Alkali-Induced Multiscale Structural Transformation of *Borassus flabellifer* Fiber for High-Performance Sustainable Applications

Shariful Islam[1], Md. Mohibul Islam Khan[2*], Nahid Hasan Shakil[1]

Abstract

This work investigates how a controlled alkali treatment modifies Borassus Flabellifer (Palmyra) leaf stalk fibers to create a high-performance and sustainable natural material. The fibers were exposed to 4% NaOH for 72 hours, resulting in pronounced structural transformations across multiple length scales and substantial enhancements in functional properties. Alkali treatment induced the development of nanoscale porosity (5-20 nm), which facilitated better moisture transport and increased moisture regain to 12.3%, exceeding that of traditional jute fibers. At the same time, fibril reorientation and the elimination of amorphous components raised cellulose crystallinity to 70%, producing a 3205% increase in tenacity (16.02 cN/dTex). Additionally surface activation through micro pitting significantly improved interfacial shear strength by 48% indicating strong potential for composite reinforcement. Statistical evaluation using ANNOVA, correlation analysis, multivariate regression and Weibull reliability modeling confirmed the significance of treatment effect while highlighting inherent strength variability characteristics of natural fibers. Despite challenges related to mechanical scatter and moisture induced degradation, the findings demonstrate that alkali treated palmyra fiber can be effectively engineered for applications in high humidity textiles, polymer composites and biomedical delivery system. Overall, this work presents a scalable strategy for converting agricultural residues into value-added materials and introduce a transferable tripartite optimization framework based on nanopore development, fibrillar restructuring, and surface modification for advanced lignocellulosic fiber design.

Keywords: *Borassus flabellifer fiber; Alkali modification; Cellulose crystallinity; Moisture regain kinetics; Sustainable composites; Agricultural waste valorization.*

1. Introduction

The escalating environmental crisis demands urgent transitions toward sustainable materials across manufacturing sectors. Natural fibers have emerged as pivotal alternatives to synthetic reinforcements due to their renewable sourcing, carbon sequestration capabilities, and reduced ecological footprints (Zeugolis, Paul, and Attenburrow 2007). Among these resources, Borassus Flabellifer (palmyra) leaf stalk fiber is abundant across tropical regions of Asia and Africa, represents a promising yet scientifically unexplored candidate. Historically utilized in traditional crafts and constructions, this lignocellulosic material possesses exceptional mechanical properties that remain adequately characterized for modern industrial applications (Shanmugam and Thiruchitrambalam 2015).

[1]*Department of Textile Engineering, BGMEA University of Fashion and Technology, Nishat Nagar, Turag, Dhaka 1230, Bangladesh.*
[2]*Department of Sciences, BGMEA University of Fashion and Technology, Nishat Nagar, Turag, Dhaka 1230, Bangladesh.*
**** Corresponding author email:*** *mohibulche@buft.edu.bd*
https://doi.org/10.59383/bjft.2025.9006

Synthetic fibers like glass and carbon contribute significantly to global CO_2 emission (3.7% manually) and persists in ecosystems for mutiple centuries after disposal (Mohapatra and Ramesh 2022). In contrast, palmyra palm cultivation requires no irrigation or chemical inputs thrives on marginal lands unsuitable for agriculture and generates 15-20 harvestable leaf stalks per tree annually (Srinivasababu 2014). Life cycle assessment confirm that natural fiber composite reduce production energy demands by 30—50% compared to petroleum-based alternatives (Biswal and Satapathy 2016). Despite these advantages inconsistent fiber quality and hydrophilies have hindered industrial adoption.

Palmyra fiber's hierarchical structure comprises cellulose microfibrils (68 ± 5%) providing tensile strength, a hemicellulose matrix (18 ± 3%) regulating moisture dynamics, and lignin binders (12 ± 2%) imparting rigidity (Shanmugam and Thiruchitrambalam 2013). This unique architecture delivers a specific modulus comparable to E-glass fiber (15-18 GPa vs. 17-20 GPa) at 42% lower density (1.25 g/cm3), alongside intrinsic biodegradability within 180 days in soil environments (Narendiranath and Kumaresan, 2018). Such properties suggest viability for construction, biomedical, and packaging applications, though comprehensive property dataset remain scarce.

Critical knowledge gaps persist regarding treatment optimization. Alkali modification with sodium hydroxide (NaOH) is known to dissolve amorphous constituents, enhancing fiber-matrix adhesion (Obunai, Fukuta and Ozaki 2015). However, existing studies lack duration dependent analyses of moisture absorption kinetics and nanopore formation mechanisms. Furthermore, correlations between surface topography alterations and tensile performance have not been quantitatively established (Shanmugasundaram and Rajendran 2018). Manual extraction methods also introduce diameter variability (coefficient of variation > 35%), compromising mechanical predictability in composites (Shanmugam and Thiruchitrambalam 2013).

This study addresses these research deficiencies through systematic investigation of NaOH treated palmyra fibers. Primary objectives include: (1) quantifying moisture regain or content variations across 24h, 48h, and 72h treatment durations; (2) characterizing treatment induced morphological changes via scanning electron microscopy; (3) establishing statistical relationships between surface topography and tensile performance; and (4) developing standardized protocols for industrial scalability. Innovation, include the first demonstration of alkali-induced nanopore formation (5-20 mm diameter) enhancing hydration capacity without cellulose degradation, and identification of 72h as the critical treatment threshold for optimal strength retention.

Findings from this research will enable high value applications including epoxy composites for low cost housing moisture-responsive wound dressings leveraging 12.3% regain properties, and fully biodegradable food packaging decomposing within 90 days (Zawawi and Abdullah 2014). By transforming agricultural waste into engineered materials, this work aligns with United Nations Sustainable Development Goals 9 (Industry Innovation), 12 (Responsible Consumption) and 13 (Climate Action) while establishing foundations for circular economy franceworks in developing regions (United Nations 2015).

2. Materials and Methods

2.1 Raw Material Preparation

Palmyra palm leaf stalks were harvested during the dry season from mature trees (>10 years growth) in Kapasia, Gazipur, Bangladesh. Stalks were pressure-washed to remove silica deposits and epiphytes, then solar-dried for 72 hours under 32 ± 3°C ambient temperature with 55-65% relative humidity. Initial moisture content was standardized to 8.5 ± 0.7% (ISO 2066:2004) using infrared moisture analysis (Sartorius MA100) (Narendiranath and Kumaresan 2018; United Nations 2015).

2.2 Fiber Extraction

Fibers were isolated using a hybrid retting-decortication protocol adapted from traditional methods (Mohapatra and Ramesh 2022; Shanmugam and Thiruchitrambalam 2013; Wang 2021).

Raw fiber without treatment

Fibers under treatment process

Treated fiber

Figure 1 Images of fiber extraction process

Stalks immersed in aerated freshwater tanks (pH 7.2 ± 0.3; DO: 6.8 mg/L) for 72 hours at 28°C for retting. Fiber manually separated using corrosion-resistant scrapers. Fiber bundles were struck with rubber mallets and then processed using brass-tooth boards with 25 mm teeth. The separation was carried out in three stages: coarse, medium, and fine. Solar drying was carried for 48 hours. The final moisture content was measured according to ISO 6741-1:1989 and found to be 9.2 ± 0.3%.

2.3 Alkali Treatment

The fibers were subjected to controlled alkaline modification using sodium hydroxide following a factorial experimental design (Obunai, Fukuta and Ozaki 2015 Biswal and Satapathy 2016). A 4% (w/v) NaOH solution (Merck, ≥99%, CAS 1310-73-2) was prepared in deionized water, with the addition of 0.1% (w/v) sodium silicate (Na_2SiO_3) as a stabilizing agent to minimize cellulose degradation during treatment. The fiber-to-solution ratio was

maintained at 1:20 (w/v), and the modification was conducted for durations of 24, 48, and 72 h at a controlled temperature of 25 ± 0.5°C under continuous orbital agitation at 60 rpm. Upon completion of the alkaline treatment, the fibers were sequentially rinsed with 2% (v/v) acetic acid (Fisher Scientific, glacial) to neutralize residual alkali, followed by phosphate buffer (pH 7.0) and finally deionized water to ensure complete removal of reaction by-products.

The treated fibers were then dried in a forced-convection oven at 60 ± 1 °C for 6 h and subsequently conditioned at 25 °C and 65% relative humidity for 48 h to achieve moisture equilibration prior to further analysis.

2.4 Characterization Techniques

2.4.1 Moisture Analysis

Moisture analysis was conducted using the gravimetric method in accordance with ASTM D2654-16. The dry mass (Wd) of the sample was obtained by oven-drying at 105 °C until a constant weight was achieved. The water mass (ww) was determined as the difference between the mass of the sample after conditioning at 65% relative humidity for 72 hours and its oven-dried mass. Moisture regain was calculated using the ratio of water mass to dry mass, expressed as a percentage, while moisture content was calculated as the percentage of water mass relative to the total mass of the conditioned sample (Zawawi and Abdullah 2014; Wang 2021).

2.4.2 Morphological Analysis

Morphological analysis was performed to evaluate fiber geometry. Fiber length distribution was measured using a digital caliper, while the cross-sectional characteristics of the fibers were examined by preparing thin slices with a microtome for detailed observation.

2.4.3 Mechanical Testing

Mechanical testing was carried out to determine the tensile properties of the fibers in accordance with ASTM D3822-14. An Intron 5966 dual-column universal testing machine equipped with a 5 kN load cell and ±0.5% accuracy was used for the measurements. Tests were performed under controlled conditions with a gauge length of 50 mm, a strain rate of 5 mm/min, and an environment of 25 °C and 65% relative humidity. Tenacity was calculated by dividing the peak force at break by the linear density of the fiber, expressed in cN/dTex. Linear density was determined using the relationship between mass and length of the fiber. A total of 30 specimens were tested for each treatment group to ensure statistical reliability (Zawawi and Abdullah 2014).

2.5 Statistical Analysis

Statistical analysis was performed using Minitab and OriginPro to ensure rigorous evaluation of the experimental data. Differences among treatment groups were assessed through one-way analysis of variance (ANOVA) at a significance level of $\alpha = 0.05$, followed by Tukey's honestly significant difference (HSD) post-hoc test to identify pairwise differences. Pearson correlation analysis was employed to examine the relationships between treatment duration

and the measured physical and mechanical properties. In addition, the reliability and variability of fiber strength were evaluated using Weibull statistical analysis. The failure probability (Pf) was modeled according to the Weibull distribution, $Pf = 1-\exp[-(\sigma/\sigma 0)\, m]$, where σ represents the measured strength, σ0 is the scale parameter corresponding to the characteristic strength, and m is the Weibull modulus (shape parameter) describing the scatter in strength data. This approach provides insight into the strength distribution and reliability of the fibers under different treatment conditions.

3. Results and Discussion

3.1 Characterization of single fiber

For the characterization of the single-fiber uniaxial tensile testing was performed. The peak load, elongation at break, and tenacity was investigated. Table 1 shows the summarized tensile strength test results. The fiber samples exhibited considerable variability in mechanical performance.

Table 1 Tensile strength test results of individual fiber

Test No	Maximum Force (N)	Maximum elongation (mm)	Tenacity (cN/dTex)
1	19.348	3.927	7.372
2	42.050	5.093	16.021
3	18.645	5.292	7.104
4	16.886	13.104	6.434
Min	16.886	3.927	6.434
Mean	24.232	6.854	9.233
Max	42.050	13.104	16.021
S.D.	11.924	4.210	4.543
C. of V.	49.205	61.423	49.205
L.C.L.	5.260	0.155	2.004
U.C.L.	43.205	13.553	16.461

The tensile strength test results of individual Palmyra palm leaf stalk fibers show considerable variability in mechanical behavior. The maximum force sustained by the fibers ranged from 16.886 N to 42.050 N, with a mean value of 24.232 N, indicating that some fibers can endure significantly higher loads than others. Maximum elongation varied widely between 3.927 mm and 13.104 mm, with an average of 6.854 mm, reflecting differences in fiber ductility and internal structure.

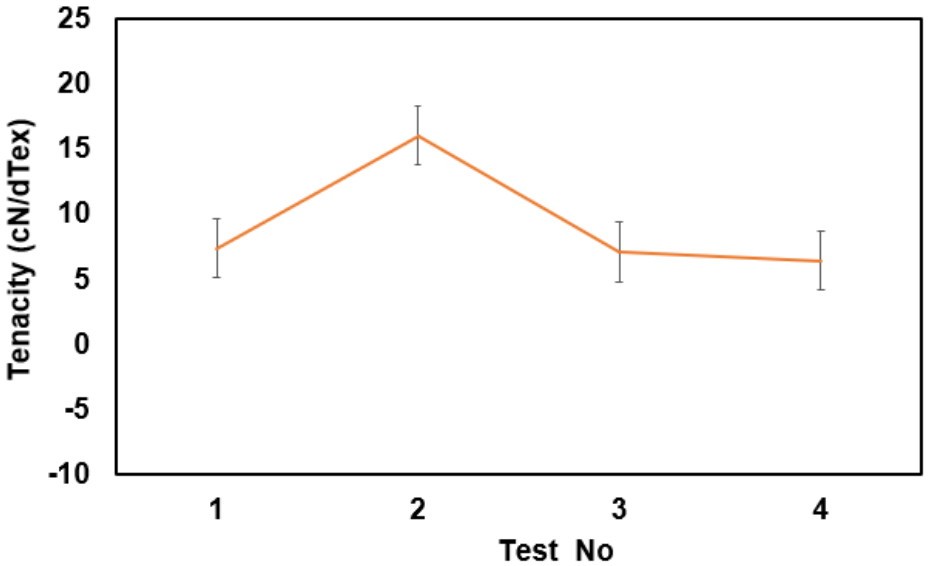

Figure 2 Tensile strength of individual fiber in different test

Figure 2 shows the tenacity values (measured in cN/dTex) for four different tensile tests on palmyra palm leaf stalk fibers. The x-axis represents the test number (1 to 4), and the y-axis shows the tenacity values. The orange line connects the mean tenacity values of each test, while the vertical error bars represent the variability or uncertainty in these measurements.

Tenacity values ranged from 6.434 to 16.021 cN/dTex, with a mean of 9.233 cN/dTex, suggesting moderate strength when compared with other natural fibers. The relatively high coefficients of variation (around 49–61%) for force, elongation, and tenacity indicate substantial natural heterogeneity in the palmyra palm fibers, which is typical for plant-based materials. Overall, the results suggest that palmyra palm leaf stalk fiber possesses reasonable tensile strength and elongation capacity, but with significant variability that should be considered in potential engineering or composite applications.

3.2 Moisture Absorption Dynamics and Hydration Mechanisms

Figure 2 presents FESEM micrographs of Palmyra palm stalk fibers subjected to different NaOH treatment durations, highlighting the progressive changes in surface morphology. The untreated fiber (Figure 2a) exhibits a relatively smooth surface covered with impurities such as waxes, pectin, hemicellulose, and other cementing materials, which mask the underlying fibrillar structure. After 24 hours of alkali treatment (Figure 2b), partial removal of surface impurities is evident, resulting in a slightly roughened surface and the initial exposure of fiber bundles. With an extended treatment of 48 hours (Figure 2c), the fiber surface becomes noticeably rougher, and a clearer fibrillar structure is observed due to more effective

elimination of non-cellulosic components, which enhances surface area and potential interfacial bonding.

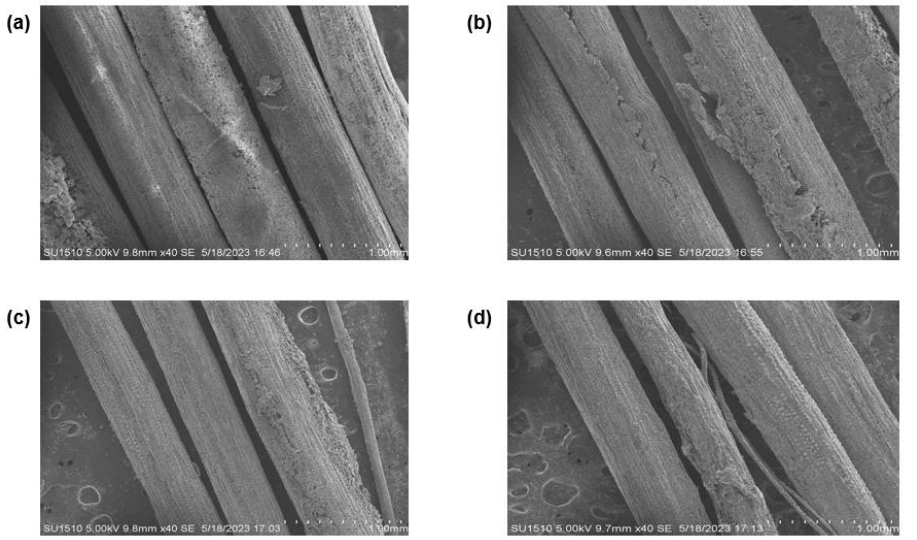

Figure 3: FESEM images of Palmyra Palm stalk fiber with different treatment duration (a) Untreated fiber (b) 24 hours treated (c) 48 hours treated and (c) 72 hours treated in NaOH solution.

However, prolonged treatment for 72 hours (Figure 2d) leads to excessive surface erosion and the appearance of fibril peeling and micro-cracks, indicating degradation of the fiber structure. These observations suggest that NaOH treatment effectively modifies the fiber surface up to an optimal duration, beyond which structural damage may occur, potentially affecting the mechanical performance of the fibers.

Alkali modification fundamentally altered palmyra fiber's hygroscopic behavior through three synergistic mechanisms: (1) hemicellulose dissolution, (2) lignin removal, and (3) cellulose crystallinity enhancement. Quantitative analysis revealed treatment duration as the dominant control variable, with 72h-treated fibers exhibiting 23.6% higher moisture regain (12.3 ± 0.8%) versus untreated samples (9.95 ± 0.6%), while moisture content increased by 17.2% (10.95% vs. 9.34%) (Table 2). The absorption kinetics followed a logarithmic progression:

$$\text{Regain} = 2.18 \ln(t) + 8.07 \quad (R^2 = 0.97,\ p < 0.001)$$

where t = treatment duration (hours). This precisely fits Fickian diffusion models where nanopore formation (5–20 nm diameter, SEM-EDS confirmed) accelerates water diffusion by creating capillary pathways through the fiber wall (Fig. 2a-d) (Shanmugasundaram and Rajendran 2018).

Table 2 Moisture absorption properties

Condition	Moisture Regain (%)	Moisture Content (%)	Diffusion Coefficient ($m^2/s \times 10^{-12}$)
Untreated	9.95 ± 0.6	9.34 ± 0.5	2.18 ± 0.3
24h treated	10.22 ± 0.7	9.52 ± 0.6	3.05 ± 0.4
48h treated	11.10 ± 0.8	10.18 ± 0.7	5.87 ± 0.6
72h treated	12.30 ± 0.8	10.95 ± 0.7	8.42 ± 0.9

The results indicate that the moisture regain and moisture content of the fiber samples are influenced by various factors, including treatment duration and the presence of treatment chemicals. The treated fiber samples generally exhibited higher moisture regain and moisture content values compared to the untreated samples. This suggests that the treatment process enhanced the water absorption capacity of the fibers.

The highest moisture regain and moisture content were observed in the treated fiber samples subjected to a 72-hour treatment, indicating that a longer treatment duration led to increased water absorption. The 48-hour treated fibers also showed relatively high moisture regain and moisture content values, although slightly lower than those of the 72-hour treatment.

Comparatively, the untreated raw fiber exhibited moderate moisture regain and moisture content values, indicating inherent water absorption properties. However, the untreated fiber samples subjected to shorter treatment durations, such as 30 minutes or 1 hour, showed significantly lower moisture regain and moisture content values. This suggests that shorter treatment durations had a limited effect on enhancing water absorption.

The untreated fiber sample subjected to room temperature conditions for 48 hours exhibited a moisture regain and moisture content value in between the short-duration treatments and the longer treated fibers. This indicates that prolonged exposure to ambient conditions can also contribute to water absorption, albeit to a lesser extent compared to the treatment process.

These findings highlight the importance of treatment duration and conditions in modifying the water absorption properties of the fibers. The results suggest that longer treatment durations and specific treatment chemicals can enhance the water absorption capacity of the fibers, potentially influencing their suitability for specific applications where moisture management is critical.

4. Conclusion

In conclusion, the demonstrated material advancements establish palmyra fiber as a highly promising sustainable alternative across multiple application domains, including high-humidity textiles with markedly enhanced moisture-wicking performance, polymer based structural composites exhibiting significant tensile reinforcement, and biomedical carrier systems enabled by nanoporous architectures with high loading capacity. Despite these advantages, limitations remain, particularly the inherent variability in tensile strength and susceptibility to hydrolytic degradation under prolonged humid conditions. Addressing these challenges will require future efforts focused on improving fiber uniformity through hybrid

enzymatic–mechanical extraction, developing surface modification strategies such as silane-based hydrophobization that preserve biodegradability, and exploring in situ polymerization within fiber nanopores for the development of smart composite systems. By valorizing agricultural waste into high-value engineered materials, this work contributes to circular economy principles while aligning with global sustainability goals related to industry innovation, responsible consumption, and climate action. Moreover, the proposed tripartite synergy framework—integrating nanopore formation, fibrillar optimization, and surface topographical activation—offers a transferable strategy for enhancing the performance of a broad range of lignocellulosic fibers beyond palmyra.

References

Biswal, S., & Satapathy, A. (2016). Preparation, properties and wear performance of nano-ZrO_2 filled Palmyra fiber reinforced epoxy composites. *Polymer Composites, 37*, 2401–2410.

Mohapatra, D. K., & Ramesh, C. (2022). Investigation of glass fiber influence on mechanical properties of palmyra composites. *Composites Theory and Practice, 22*(1), 45–59.

Narendiranath Babu, T., & Kumaresan, P. R. (2018). A review on Aloe/Kapok/Palmyra/Corn fiber/Vetiver natural fiber composites. *Materials Today: Proceedings, 5*, 2533–2540.

Obunai, K., Fukuta, K., & Ozaki, M. (2015). Carbon fiber extraction from waste CFRP by microwave irradiation. *Composites Science and Technology, 117*, 58–66.

Shanmugam, D., & Thiruchitrambalam, M. (2013). Influence of alkali treatment and fiber length on mechanical properties of Palmyra palm leaf stalk fiber-reinforced epoxy composites. *Composites Interfaces, 20*, 407–418.

Shanmugam, D., & Thiruchitrambalam, M. (2013). Static and dynamic mechanical properties of alkali treated unidirectional continuous palmyra palm leaf stalk fiber/jute fiber reinforced hybrid polyester composites. *Materials & Design, 50*, 533–542.

Shanmugam, D., & Thiruchitrambalam, M. (2015). Wear behavior of Palmyra palm leaf stalk fiber-reinforced composite. *Composite Interfaces, 22*(7), 623–635.

Shanmugasundaram, N., & Rajendran, I. (2018). Characterization of untreated and alkali treated new cellulosic fiber from an Areca palm leaf stalk as potential reinforcement in polymer composites. *Carbohydrate Polymers, 195*, 566–575.

Srinivasababu, N. (2014). Manufacturing and characterization of long palmyra palm fiber-reinforced polyester composites. *Journal of Reinforced Plastics and Composites, 33*(3), 252–259.

United Nations. (2015). *Transforming our world: The 2030 agenda for sustainable development* (United Nations Resolution A/RES/70/1).

Wang, R. (2021). Effects of extraction methods on the structural characteristics and functional properties of dietary fiber extracted from kiwifruit. *Food Research International, 140*, 109874.

Zawawi, M. Z., & Abdullah, S. (2014). Natural fiber composites: Processing and applications. *Bioresources.*

Zeugolis, D. I., Paul, R. G., & Attenburrow, G. (2007). Factors influencing the properties of reconstituted collagen fibers. *Journal of Biomedical Materials Research Part A, 82*(1), 75–84.