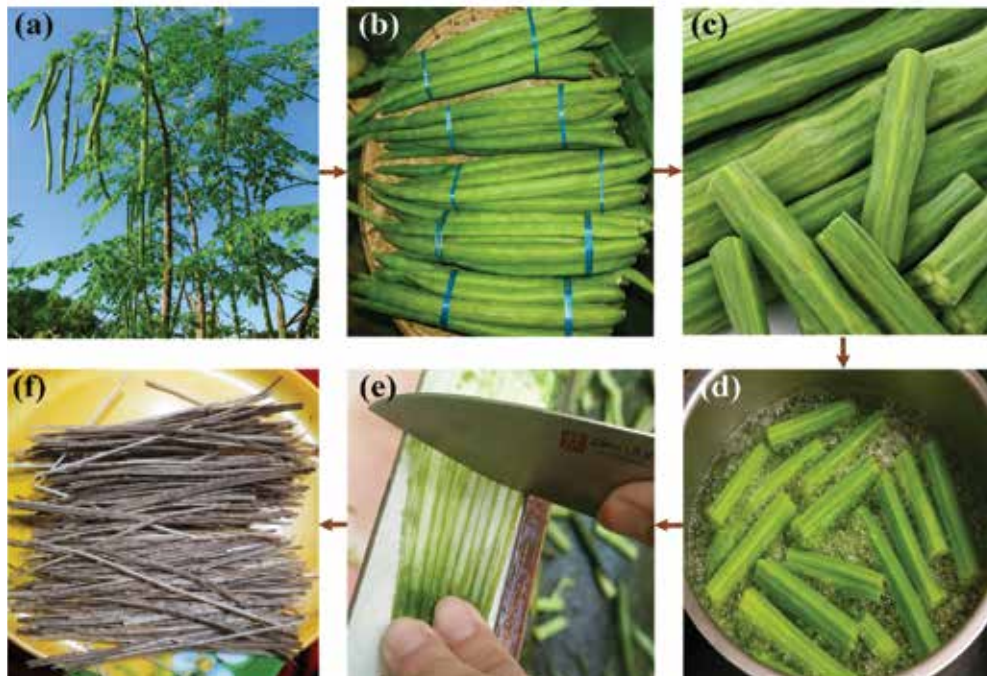


Fig. 1: Schematic representation of the MOP fiber extraction process: (a) *Moringa oleifera* tree; (b) collection of mature pods; (c) chopping of pods; (d) boiling of pods; (e) removal of non-fibrous material; and (f) extracted MOP fiber

2. EXPERIMENTAL

2.1 Materials

Mature Moringa pods were collected from a local market in Trisal Upazila, Mymensingh, Bangladesh. To chemically modify the fibers, sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), wetting agent, and sodium metasilicate (Na_2SiO_3) were purchased from the local market.

2.2 Fiber Extraction and Chemical Treatment

The collected mature Moringa pods were cut into sections approximately 10 cm in length. To make fiber separation easier, the samples were boiled in water roughly at 90°C for 60 min. Following boiling, the samples were allowed to cool for 2 h at room temperature. All the processes are shown in Fig. 1. After the softened plant material was manually removed, the remaining fibers were compressed to eliminate non-fibrous residue.

The fibers were subsequently cleaned and sun-dried until they reached a consistent moisture content. Chemical treatment of the extracted fibers (60 g) involved a 90 min immersion in a solution of NaOH (4.0%), H_2O_2 -35% (4.0%), Na_2SiO_3 (1 g/L), and a wetting agent (1 g/L) at 90°C with 1:20 (M:L). The process concluded with a distilled water wash and fan assisted air drying to prepare the samples for further analysis. The chemical treatment of MOP fiber is shown in Fig. 2.

2.3 Morphological Analysis

The surface morphology of the extracted fibers was analyzed using a scanning electron microscope (SEM). Fiber samples measuring approximately 10 mm were randomly selected for examination. The fibers were mounted on aluminum stubs using double-sided carbon adhesive tape, and sputter-coated with a thin layer of gold to enhance electrical conductivity. The fiber morphology, surface characteristics, and fibrillar structure were examined using SEM at different magnifications. Fiber diameter was measured using ImageJ 1.80 software.

2.4 Fourier Transform Infrared Spectroscopy (FTIR)

An FT/IR-4700 type FTIR spectrometer with an ATR PRO ONE attachment was used to examine the chemical constituents of the MOP fibers. To remove residual moisture, approximately 5 g of fibers were chopped, washed, and oven-dried at 60°C for 4 h. The dried fibers were then placed directly on the ATR crystal for analysis. FTIR spectra were recorded in the range of $4000\text{--}500\text{ cm}^{-1}$, and the characteristic peaks corresponding to functional groups of cellulose, hemicellulose, lignin, and pectin were identified. Every spectrum was captured at transmittance (% T) in relation to wavenumber (cm^{-1}).

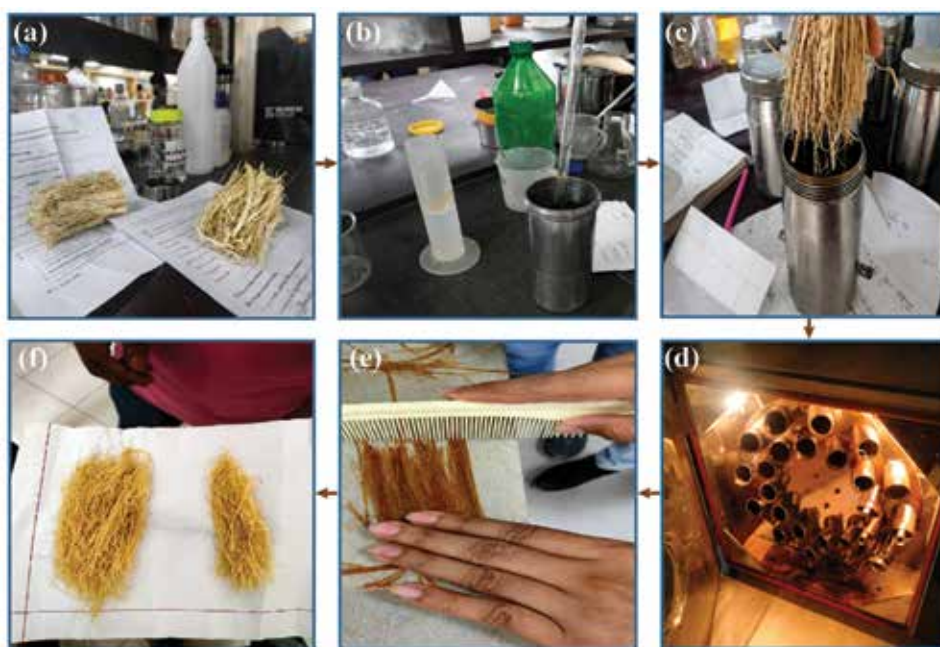


Fig. 2: Sequential steps involved in the chemical treatment of MOP fibers: (a) extracted fibers; (b) preparation of chemical solution; (c) fibers immersion; (d) chemical treatment; (e) combing of the fibers; and (f) treated MOP fiber

2.5 X-ray Diffraction (XRD)

The crystalline structure of the MOP fibers was analyzed using a PANalytical X'Pert PRO X-ray diffractometer equipped with Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$). Approximately 4 g of fibers were ground into a fine powder, oven-dried at 110°C for 15 min, and the resulting dried sample (3.2 g) was stored in a sealed container prior to analysis to prevent moisture absorption. XRD patterns were recorded in the 2θ range of 10°–60° with an operating voltage of 40 kV, current of 30 mA, a step size of 0.02°, and a scanning speed of 2°/min. The crystallinity index (CI) of the fibers was calculated using the Eqn. (1):

$$\text{CI (\%)} = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

Where, I_{002} is the maximum intensity of the (002) lattice reflection at around $2\theta = 22^\circ$ and I_{am} is the intensity of the amorphous background at around $2\theta = 18^\circ$.

2.6 Chemical Composition and Solubility Analysis

The TAPPI T429 cm-23 and T249 cm-85, T 236 cm-85 procedures were used to determine the percentages of cellulose, hemicellulose, and lignin. Additionally, determining the solubility of MOP fiber and to investigate the chemical resistance, sulfuric acid solutions of varying concentrations (50%, 60%, and 70%) are used.

2.7 Moisture Content and Moisture Regain Analysis

The oven drying method (ASTM D 2495–07 (2019)) was used to determine the weight of dried samples. The moisture content and moisture regain were evaluated as described in Eqn. (2) and (3).

$$\text{Moisture Content \%} = \frac{M_1 - M_2}{M_1} \times 100 \quad (2)$$

$$\text{Moisture Regain \%} = \frac{M_1 - M_2}{M_2} \times 100 \quad (3)$$

Where, M_1 is the weight of fiber samples at 25 °C and M_2 is the weight of the dried samples at 105°C for 4 h.

2.8 Fiber Density

The fiber density was measured using a gas pycnometer according to Eqn. (4). A precision balance was used to calculate the mass, and a pycnometer (Ultracyc 5000, Anton Paar) was used to measure the volume.

$$\rho = \frac{m}{V} \times 100 \quad (4)$$

Here, ρ represents the fiber density, m is the mass of the fiber bundle (g), and V is its volume (cm^3).

2.9 Mechanical Properties Analysis

A universal testing machine (UTM) equipped with a 10 N load cell was used to evaluate the tensile properties of the extracted MOP fibers in accordance with ASTM D3822-14. Prior to testing, approximately 5 g of fibers were conditioned for 24 h at $25 \pm 2^\circ\text{C}$ and $65 \pm 2\%$ relative humidity. To ensure proper alignment and prevent slippage during testing, individual fibers were mounted on paper frames. The UTM was operated with a gauge length of 20 mm and a crosshead speed of 10 mm/min.

2.10 Thermogravimetric Analysis (TGA)

A thermogravimetric analyzer (TGA) was used to evaluate the thermal stability of MOP fibers under a nitrogen atmosphere to prevent oxidation. The fiber samples were cut into small pieces, thoroughly washed, and oven-dried at 60°C prior to analysis to eliminate residual moisture. For examination, around 5 g of the dried fibers were put in a platinum sample pan. The TGA test was carried out with a steady nitrogen flow of 50 mL/min from ambient temperature to 600°C at a heating rate of 10 °C/min. To ascertain the thermal stability, stages of disintegration, residual char content of the fibers, and the weight loss profile were monitored during the heating process.

3. RESULTS AND DISCUSSION

3.1 Morphological Analysis

Morphology and microstructural analyses of MOP fibers were studied using SEM analysis at 2000 $\times$, 5000 $\times$, and 10,000 $\times$ magnifications which are shown in Fig. 3. Fig. 3(a) exhibits the lash fiber bundles compacted by several elementary fibers and exhibits relatively good structural continuity and few void contents. Fig. 3(b) demonstrates the longitudinal grooves and surface irregularities. The morphology of the intact bundles shows that the extraction process kept the integrity size of the fibers, which is an advantage for load bearing and reinforcement [19]. In Fig. 3(c), more distinct boundaries of individual fibrils are detectable along with surface irregularity and partial separation lines. These characteristics indicate the residual existence of non-cellulosic materials, such as lignin and hemicellulose that are typically present in untreated natural fibers. These whisker surface properties affect the fiber matrix interaction and are enhanced by chemical pretreatment.

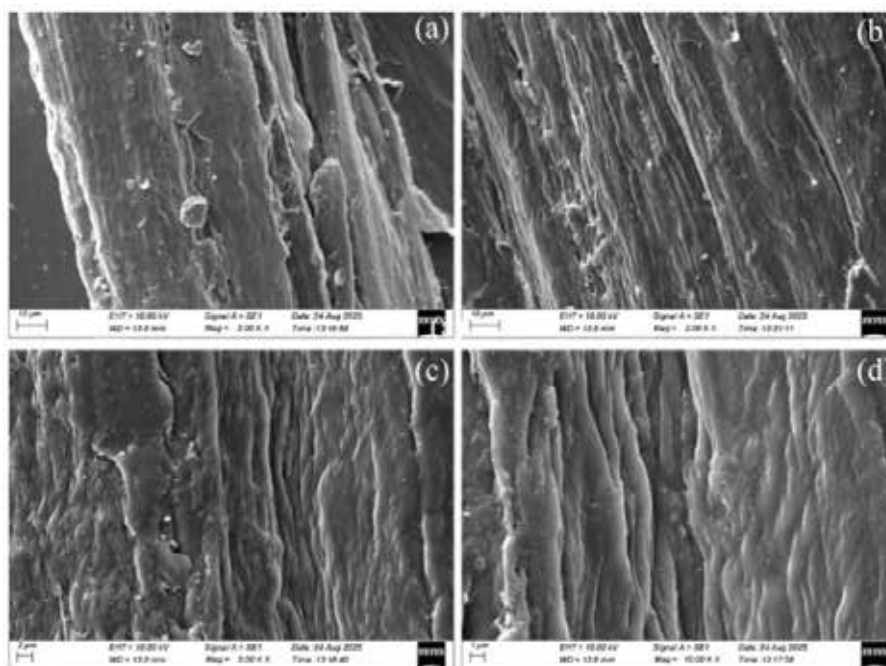


Fig. 3: SEM micrographs of chemically treated MOP fiber: (a) fiber bundle structure and surface irregularities; (b) longitudinal grooves and uneven surface texture, and (c) fibrillar structure, and (d) well-defined microfibrils

The fibers are shown in Fig. 3(d) densely packed and well-orientated cellulose microfibrils that are parallel to the fiber. This alignment of surfaces microfibrillar is responsible for the increase in tension and stiffness [20]. Moreover, the micro scale rough surface texture was good for mechanical interlocking in composites. Additionally, the average fiber diameter for tuft fiber was $116.92 \pm 2.65 \mu\text{m}$, as determined using ImageJ software.

3.2 Structural Analysis

FTIR spectroscopy was done to determine the functional groups of MOP fiber and to confirm its lignocellulosic nature, represented in Fig. 4. The wide band at around 3476 cm^{-1} in the FTIR spectrum is attributed to O–H stretching vibration of cellulose and hemicellulose implies hydrophilic nature of fiber [21]. This hydroxylated structure is responsible for both moisture absorption and chemical reactivity.

Furthermore peaks at around $1500\text{--}1600 \text{ cm}^{-1}$ were assigned to aromatic C=C stretching vibrations of lignin present in fiber [22]. The presence of hydroxyl (–OH), carbonyl (C=O), and lignin-associated aromatic groups suggests that MOP fiber possesses chemical characteristics favorable for various value-added applications. The FTIR spectrum also reveals characteristic band in ($1200\text{--}900 \text{ cm}^{-1}$) due to C–O–C and C–O stretching of cellulose [23]. The results are in good agreement with the reported

spectrum of other natural fibers i.e. jute, banana and sisal and shows that MOP fiber is made of cellulose, hemicelluloses and lignin [24]. These functional groups can promote compatibility and interfacial bonding in bio-composite systems, while the lignocellulosic composition makes the fiber potentially suitable for fiber blends, and biodegradable packaging materials [25, 26].

3.3 Chemical Composition Analysis

The chemical compositions of MOP fiber show that it consists of 54.02% cellulose, 16.57% hemicelluloses and 17.81% lignin [27]. Contents of cellulose higher than 50% are desirable for mechanical reinforcement, pulp and composites applications. This value is close to that of the banana fiber and slightly lower than that of jute, thus suggesting good prospect as a strength in stiffness, shown in Table I. The intermediary content of hemicellulose gives enough flexibility and extensibility while it raises moisture sensitivity owing to its hydrophilicity. The higher hygroscopicity of hemicellulose-rich fibers may potentially compromise the dimensional stability in a humid atmosphere. Its higher lignin content than jute or flax contributes to its stiffness, microbial resistance, and thermal stability, but may make it less soft and white in textile grade applications [28]. The near neutral chemical composition indicates that MOP fiber would be most suitable for semi-structural

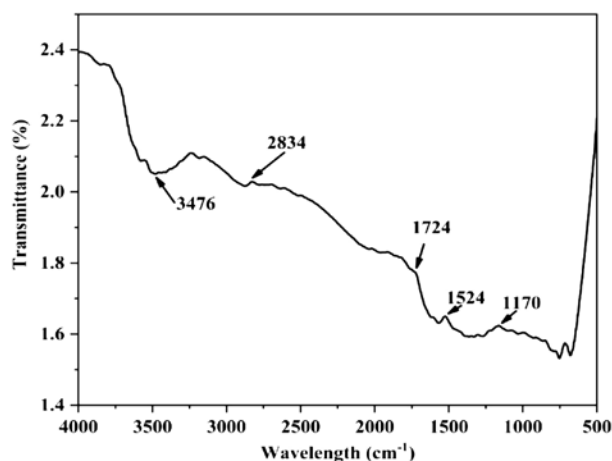


Fig. 4: FTIR spectrum of MOP fiber.

application such as composites, rope, matting paper and packaging. If the chemical treatments applied to decrease lignin and hemicellulose content to the fiber, it could be tailored for higher value textile use as well as biopolymer applications.

3.4 Solubility in Sulfuric Acid

The dissolution study was conducted using sulfuric acid (H_2SO_4), as concentrated sulfuric acid is capable of disrupting the intermolecular and intramolecular hydrogen-bonding network of cellulose, resulting in the dissolution of cellulosic fibers [34]. Accordingly, the dissolving behavior of MOP fiber was studied using concentrated H_2SO_4 solutions of 50%, 60%, and 70% to understand its chemical resistance under strong acidic conditions. The fibers were immersed in the solutions and agitated at room temperature (25 °C). At 50% H_2SO_4 , the fibers swelled and softened after 1 h but did not dissolve. A similar trend was observed at 60%, where the fibers showed swelling and some structural damage, yet remained undissolved. However, at 70% H_2SO_4 , the fibers rapidly swelled and completely dissolved within 30–60 min, forming a viscous solution. MOP fibers dissolves

in 98% sulfuric acid as retting was done by water [35]. This behavior suggests that MOP fiber has comparatively higher acid resistance than typical cellulosic fibers, which usually dissolve at lower acid concentrations [36]. The resistance up to 70% acid can be explained by its stable lignocellulosic structure, where higher cellulose crystallinity, the lignin barrier effect, and intermolecular crosslinking work together to limit acid penetration and slow down degradation [37]. As a result, this fiber shows good potential for applications that require durability and chemical resistance, such as textile processing and composite reinforcement.

3.5 XRD Analysis

The crystalline structure of the MOP fiber was studied using X-ray diffraction (XRD) which is demonstrated in Fig. 5. The diffraction pattern exhibited a main peak at $2\theta \approx 23.04^\circ$, which is attributed to the (002) plane of cellulose I, indicating a crystal structure of cellulose [38]. Additional peaks are located at $2\theta \approx 15.45^\circ$ and $2\theta \approx 17.3^\circ$, due to amorphous phase related to hemicellulose and lignin remaining in the sample [38]. The crystalline index (CI) of MOP fiber was evaluated as 43.75%. The presence of crystalline and amorphous phases proves that the fiber is a semi-crystalline material which is comparable to other natural fibers, including jute, hemp, and sisal [39, 40]. The semi-crystalline nature of MOP fiber provides a good balance of strength and flexibility, which enhances its potential for use in composites, packaging, and other sustainable reinforcement applications.

3.6 Density and Moisture Properties Analysis

The density of MOP fiber is measured to be as 1.25 g/cm³. The comparison of density of different fibers are shown in Table II. The measured moisture content (9.19%) and moisture regain (10.12%) are also moderate in the matter of the water absorbency of MOP fiber when compared with common textile fibers like cotton and silk shown in

Table I: Chemical composition of different fibers

Fiber name	Cellulose %	Hemicellulose %	Lignin %	References
MOP	54	16.5	17.8	This study
Jute	51 – 73	12.0 – 21.0	5.0 – 16.0	[29]
Flax	60 – 81	14.0 – 21.0	2.0 – 3.0	[30]
Coir	32–44	0.15–0.2	40.0–45.0	[31]
Banana	55	20.3	25.3	[32]
Cotton	82 – 92	2.0 – 6.4	0.0– 1.7	[33]

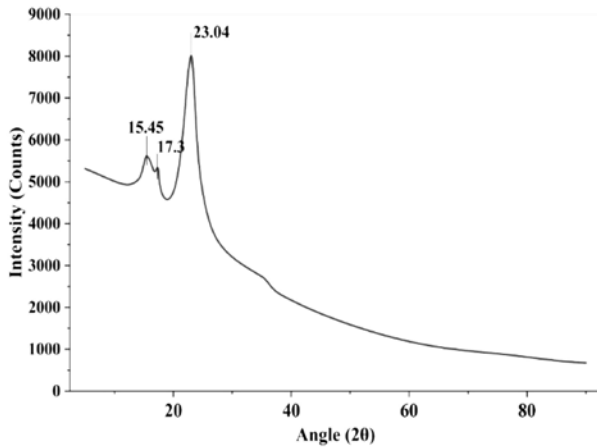


Fig. 5: XRD pattern of MOP fiber.

Table II [41]. It would be better as blended fiber however, while moisture sensitivity may limit operation in a high humidity environment, the use of surface treatment such as silane modification can greatly enhance its performance for composite and packaging applications.

3.7 Mechanical Properties Analysis

Fig. 6 illustrates the tensile behavior of MOP fibers, revealing variations in mechanical performance among the tested samples. Sample 1 reached a maximum force of 1.47 N at an extension of approximately 5%, indicating relatively brittle behavior with limited elongation before failure. In contrast, Sample 2 exhibited a higher maximum force of 2.02 N at an extension of about 15%, suggesting greater ductility and flexibility. These differences in mechanical performance may be attributed to variations in fiber maturity, microfibril orientation, or the efficiency of the extraction process [42]. The strength combined with the flexibility in tensile results indicates that, depending upon processing conditions, MOP fiber is customizable to various applications. This behavior is beneficial for blended fibers and composites where both structural strength and impact absorption are necessary.

3.8 Thermogravimetric Analysis

The thermal stability and degradation behavior of MOP fibers were analyzed using thermogravimetric analysis (TGA) under a nitrogen atmosphere, which is demonstrated in Fig. 7. Typical three stages of decomposition are found on TGA curves typical of lignocellulosic fibers. Weight loss of 5–10% below 150°C is due to evaporation of absorbed moisture and volatile material [43, 44]. The main degradation of which occurs in the range 200°C–400°C is mainly concerned with the thermal decomposition of

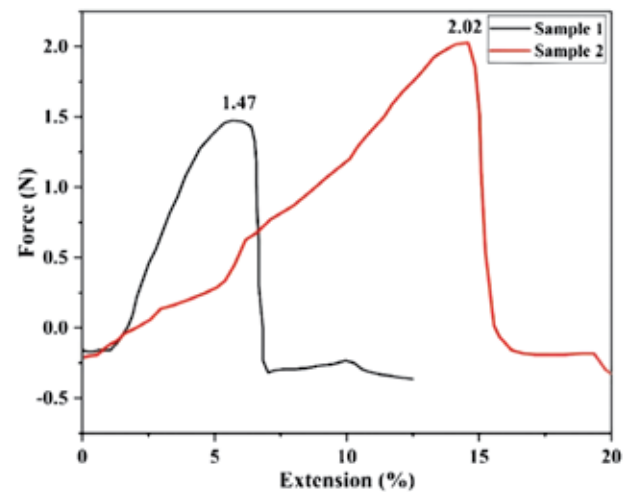


Fig. 6: Force–elongation behavior of MOP fiber

hemicellulose and cellulose [54]. Hemicellulose degrades at lower temperature because of its amorphous structure, while cellulose decomposes at higher temperature as it is crystalline in nature [55]. This first phase is the one that shows the largest mass-loss (those of greater lignin content) [56, 57]. Beyond 400°C, slow decomposition of lignin and formation of char occurs. The fiber is thermally stable at about 250 °C, suggesting potential compatibility with typical thermoset and thermoplastic polymers used in composites processing [57, 58].

Table II: Comparison of different fiber properties

Fiber	Density (g/ cm ³)	Moisture Regain (%)	Peak Force (N)	Extension (%)	Thermal Stability (°C)	Char Residue (%)	References
MOP	1.25	10.12	2.02	15.00	250.0	7.09	This work
Banana	1.35	9.00-11.00	4.00–6.00	10.87	200.0	6.00	[45, 46]
Cotton	1.52-1.56	7.00-8.50	0.20–0.50	3.00-12.00	220.0	18.80	[26, 47]
Jute	1.30-1.50	12.00-23.00	3.50–5.50	1.10-3.30	205.0	21.00	[26, 48]
Flax	1.54	12.00	6.00–12.00	1.20-12.00	220.0	22.50	[26, 49]
Coconut	1.15-1.20	13.00-14.00	1.50-3.00	14.00	228.6	15.90-29.50	[50-53]

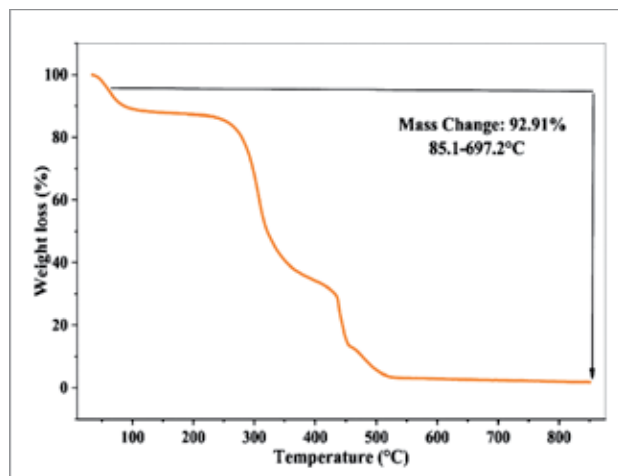


Fig. 7: TGA curve of MOP fiber.

The significant mass loss of 92.91% from 85.1°C to about 697.2°C and its char residue 7.09% suggests that MOP fiber is biodegradable with potential applications as reinforcement in sustainable polymer composites and biodegradable packaging systems.

4. CONCLUSION

The potential of *Moringa oleifera* pod fiber as a sustainable lignocellulosic material is systematically investigated in this study. The extraction, alkali treatment, and subsequent physical, mechanical, and chemical characterization of MOP fiber revealed a chemical composition of 54.02 % cellulose, 16.57 % hemicellulose, and 17.81 % lignin. The crystalline index (CI) of moringa fiber was evaluated as 43.75 %. Its semi-crystalline structure, flexibility, and micro fibril alignment contribute to good tensile strength, thermal stability, and biodegradability. Alkali-induced modifications reduced the inherent stiffness of the extracted fibers, resulting in a more flexible structure. This increase in flexibility transformed the previously un-spinnable material into a viable candidate for yarn production in fiber blends. Compared to established fibers such as jute, banana, and hemp, *Moringa oleifera* pod fiber shows competitive properties, particularly in durability and reinforcement potential. Although its higher lignin content requires chemical or enzymatic pretreatments to improve softness and whiteness, the fiber still offers excellent prospects for eco-friendly composites, packaging, and low-load construction materials. Moreover, advanced studies on fiber modification, large-scale production, composite fabrication, and life cycle assessment are required to establish moringa fiber as a viable alternative in the textile and allied industries.

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