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Exploring the effect of wood ash treatment on the mechanical performance of BF for composite application

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ABSTRACT

This study explores the effects of alkaline treatments using wood ash on the tensile strength and apparent interfacial shear strength (τ_{app}) of banana fibres (BFs) when embedded in different polymer matrices, specifically polypropylene and bio-based polyamide 11 (PA 11). The analysis revealed that while alkaline treatments generally enhanced the tensile strength of BFs, their impact on τ_{app} varied, with certain treatments improving adhesion in polypropylene. Notably, fibres treated with a harsher solution for an extended period displayed the most significant τ_{app} improvement in polypropylene. In contrast, a single series of untreated fibres embedded in bio-based PA 11 showed a markedly higher τ_{app}, highlighting the matrix's influence on the performance of natural fibre composites. This study demonstrates the critical role of selecting appropriate treatment conditions and matrix materials to optimize the mechanical properties of natural fibre-reinforced composites.

1 INTRODUCTION

In the quest for sustainable alternatives to conventional synthetic fibres like carbon and glass, natural fibres such as flax and hemp have gained prominence due to their environmental benefits, including lower carbon dioxide emissions for their processing and good tensile properties. However, their cultivation can compete with food production, necessitating the exploration of more sustainable sources. BFs, mechanically extracted from waste banana plants stem, offer a promising alternative. Accounting for 60 % of the plant's mass, the stems are typically discarded after harvesting the banana bunch, contributing to approximately 300 million tons of agricultural waste annually [1–3]. According to literatures, BFs, characterized by a cellulose content of 60-65 %, a density of 1.35 g/cm³, and tensile strength ranging between 529-914 MPa, present a viable option for composite reinforcement, as they are low-cost, renewable, and biodegradable [4]. Sodagar et al. conducted research on the incorporation of banana fibres (BFs) into polylactic acid composites, observing a notable improvement in mechanical performance. Their findings indicate that composites made exclusively with banana fibres outperform those made from hybrid composites that combine flax, hemp, and banana fibres [5]. Pilien et al. explored the use of banana fibres in geopolymer-based mortar, optimizing the mix ratios for improved properties [6]. Additionally, the work of Nasr, Bekraoui et al. delved into the extraction and utilization of banana fibres as reinforcing materials for composites, highlighting their practical applications and benefits [7]. Complementing these studies, Badanayak et al. published a critical review on the extraction, characterization, and surface modification of banana pseudo-stem fibres, shedding light on the technological and structural properties essential for enhancing composite materials [8].
Traditionally, natural fibres are treated with industrial chemicals such as sodium hydroxide (NaOH) to improve adhesion with hydrophobic matrices by enhancing the fibres' surface roughness and exposing more cellulose. This

process modifies the fibre structure for better mechanical interlocking and compatibility with polymer matrices. However, environmental concerns over the use of NaOH have led to interest in alternative treatments using sustainable resources. Wood ash, a by-product of wood combustion, has emerged as an eco-friendly option, offering a way to recycle waste while enhancing fibre properties. Previous studies have shown that wood ash treatment can improve the mechanical qualities of natural fibres, making them more suitable for composite applications [9,10].

Table 1. Mechanical properties of different fibre materials [4,11–14]

Fibre	Tensile Strength [MPa]	Young's Modulus [GPa]	Elongation at break [%]	Density $[\frac{g}{cm^3}]$
E-glass	3445	72.3	4.8	2.58
Flax	343 - 2000	27.6 - 103	1.2 - 3.3	1.4 - 1.5
Hemp	270 - 900	23.5 - 90	1 – 3.5	1.4 – 1.5
Banana	529 - 914	12 - 43	1.5 - 9	1.35

This study addresses the identified gaps by exploring sustainable treatment alternatives, focusing on wood ash as a viable option for enhancing the mechanical properties of BFs. It compares the effects of different treatment durations and concentrations on both tensile strength and interfacial shear strength across PP and PA11 polymer matrices. By systematically analysing these factors, the research aims to optimize treatment protocols that improve the performance and sustainability of BF reinforced composites.

2 Materials

2.1 Banana fibre

For this study, BFs were sourced from *Musa Acuminata ('Dwarf Cavendish')* cultivated in Mauritius. The fibres were extracted from the pseudo-stem sheaths using a BF extraction machine manufactured by Riddhi Enterprise, India, located at the University of Mauritius. Following extraction, the fibres underwent a two-stage drying process: initially air-dried for 24 hours and subsequently oven-dried for another 24 hours. To assess the impact of drying methods on the mechanical properties of the fibres, one set of fibres (S3_AD) is entirely air-dried to serve as a comparative study of the effects of oven drying versus air drying. Additionally, to evaluate the influence of chemical treatment on the mechanical properties of fibres in both wet and dry states, another series of fibres was treated with a wood ash solution immediately after extraction before being oven-dried (S2WT_P2H3).

2.2 Wood ash treatment

Wood ash powder was sourced from a local wood-fired pizza outlet. The as-received wood ash contained extraneous materials, such as unburnt wood and large charcoal pieces. The latter were removed by a sieving process. Tap water was then used to prepare alkaline solutions having pH levels of 12.4 and 13.5 (series including P1 and P2, respectively), achieved by adjusting the concentration of wood ash. Given the natural variability of wood ash composition, the solutions were not prepared based on a fixed weight percentage of wood ash to water. Instead, wood ash was added until the desired pH was reached. After mixing, the solutions were left to rest for 24 hours and subsequently filtered using standard filter paper. The pH of each solution was subsequently measured using a Cyberscan pH meter from Eutech Instruments. BFs were submerged in two different alkaline solutions prepared at a 1:30 fibre to liquor ratio, and subjected to two distinct soaking periods of 3 hours and 24 hours (series included H3 and H24), respectively. These conditions created four experimental series, differentiated by two pH levels and two respective fibre soaking durations. Following the soaking process, the fibres were neutralized by immersing

them in tap water at a fibre to water ratio of 1:100. The neutralization process was completed by adding a few drops of 80 % acetic acid to the solution, until a neutral pH was obtained. The fibres were immersed in this neutralized solution for 5 minutes, followed by a fresh water rinse. The pH of the fibres was tested after the rinsing process using litmus paper to ensure pH neutrality. The fibres were then initially air-dried for 24 hours and subsequently oven-dried at 60 °C for an additional 24 hours.

2.3 Polymer matrix

For the pull-out test, Tepex polypropylene (PP) granulate, supplied by Bond-Laminates GmbH, was utilized to assess the mechanical adherence of the BFs. As a point of comparison, a 100 % bio-based Polyamide 11 (PA 11), provided by Arkema Group was also employed. This allowed for the evaluation of different material interactions and the comparative analysis of synthetic versus bio-based polymers in terms of fibre bonding effectiveness and mechanical properties. Moreover, the apparent interfacial shear strength of untreated flax and hemp fibres embedded in the polypropylene, which were tested in a previous study at ITA are also compared with BFs [15].

Table 1: Annotation of series

Series	**Fibre**	**Treatment pH**	**Treatment duration**	**Pull-out test matrix**
S3	Untreated BF	-	-	PP
S3_P1H3	Treated BF	pH of 12.4	3 hours	PP
S3_P2H3	Treated BF	pH of 13.5	3 hours	PP
S3_P1H24	Treated BF	pH of 12.4	24 hours	PP
S3_P2H24	Treated BF	pH of 13.5	24 hours	PP
S3_PA11	Untreated BF	-	-	PA 11
Flax [15]	Untreated flax fibre	-	-	PP
Hemp [15]	Untreated hemp fibre	-	-	PP
Special tests*				***Characteristic***
S3_AD	Untreated BF	-	-	Entirely air-dried BF
S3WT_P2H3	Treated BF	pH of 13.5	3 hours	BF treated directly after extraction

*Only for tensile test

3 Experiments

3.1 Single fibre tensile test

Tensile tests were carried out on single BFs according to ASTM C1557 standard using a Testometric M 500-50AT universal testing machine located in the Materials Engineering laboratory at the University of Mauritius. The tensile testing machine was equipped with a 10 kgf load cell and set to a cross-head speed of 5 mm/min. Each fibre sample was clamped in the pneumatically actually rubber padded grippers with a gauge length of 25.4 mm. Validity of each test was ensured by requiring that fibres fracture near the midpoint, away from the grippers, and within 30 seconds. The protocol aimed to accumulate a minimum of 50 valid tests per series to establish a reliable dataset.

To determine the specific tensile strength of the BFs, microscopic images of the fracture areas were captured using a Digimicro-Profi USB microscope. Consistent with findings by Raghoo et al. [10], which reported the fibres' cylindrical morphology, the diameters at the fracture points were measured at four distinct locations using ImageJ software. These measurements allowed for precise calculation of the cross-sectional areas. Tenacity, defined as the

maximum breaking force divided by the calculated cross-sectional area, was then computed. This meticulous process of validation ensured that the collected data were not only statistically robust but also accurately reflected the mechanical properties of the fibres.

3.2 Pull out test

The interaction between fibres and matrix materials is crucial for reinforcing composites, where force transfer was dependent on effective adhesion. Recognizing the complexity of this interaction and the lack of standardized testing methods, a significant development occurred in 2022. Textechno Herbert Stein GmbH & Co. KG in collaboration with Fraunhofer Institute for Casting, Composite and Processing Technology IGCV and Faserinstitut Bremen e.V., established DIN SPEC 19289. This preliminary standard, though not yet part of the formal German standards, lays the groundwork for defining uniform bonding test methodologies, aiming to facilitate consistent and comparable evaluations of fibre-matrix adhesion.

The fibres were conditioned for 24 hours in an EN ISO 139 standard environment. In accordance with DIN SPEC 19289, for the preparation of samples, fibres at least 5 mm in length were embedded in a polymer matrix using the Fimabond device from Textechno Herbert Stein GmbH & Co. KG located at Institut für Textiltechnik (ITA) of RWTH Aachen University (see Figure 1 a). This process was carefully controlled with an embedding speed of 560 µm/min and an embedded length of 280 µm to ensure accurate placement without causing deformation. The temperatures were set to 240 °C for PP and 190 °C for PA 11, based on preliminary trials, to achieve optimal flow conditions for embedding. The diameter of each fibre was measured prior to embedding to facilitate subsequent calculations of shear strength. At least 10 samples were prepared per series to maintain robustness in data collection.

Following the sample preparation, the pull-out tests were conducted using the Favimat+ testing apparatus from Textechno Herbert Stein GmbH & Co. KG located at ITA (see Figure 1 c). The procedure resembles a conventional tensile test where the fibre ends protruding from the polymer matrix were clamped and pulled at a constant speed of 0.1 mm/min until detachment. The force required to extract the fibre was meticulously recorded, and a force-path diagram was generated. The apparent interfacial shear strength (τ_{app}), which was the maximum force per unit contact area was calculated using the equation 1, where F_{max} represents the maximum force applied, d_f the diameter of the fibre, and L_e the embedded length. This process not only measures the bond strength but also helps compare the adhesion properties across different fibre-matrix combinations.

$$\tau_{app} = \frac{F_{max}}{\pi \times df \times \mathrm{l_e}} \qquad \text{Equation (1)}$$

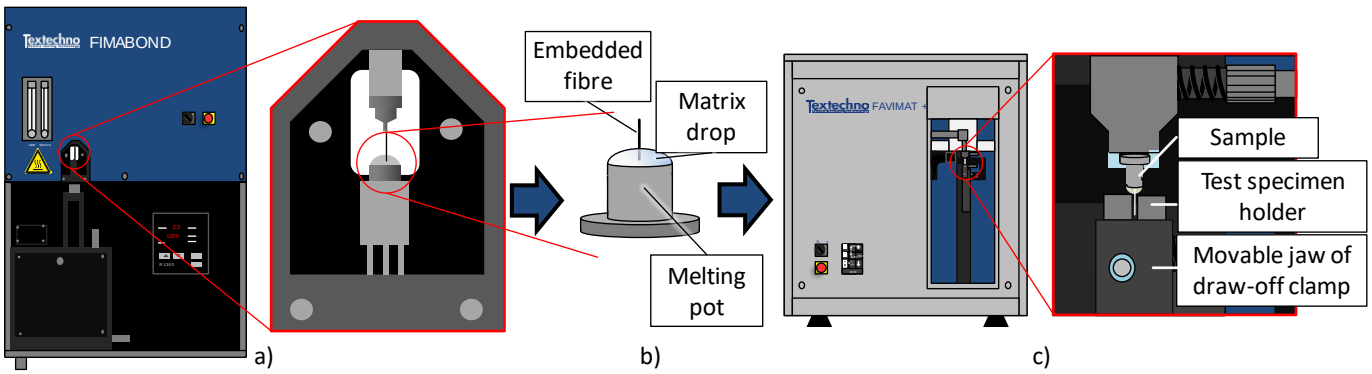

Figure 1: a) Pull-out sample preparation machine FIMABOND; b) Pull-out test sample; c) Pull-out testing machine, FAVIMAT

4 Results and discussion

4.1 Tensile test results

This study evaluated the tensile strength of both treated and untreated BFs, as shown in the box plots illustrated in Figure 2. For each series, 50 valid results have been considered, with only outlier points displayed to simplify the visual representation. Annotations and their explanations are detailed in Table 1. A significant variation is noted between oven-dried and air-dried BFs; oven-dried fibres (S3) had an average tensile strength of 334 MPa, while air-dried fibres exhibited a higher average strength of 415 MPa, a 25 % increase. This difference largely results from the drying methods. Air-drying, which occurs at ambient temperatures, is a gentler process that gradually removes moisture without disrupting the cellulose's internal hydrogen bonds, thus preserving the fibres' structural integrity. Conversely, oven-drying at higher temperatures accelerates moisture loss, inducing internal stresses and potential micro-cracking that weaken the fibres. This contrasts with Bai et al.'s findings, where bamboo fibres increased in strength post oven-drying [16]. The discrepancy lies in the compositional differences; BFs have lower lignin and hemicellulose content, making them less prone to moisture-related softening effects seen in lignin-rich bamboo. Additionally, the slower pace of air-drying allows BFs to maintain a more natural alignment of cellulose microfibrils, potentially enhancing their load capacity. Enzymatic activities at lower drying speeds might also reinforce the cellulose structure, preserving or strengthening the intermolecular hydrogen bonds, effects likely reduced in the rapid, high-temperature environment of oven drying. In the treated series, S3WT_P2H3, involving wet BFs treated with a pH 13.5 alkaline solution for 3 hours, showed the highest tensile strength at 558 MPa with a coefficient variation of 70 %. However, the same series treated dry (S3_P2H3) showed a 12 % lower strength of 490 MPa but displayed a less coefficient variation of 34 %, indicating higher consistency. This suggests that while wet state treatment can boost tensile strength, it may also introduce variability in fibre quality. The most consistent results are seen in dry fibres treated with the same pH 13.5 solution for 3 hours, achieving a strength of 490 MPa, 47 % higher than untreated BFs. Extending the treatment to 24 hours (S3_P2H24) decreased strength by 12 %, likely due to prolonged alkaline exposure deteriorating the cellulose structure. Conversely, treating fibres with a milder pH solution for 24 hours (S3_P1H24) increased strength by 10 % compared to a shorter 3-hour treatment (S3_P1H3), suggesting that milder conditions require longer exposure to effectively enhance crystallinity and remove wax, impurities and the amorphous components.

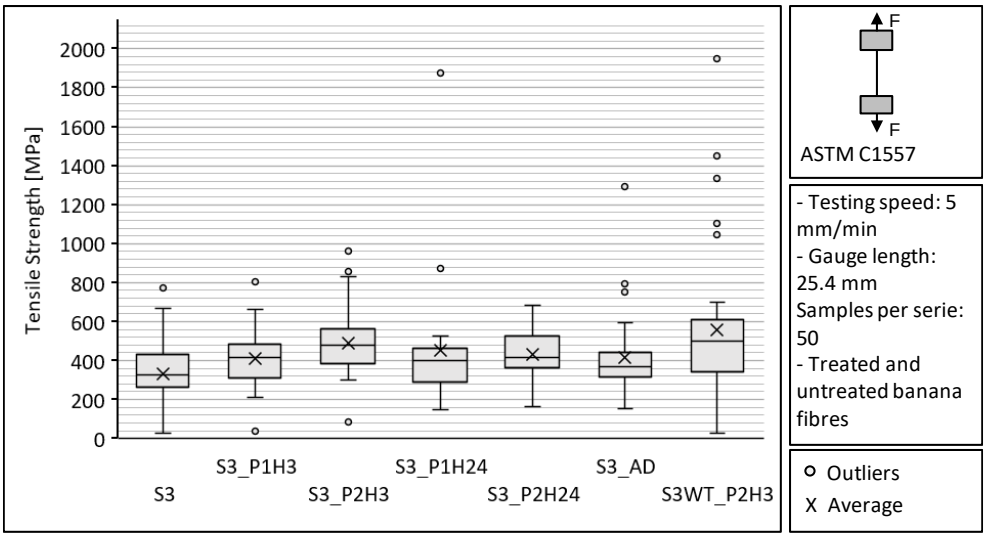

Figure 2: Tensile strength of treated and untreated BFs (only outlier points are shown)

4.2 Pull-out test results

Untreated BFs (S3) embedded in PP showed a τ_{app} of 8 MPa, serving as a baseline for comparison. When treated with a milder wood ash solution for 3 hours (S3_P1H3), the τ_{app} unexpectedly decreased to 5.90 MPa. This reduction suggests that the alkaline treatment, while beneficial in modifying the fibres' tensile properties, might not favourably alter the surface for adhesion to the inherently hydrophobic PP. This decrease could be due to the treatment increasing the fibre's hydrophilicity through exposure of more hydroxyl groups, which poorly interfaces with the PP matrix. In cases where the treatment intensity is increased or extended, the τ_{app} responses varied. For fibres treated with a higher pH solution for the same duration (S3_P2H3), τ_{app} was measured at 8.04 MPa. Given the coefficient of variation for untreated fibres at 27% and for S3_P2H3 at 29%, this result is statistically comparable to that of untreated fibres, essentially showing no significant improvement from the treatment. However, extending the milder solution treatment to 24 hours (S3_P1H24) yielded a modest τ_{app} increase to 6.80 MPa as compared to the treatment for a duration of 3 hours (S3_P1H3), suggesting that longer durations might start to improve fibre surface compatibility with PP, but not enough to surpass the performance of untreated fibres. A notable improvement is observed with the harsher solution treatment extended to 24 hours, achieving a τ_{app} of 10.07 MPa. This enhancement implies that prolonged and intense alkaline treatment may facilitate deeper chemical modifications and surface roughening due to the removal of a significant amount of the surface hemicellulose and to some extent the lignin component, leading to higher degree of defibrillation. Thus this effectively enhances the fibre-matrix bonding, and overcoming the hydrophilic-hydrophobic barrier to some extent. When untreated fibres were embedded in bio-based PA 11, the τ_{app} soared to 17.18 MPa, underscoring a markedly better compatibility. This pronounced increase likely results from more favourable chemical interactions and enhanced mechanical interlocking facilitated by the similar nature of the fibre and matrix materials.

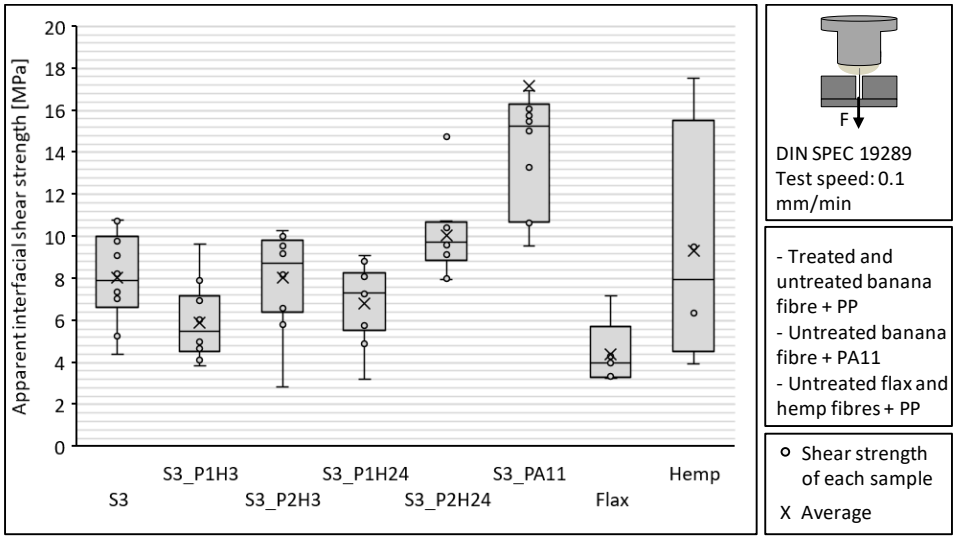

Figure 3: Apparent interfacial shear strengths (τ_{app}) of BFs embedded in PP; S3_PA11, Flax and hemp as references

5 Conclusion

This study highlights the significant impact of alkaline treatment on the mechanical properties of BFs, focusing on the importance of treatment conditions such as drying method, alkaline solution concentration, and treatment duration. Optimized treatment parameters proved essential for enhancing BFs' tensile strength, maintaining structural integrity, and performance consistency. Notably, air-drying followed by a 3-hour treatment with a pH 13.5

wood ash solution showed potential to elevate the tensile strength of BFs to 650 MPa. However, the achieved strength does not match the levels documented in the literature, likely due to inconsistencies in the fibre extraction process performed by untrained personnel, leading to variable fibre quality. Moreover, the study reveals that the effectiveness of alkaline treatments on fibre-matrix adhesion varies significantly depending on the specific treatment and the type of polymer matrix used. This indicates that treatment protocols must be precisely tailored to the unique properties of both the fibre and the matrix to optimize bonding and enhance composite performance.

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