Evaluation of Alkaline Hydrogen Peroxide Pretreated Bagasse, Paddy Straw, and Banana Stem Fibers for Cellulose-Based Composite Development

W. M. I. S. Jayaweera[1], M. M. H. Malshan[1] and T. N. Fernando[2]

[1]University of Moratuwa, Moratuwa, Sri Lanka
[2]General Sir John Kotelawala Defence University, Sri Lanka
isharasch.jaya@gmail.com

Abstract. Natural fiber reinforcement in sustainable construction materials has gained significant attention due to environmental concerns and resource availability. This study evaluates the comprehensive performance of cellulose fibers extracted from three Sri Lankan agricultural residues (bagasse, paddy straw, and banana stem) using alkaline hydrogen peroxide (AHP) pretreatment for composite development applications. Three pretreatment solutions with varying NaOH concentrations (1000, 1500, and 2000 mol/m³) combined with hydrogen peroxide were systematically evaluated through a factorial experimental design with 27 sample combinations, each replicated three times. Comprehensive characterization including weight loss analysis, holocellulose content determination, tensile strength testing (18.34-171.40 MPa), fiber diameter measurements (134.0-268.1 µm), FTIR spectroscopy, and SEM morphological analysis was conducted. Results demonstrated holocellulose content ranging from 86.63% to 95.90% with weight losses between 49.99% and 74.32%. Statistical analysis revealed that banana stem fibers exhibited the highest tensile strength (171.40 MPa), while the C1 pretreatment solution (1000 mol/m³ NaOH) was identified as the optimum concentration for maximizing holocellulose content (93.75%) while minimizing weight loss and processing costs.These findings demonstrate the significant potential of AHP-pretreated agricultural waste fibers as sustainable alternatives to synthetic reinforcements in construction composites, contributing to both waste valorization and environmentally friendly building materials development.

Keywords: Lignocellulosic. pretreatment, cellulose, alkaline hydrogen peroxide

1 Introduction

The construction industry's growing emphasis on sustainable materials has intensified research into natural fiber reinforcement systems as alternatives to synthetic counterparts. Environmental concerns, resource depletion, and health hazards associated with conventional reinforcing materials like asbestos have necessitated the development of eco-friendly alternatives [1-3].

Most cellulose in nature exists as lignocellulosic compounds containing cellulose, hemicellulose, and lignin in complex structural arrangements. The lignocellulosic matrix consists of hemicellulose polymers surrounding long cellulose chains, with both encased by lignin networks [1]. This hierarchical structure presents both opportunities and challenges for cellulose extraction, as the valuable cellulose component must be separated from lignin and hemicellulose while maintaining its structural integrity and mechanical properties.

Agricultural waste streams globally generate substantial quantities of lignocellulosic materials, including corn husks, corn stoves, bagasse, sugarcane leaves, guinea grass, banana stems, paddy straw, and various wood fibers. In Sri Lanka specifically, bagasse from sugar processing, paddy straw from rice cultivation, and banana stems from fruit production represent abundant, underutilized resources with significant potential for value-added applications [4-5].Cellulose, with its chemical formula $(C_6H_{10}O_5)_n$, exhibits favorable properties including low density, biodegradability, renewability, and satisfactory tensile strength and water absorption characteristics [2]. These properties make cellulose fibers suitable for diverse applications including food packaging, sustainable cement-based products, mortar systems, reinforcement of brittle building materials, and specialized filtration systems [3].

Several pretreatment methodologies exist for lignin removal from lignocellulosic materials, categorized into biological, chemical, and physical approaches. While biological methods offer environmental benefits, they typically require extended processing times. Physical methods may compromise fiber integrity, whereas chemical pretreatments provide controllable and efficient lignin removal [6-8]. Among chemical methods, alkaline pretreatment, microwave-assisted alkaline treatment, and alkaline hydrogen peroxide (AHP) pretreatment have demonstrated industrial viability.

Recent research has identified significant gaps in comprehensive performance evaluation of AHP-pretreated fibers from diverse agricultural sources, particularly regarding their suitability for composite applications. While individual studies have examined extraction efficiency, limited research has systematically evaluated the relationship between pretreatment parameters, fiber characteristics, and composite performance potential [9-11]. Fernando et al. [1] demonstrated that AHP pretreatment represents an effective, simple, and cost-efficient method operating at ambient temperature and atmospheric pressure, making it particularly suitable for industrial applications. However, comprehensive optimization studies comparing multiple agricultural residues and systematic evaluation of resulting fiber performance for composite applications remain limited.

Although cotton is naturally available cellulose, it is not suitable for this application as it is not abundantly available in Sri Lanka [5]. Therefore, in the present study, bagasse, paddy straw, and banana stems were selected as target materials due to their abundant availability as agro-plant residues in Sri Lanka and their potential for sustainable fiber extraction.

The present study addresses these gaps by providing comprehensive performance evaluation of cellulose fibers extracted from three abundant Sri Lankan agricultural residues (bagasse, paddy straw, and banana stems) using systematic AHP pretreatment optimization. The research specifically aims to: (1) determine optimal pretreatment solution concentrations for maximum cellulose extraction efficiency with minimum material loss, and (2) comprehensively evaluate the structural, mechanical, and morphological characteristics of extracted fibers for composite reinforcement applications.

2 Materials and Methodology

2.1 Materials

Industrial-grade sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), and hydrochloric acid (HCl) were purchased. Bagasse samples were collected from Palwattha Sugar Industry, and other lignocellulose materials were collected from Hambantota District. All raw materials were sun dried and shredded using shredder machine.

2.2 Alkaline Peroxide Pretreatment

Pre-treatment solutions were prepared by mixing three different concentrations (C1, C2 & C3) of NaOH with equal volume (10 ml) and an equal volume (0.5 ml) of hydrogen peroxide (H_2O_2) as given in Table 1. Selected three lignocellulose materials were named as follows: bagasse (B), paddy straw (PS), and banana stem (BS). The experimental design was a two-factor factorial. Two variable factors were three plant residues and three pretreatment solutions. The experiment consists of nine treatments. Further, replicating each treatment three times, the experiment consisted of 27 samples. The experimental protocol was designed with a standardized biomass-to-solution ratio: each 1 g of lignocellulosic material was treated with 10 mL of pretreatment solution. For practical handling and replication consistency, batch processing involved scaling up to 5 g biomass with 50 mL solution in 500 mL beakers.

Table 1. Concentration of Pre-treatment Solution

Pre-treatment solution	NaOH Concentration
C1	1000 mol/m^3
C2	1500 mol/m^3
C3	2000 mol/m^3

The treatment protocol involved continuous mixing every 4 hours over a 24-hour degradation period at ambient temperature (25±2°C) and atmospheric pressure. Following pretreatment, samples underwent triple washing with distilled water, 2-hour immersion in 10% HCl solution for removing residual alkali and mineral impurities, final washing, and thermal treatment at 60°C for 4 hours according to AHP treatment procedure [1] to ensure complete moisture removal and fiber stabilization.

2.3 Fourier Transform InfraRed Spectroscopy Analysis

Both of Raw lignocellulosic materials of bagasse, paddy straw, banana stem, and pretreated fibers were analyzed using Fourier Transform Infra-Red spectroscopy to confirm successful cellulose extraction. The spectra were collected in the 4000–600 cm^{-1} range at 4 cm^{-1} resolution with an accumulation of 24 scans in transmittance mode. The test was performed at ambient conditions on a Bruker ALPHA spectrometer (Bruker Corporation, Billerica, MA).

2.4 Weight Loss Analysis and Holocellulose Content

Weight loss percentage was calculated using the formula: Weight Loss % = [(Initial dry weight - Final dry weight)/Initial dry weight] × 100. Initial moisture content was determined by oven-drying samples at 105°C until constant weight, and all calculations were based on moisture-free basis to ensure accuracy. Holocellulose content was determined using the chlorination method as described by Fernando et al. [1], calculated as: Holocellulose % = [(Weight after delignification)/Initial dry weight] × 100.

2.5 Scanning Electron Microscopy (SEM)

The selected three raw lignocellulosic materials and pretreated fibers from the AHP pretreatment were observed for surface morphology using Scanning Electron Microscopy (SEM). The samples were gold sputter coated prior to observation and examined with an accelerating voltage of 10 kV. Morphological analysis of fibers was performed on EVO 18, Carl Zeiss AG, Germany.

2.6 The Tensile Strength and Diameter

According to ASTM D3822–07, the tensile strength of pretreated fibers was measured using an Instron Universal Testing Machine. The diameter of pretreated fibers was measured by a Projectina microscope.

3 Results and Discussion

3.1 Weight Loss Percentage and Holocellulose Content

Fig. 1 and Fig. 2 show the variation of percentages of weight loss and percentage of hollow cellulose content of pretreated fibers, respectively. The concentration-dependent increase in weight loss (C1 < C2 < C3) reflects enhanced lignin and hemicellulose removal efficiency with increasing alkaline strength, consistent with previous findings [12-14]. ==The relationship between fiber type and weight loss percentage can be explained by the initial chemical composition: bagasse exhibited the lowest weight loss due to its higher lignin content (25.33%) [4], which provides more resistant structural components, while banana stem showed the highest weight loss corresponding to its lower lignin content (13%) [6], making it more susceptible to alkaline degradation. This relationship indicates that lower lignin content materials experience higher weight loss but potentially yield purer cellulose fractions.

Holocellulose content analysis demonstrated values exceeding 86.63% . Interestingly, bagasse samples showed higher holocellulose content in pretreated fibers compared to banana stem samples, which correlate with their initial cellulose content and structural stability during processing.

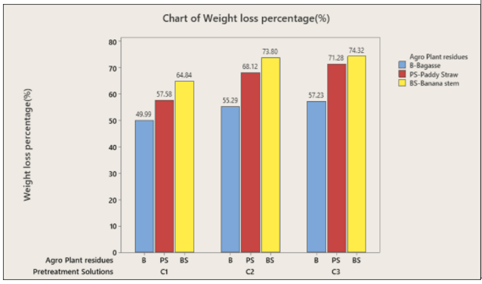

Fig.1. Variation of weight loss percentage

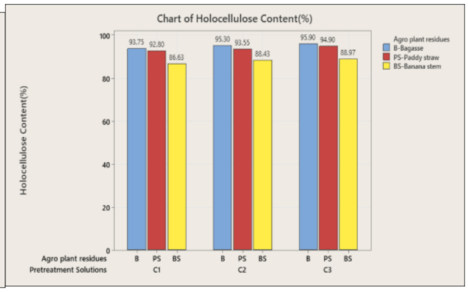

Fig.2. Holocellulose Content

3.2 Structural Characteristics

The comparison of FTIR results of raw bagasse, raw paddy straw, raw banana stem fibers, and pretreated fibers from the AHP pretreatment with three different pretreatment solutions (C1, C2, & C3) of lignocellulosic materials has confirmed that the extracted fibers were cellulose as a comparison of the wave number. Fig. 3, Fig. 4, and Fig. 5 show the FTIR spectra of raw and pretreated fibers from three different pretreatments of bagasse, paddy straw, and banana stem. The peak at 1732 cm^{-1} in raw fibers is assigned to the characteristic of aliphatic ester in lignin and/or hemicellulose [7]. Disappearing of this peak in the treated fibers is due to the removal of most of the lignin and hemicellulose from the pretreated fibers. The peak at 893 cm⁻¹ represents the glycosidic C–H deformation with ring vibration contribution and OH bending in the treated fibers, which indicates the typical structure of cellulose [3]. The broad peak was observed around 3445 cm⁻¹ in the -OH stretching vibrations, and a small sharp peak that appeared at 2922 cm⁻¹ is attributed to the C-H stretching vibration of cellulose.

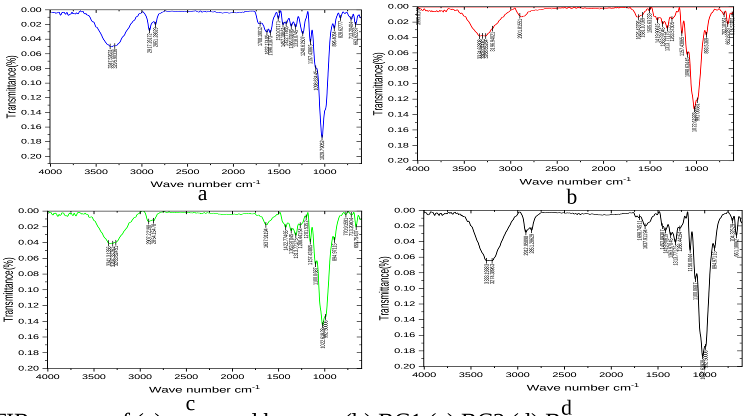

Fig.3. FTIR spectra of (a) untreated bagasse (b) BC1 (c) BC2 (d) BC3

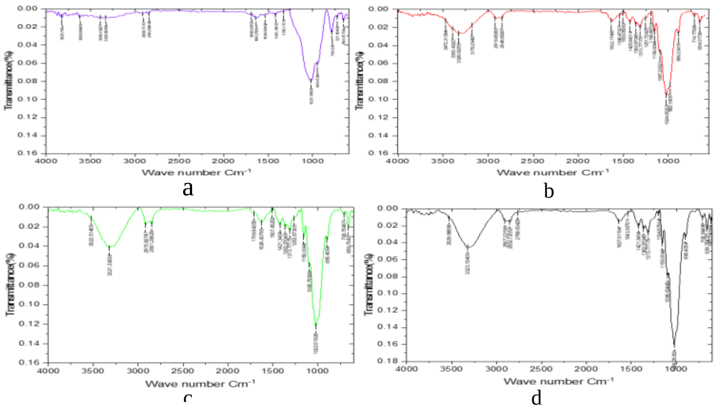

Fig. 4. FTIR spectra of (a) untreated paddy straw (b) PSC1 (c) PSC2 (d) PSC3

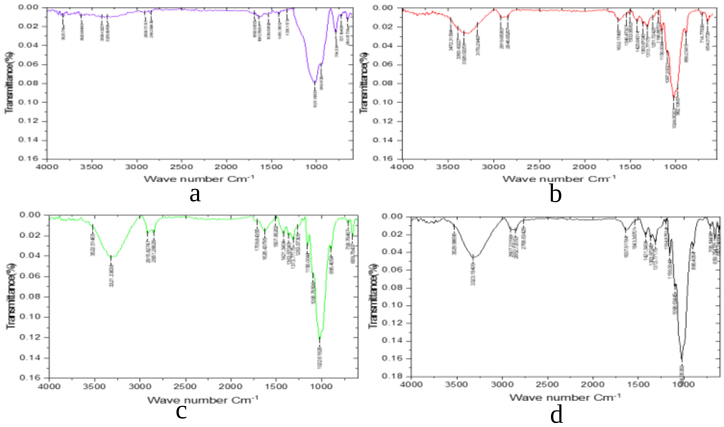

Fig. 5. FTIR spectra of (a) untreated banana stem (b) BSC1 (c) BSC2 (d) BSC3

3.3 Morphological Analysis and Mechanical Properties

The morphological features of raw lignocellulosic and pretreated three samples of bagasse, paddy straw, and banana stem from three different AHP pretreatment solutions Samples were analyzed by SEM images in Fig. 6 (a, b, c, d). Visual observation of the Image picture shows a comparatively smooth surface in raw fibers, and all pretreated fibers show a rough surface. This surface roughening indicates successful removal of lignin and hemicellulose matrices, exposing underlying cellulose fibrils and potentially enhancing fiber-matrix bonding in composite applications.

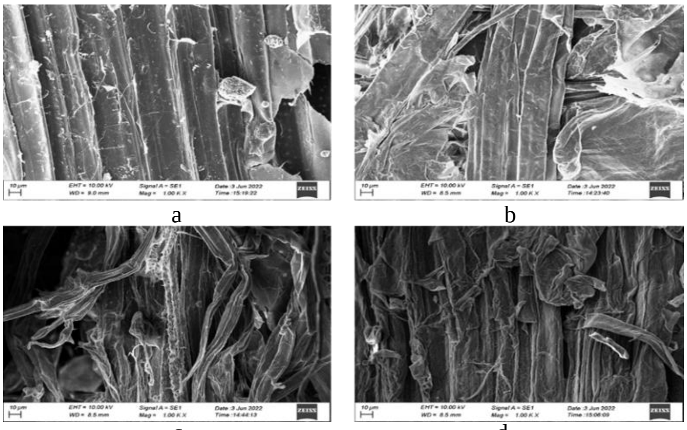

Fig. 6. SEM images of (a) untreated bagasse (b) BC1 (c) BC2 (d) BC3

Table 2. Fiber Diameter and Tensile Strength

Pre treatment Solution	Fiber Diameter (µm)	Tensile Strength (MPa)
B+C1	238.4	43.1
PS+C1	238.1	42.2
BS+C1	238.0	40.2
B+C2	268.1	20.0
PS+C2	268.0	18.5
BS+C2	265.0	18.3
B+C3	136.4	171.4
PS+C3	136.1	169.7
BS+C3	134.0	143.14

Table 2 shows the pretreated fiber's diameter and the fiber's tensile strength. Paddy straw fibers have a lower tensile strength. This may be due to the presence of significant amounts of silica particles; those particles reduce the hydrogen bond between the nano-fibers [8]. Although the same lignocellulosic material was pretreated with three different pretreatment solutions, there are no differences in tensile strength and dimeters.

3.4 Comparative Performance Analysis

According to Humphrey Danso, soil building blocks can be reinforced with natural fibers (coconut, oil palm, bagasse). The tensile strength and diameter of these fibers vary within a certain range. Bagasse fiber exhibits a tensile strength of 25-62 MPa and a diameter of 310-1190 µm. Oil palm fibers have a tensile strength of 65-141 MPa and a diameter of 190-820 µm. Coconut fibers exhibit tensile strength of 83-222 MPa and a diameter of 180-1010 µm [9]. Moreover, these pre-treated fibers (bagasse, paddy straw, banana stem) also can be effectively used to reinforce soil building blocks because these experimental pre-treated fibers' tensile strength and diameter meet requirements for developing soil building blocks.

A study by Elsaid reported the use of natural kenaf fibers to reinforce concrete, with the fiber tensile strength being nearly 160 MPa[10]. BSC1 and BSC2 pretreated fibers' tensile strength align with kenaf fiber tensile strength. Acidic pretreatment and ionic liquid are both pretreatment methods that can be used for the extraction of fibers from lignocellulosic materials. However, reactions happened in some elevated temperature and pressure conditions [10]. Comparing AHP, pre-treatment can be performed at room temperature and atmospheric pressure [1]. It is a low-cost and simple method for the extraction of fibers from lignocellulosic materials (bagasse, paddy straw, banana stem). Cement mortar composite mixers can be reinforced with several fibers, such as asbestos, PET, PP, PE, carbon fiber, etc.

However, chemical instability and higher cost are several disadvantages of those composites; further, asbestos fibers are harmful to human health[11]. The incorporation of cellulose as a reinforcing material in cement mortar has the potential to mitigate these disadvantages.

3.5 Optimization and Selection Criteria

Based on a comprehensive evaluation considering multiple criteria, the optimization strategy aims to balance extraction efficiency, mechanical properties, and economic factors. The C3 treatment provides the highest tensile strength (up to 171.4 MPa) and maximum holocellulose content but requires greater chemical usage and higher processing costs. In contrast, the C1 treatment offers an optimal balance, with an acceptable holocellulose content (93.75%), minimal weight loss, lower chemical consumption, and cost-effectiveness. Therefore, C3 treatment is recommended for applications requiring maximum strength despite its higher costs, while C1 treatment is the preferred choice for general composite reinforcement applications, as it effectively balances performance and economic considerations.

4 Conclusions

This comprehensive study successfully evaluated the performance of cellulose fibers extracted from three Sri Lankan agricultural residues using alkaline hydrogen peroxide (AHP) pretreatment, addressing both extraction optimization and composite application potential. The findings reveal that pretreatment concentration significantly influences both extraction efficiency and fiber mechanical properties, with the C3 treatment achieving the highest tensile strength (up to 171.4 MPa), while the C1 treatment offers an optimal balance of holocellulose extraction (93.75%) with minimal weight loss and greater economic feasibility. Statistical analysis confirms significant performance differences among the fiber types, with banana stem fibers exhibiting superior mechanical properties and bagasse fibers demonstrating excellent extraction efficiency. FTIR and SEM analyses further verify successful lignin removal and retention of structural integrity, confirming the effectiveness of AHP pretreatment for cellulose preservation. Comparative analysis highlights that the pretreated fibers possess mechanical properties competitive with established natural fiber reinforcements, demonstrating their viability for sustainable composite applications. For practical implementation in Sri Lankan cement mortar composites, the C1 pretreatment solution is recommended for cost-effective production, as it balances acceptable holocellulose content, minimal processing costs, and sufficient mechanical performance for general reinforcement applications. However, for specialized high-strength applications, the C3 treatment is preferable despite its higher processing costs due to its superior mechanical properties. The demonstrated environmental benefits and efficient resource utilization position this technology as a valuable approach for sustainable construction material development and agricultural waste valorization. Future research should focus on composite fabrication and performance testing, scaling up the process, and conducting comprehensive techno-economic assessments to facilitate the commercial implementation of this sustainable fiber extraction technology.

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