

Research Article

# Influence of Wet-Dry Treatment of *Urena Lobata* Fibers Reinforced Cement Boards

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## Abstract

The impregnation of wood in fiber cement boards aims to replace traditional ceiling boards, which are prone to moisture damage over time. This study explores a modified hornification process involving fiber treatment with a solution of hexamine and gallic acid (H/G method) compared to a conventional hornification method (H method), where fibers are soaked in tap water. The objective is to evaluate the influence of this modified process on the morphological, physicochemical, and mechanical properties of *Urena lobata* (UL) bast fibers from the Littoral region of Cameroon. The fibers underwent four wet-dry treatment cycles using both the H/G and H methods. Results revealed significant reductions in water and moisture absorption compared to untreated (UT) fibers. Water absorption decreased from  $227.79 \pm 0.05\%$  (UT) to  $200.34 \pm 0.05\%$  (H) and  $130.37 \pm 0.03\%$  (H/G), while moisture absorption reduced from 9.286% (UT) to 7.03% (H) and 5.854% (H/G). Additionally, an increase in fiber density was observed, rising from  $1.72 \pm 0.012 \text{ g/cm}^3$  (UT) to  $1.78 \pm 0.012 \text{ g/cm}^3$  (H) and  $1.87 \pm 0.04 \text{ g/cm}^3$  (H/G), attributed to the infiltration of hexamine and gallic acid into the fiber cells. Mechanical performance was assessed through flexural and compressive tests after 7, 14, and 28 days of curing. Both elastic modulus and compressive strength improved progressively from untreated fibers through the H method to the H/G method, with increases of 20% and 30%, respectively. These findings demonstrate that the hexamine and gallic acid treatment enhances the effectiveness of the hornification process, significantly improving the water resistance and mechanical performance of the treated fibers.

## Keywords

*Urena Lobata*, Hexamine, Gallic acid, Conventional Hornification, Modified Hornification Treatment

## 1. Introduction

The profitable application of natural lignocellulosic fibers has garnered significant attention from researchers seeking to

integrate these fibers into cement board production. Natural lignocellulosic fibers offer numerous advantages, including

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sustainability, availability, renewability, well-developed extraction technologies, low cost, low density, flexibility, and non-abrasive properties compared to synthetic fibers [1]. Furthermore, their favorable elastic modulus makes them suitable for various composite applications. Despite these benefits, the hydrophilic nature and variability in the physicochemical and mechanical properties of natural fibers remain significant challenges. These limitations are primarily attributed to the complex functional group chemistry, high incidence of structural defects, and variability in composition inherent in lignocellulosic materials [2]. Hemicellulose and cellulose, two major structural components of lignocellulosic biomass, are highly hydrophilic, leading to poor bonding with matrix materials and reduced dimensional stability and durability in both organic and inorganic matrices. Poor resistance to water and moisture is a critical barrier to using wood and lignocellulosic fibers in structural applications [3]. To address these issues, researchers have explored physical and chemical modifications to improve the bonding characteristics and water resistance of natural fibers. Chemical treatments such as oxidation, esterification, salinization, and controlled alkali or acid pre-treatments, along with deposition of thin hydrophobic layers, have shown varying degrees of success [1]. Physical methods, including plasma treatments, hornification, and impregnation, have also been studied extensively. Hornification, in particular, has demonstrated significant improvements in water resistance and matrix bonding by reducing the water absorption capacity of fibers [4]. Recent studies have highlighted the positive effects of hornification treatments on the mechanical, thermal, and water-resistance properties of natural lignocellulosic fibers. For example, [5] observed water retention reductions of 56.3% for okra fibers and 18.3% for raffia fibers. Similarly, [6, 7] reported reductions in water retention values for unbleached eucalyptus pulp (24%) compared to bleached pulp (10%). [8] emphasized the durability

and mechanical benefits of hornified vegetable fibers, such as softwood kraft pulp and cotton linters, in cement mortar composites. In addition, research on various cellulose fibers, including bagasse, wheat, and eucalyptus, demonstrated that bagasse fibers significantly improved the flexural properties of fiber cement boards, with a 50% increase in flexural strength compared to control specimens [9, 10] further illustrated the advantages of hardwood short fiber pulp (e.g., eucalyptus) as an alternative to softwood pulp and polymer fibers in cement-based materials. The present study builds on these findings by evaluating *Urena lobata* (UL) fibers, which exhibit promising mechanical properties and are abundant in the Littoral region of Cameroon. A systematic characterization of their proximate composition and response to a modified hornification treatment was conducted. This novel hornification method integrates advancements in tannin-hexamine-based treatments [11] to enhance fiber performance, aiming to address the limitations associated with natural fiber-based composites.

## 2. Materials and Methods

### 2.1. Raws Materials

Stem of *Urena Lobata* was collected in the Littoral region of Cameroon which has an average temperature of 26.9 °C the plants was identified in the mechanical department of the higher technical teacher training college (HTTTC Douala) Douala Cameroon and the bast fiber extracted from the bark of stem by running fresh water body immersion for 15 days. The fibers were separated by wafting manually after the retting treatment as shown in Figure 1 below.



**Figure 1.** Illustration of the *Urena Lobata* fiber extraction process: (a) fresh UL stem, (b) UL stem cross-section, (c) UL stem bark, (d) UL fiber soaking, (e) UL fiber separation, and (f) UL fiber.

### Fiber Treatment

The wet-dry cycle treatment was done using two methods that is the conventional and hexamine and gallic acid methods and it was done according to the methodology developed by ([12, 13]). 200 g of raw material was soaked in water which include 20.19 grams of hexamine and 10 grams of gallic acid both mixed in 2 liters of tap water; on the others hand, another 200 g of raw material was mixed separately inside two liters of tap water; this case was known as the conventional method; the fiber was held at 22 °C and soaked for 3 hours in water for it to reach its maximum water absorption capacity. The fibers were removed and dried at a temperature of 70 °C in an oven. After 12 hours of drying, the sample was removed and cooled to 22 °C to avoid possible thermal shock of the fibers. This procedure was repeated 4 times [12].

## 2.2. Determination of Proximate Composition

According to preliminaries study [14], The fibers of the treated and untreated UL was dried in an oven at  $100 \pm 2$  °C until they have a constant weight. Proximate composition was determined for treated and untreated fibers to constant weight, and the fibers was dewaxed in 200 ml 70% (v/v) ethanol, with a fiber to solvent ratio of 1: 100 ( $\text{g L}^{-1}$ ) using a Soxhlet extractor for 6 hours to remove all the oil and wax [15]. Lignin was determined by the klaxon methods [14] after crushing the extracted fibers with dichloromethane before being hydrolysed in a 72% solution of sulphuric acid. [16] The Neutral detergent fibers method was used to determine the ash content of the fibers.

## 2.3. Scanning Electron Microscopy Analysis

The surfaces of the samples were coated with a thin layer of gold using an AGAR AUTO SPUTTER COATER to ensure electrical conductivity for SEM observation. The surface of the samples was observed using the Carl Zeiss AG - Supra 25 Scanning Electron Microscope (SEM) equipped with an EBSD sensor. Several images were taken with an acceleration voltage of 20 kV and magnifications ranging from x100-2500.

## 2.4. Moisture Content

The moisture content of the treated and untreated fibers was determined using the gravimetric method in a thermo scientific oven. 6.512 g of UL fibers was weighed and dried at 105 °C in an oven for 24 hours. The samples were weighed immediately on removal from the oven and stored in a desiccator.

The calculations were done as follows:

$$\text{Moisture content (\%)} = \frac{(\text{weight of dry sample})}{(\text{weight of wet sample})} \times 100 \quad (1)$$

## 2.5. Density Measurement

The density ( $\rho$ ) of UL fiber was obtained according to the method reported by [17]. Ten (10) samples were used. The fibers were grinded using a mortar and pestle to a particle size of 180  $\mu\text{m}$ . The weight of an empty graduated cylinder was noted and a given quantity of powder put in it. It was then stacked into the cylinder to eliminate the voids between the fibers using a broom. The volume of the fibers in the cylinder,  $V_f$  was recorded and their total mass taken.

The mass of fibers in the cylinder was determined using the formula below:

$$m_f = m_{c+f} - m_c \quad (2)$$

Where  $m_f$  = mass of fiber,  $m_c$  = mass of cylinder and  $m_{c+f}$  = mass of cylinder + fiber;

The density is given by the expression below;

$$\rho = \frac{m_f}{V_f} \quad (3)$$

## 2.6. Water Absorption

The UL water absorption before and after treatment was performed using the gravimetric analysis method. An initial weight of 2 g of fibers was oven dried at 60 °C for 24 hours and immersed into distilled water at room temperature. Five samples were prepared and the mean percentage of water absorption was calculated using equation (4) below. The weight was recorded until stability was achieved [18].

$$Abs = \frac{M_1 - M_0}{M_0} \times 100 \quad (4)$$

## 2.7. FTIR-ATR Spectrometry

FTIR analysis is performed using a SHIMADZU IRAffinity-1 CE Shimadzu instrument equipped with a diamond crystal operating in ATR mode. The infrared spectrum was recorded from 400 to 4000  $\text{cm}^{-1}$  by accumulation of 32 scans with a resolution of 4  $\text{cm}^{-1}$ .

## 2.8. XRD (Degree of Crystallinity)

The crystallinity of UL treated and untreated fibers was determined using a Rigaku Miniflex 600 diffractometer, model RU 200 B (Rigaku Corporation, Japan) and the procedure reported by (Stanislas et al., 2020). The evaluation of the crystallinity index (Crl) was carried out by using the empirical method (Segal et al., 1959) according to equation (5).

$$\text{Crl} = 100 \times \frac{I_{002} - I_{am}}{I_{002}} \quad (5)$$

Where,  $I_{002}$  is the value of the intensity of (002) reflection.

tions corresponding to  $22^\circ - 23^\circ$  ( $2\theta$ ), and  $I_{am}$  diffraction intensity corresponding to  $18^\circ - 19^\circ$  ( $2\theta$ ). The values were obtained directly from the XRD diffractogram of TP fibers [19].

## 2.9. Determination of Porosity of the UL Fibers

Porosity is the set of voids (pores) in a solid material, these voids are usually filled with fluids (liquid or gas). It is a physical quantity between 0 and 100%, conditioning for a composite material the retention capacities of a substrate. It has an influence on the physical properties of the composite material. The porosity coefficient of the different samples submitted to the study is determined by the Equation (6) below [20].

$$n = \frac{M_s - M_i}{M_i} \times \rho \times 100 \quad (6)$$

With  $m_i$ : Initial sample mass and  $m_s$ : Sample mass at the

time  $t$  of saturation (in g),  $\rho$ : density's sample,  $n$ : porosity coefficient.

## 2.10. Formulation Preparations of UL Fibers

The samples formulations are shown on Table 1 below. The design mix proportion selected for the preparation of fiber cement blocks was 3: 1 (cement: sand) with a constant water cement ratio of 0.50 following the specification of ([21, 22]). To replace cement by weight, a varying percentage of urena Lobata fibers (0%, 1%, 1.5% and 2.5%) was added. Three different methods of mixing were evaluated (the cement/fibers bundles, the conventional and the modified hornification method). Based on same proportion of composite matrix, 1 fiber length of 1 mm was chosen, this is because for a micro fiber of longer length is difficult to obtain a random sampling.

**Table 1.** Design mix proportions of fibers cement board.

| Cement to sand | Water to cement ratio | Coir fibers (by weight) | Fiber                                      |
|----------------|-----------------------|-------------------------|--------------------------------------------|
| 3:1            | 0.50                  | 0%                      | H. H/G, H. HO <sub>2</sub> , and untreated |
| 2.85:1         | 0.50                  | 1%                      | H. H/G, H. HO <sub>2</sub> , and untreated |
| 2.80:1         | 0.50                  | 1.5%                    | H. H/G, H. HO <sub>2</sub> , and untreated |
| 2.70:1         | 0.50                  | 2.5%                    | H. H/G, H. HO <sub>2</sub> , and untreated |

### 2.10.1. Fibers Cement Board Mixture

In order to obtain maximum compaction of the beams for maximum strength, the samples were mix for 7 minutes immediately from the moment water was poured into the mold. Stirring was done for 5 minutes to ensure homogeneity in the mixture. After the seventh minute, the composites were viscous for pouring into the mold, and after 48 hours, demolding was done, while samples were identified following the wt.% fibers dosage in the cement composite.

### 2.10.2. Casting, Curing and Testing of Samples

The mold for compressive and flexural tests we used for the manufacturing of the composite block was dimensioned 4 cm x 4 cm x 4 cm were cast for compressive strength determination and prismatic specimens of 4 cm x 4 cm x 16 cm were cast for flexural strength respectively BS EN 1015-11 (2007). Each mold was lubricated as shown in Figure 2 below.



**Figure 2.** Oiling of the molds.



**Figure 3.** Molded samples.

### 2.10.3. Flexural Characterization of Fibers Cement Board

A three-point bending test was conducted on all the composites samples with the help of the flexural machine (RMU serial 1461288). The maximum stresses absorbed were noted and recorded as shown on the figure below. [14]. 80 samples were tested.



Figure 4. Flexural Testing Machine.

### 2.10.4. Compressive Characterization of Fibers Cement Board



Figure 5. Compressive strength machine.

The compressive test was conducted on all the composite samples with the help of compressive machine, RMU serial 121288 at the laboratory of civil engineering in Government

Technical High School BAMENDA. An increasing compressive load was applied by the specimen until failure occurred to obtain the maximum compressive load. The specimen dimension was taken before the testing. The testing was carried out for 7 days, 14 days and 28 days specimen after curing. [14, 23].

## 3. Results and Discussion

### 3.1. Proximate Composition of Fibers

The proximate composition of the fiber samples is presented in Table 2 below. The *Urena lobata* (UL) fibers treated with hexamine and gallic acid exhibited a lower extractives content compared to untreated fibers, indicating improved fiber specifications. This suggests that the additives used during the hornification process significantly enhanced the surface properties of the fibers. The modified treated *Urena lobata* fibers demonstrated a higher cellulose content (74.14%) compared to untreated fibers (71.2%), consistent with prior findings [14]. This higher cellulose content is indicative of superior mechanical properties. Conversely, the lignin content of the treated and conventionally treated fibers was comparable (13.2% and 13.4%, respectively), while the untreated fibers had a higher lignin content (14.6%). These lignin levels are within the range reported for flax fibers (14-19%) by ([24-26]) but slightly lower than those noted in more recent studies ([27, 28]). In addition to cellulose, the treated fibers exhibited higher hemicellulose content and lower ash content, suggesting improved thermal and mechanical behavior. The reduced ash and extractives content enhances the fibers' combustion characteristics, making them more suitable for energy-related applications. However, variations in lignin content did not yield significant insights regarding lignin-related recalcitrance. Recent studies have corroborated these observations, noting the importance of chemical modifications in improving the structural properties of natural fibers for advanced applications ([29, 30]). Such treatments not only enhance cellulose accessibility but also reduce undesirable components, thus optimizing the fibers for composite reinforcement and energy applications.

Table 2. Chemical composition of plant fibers.

| Fibers                    | Cellulose (wt%) | Hemicellulose (wt%) | Lignin (wt%) | Pectin (wt%) | Extractives (wt%) | Ash content (wt%) | References |
|---------------------------|-----------------|---------------------|--------------|--------------|-------------------|-------------------|------------|
| Flax                      | 60 - 81         | 18.6 - 20.6         | 14 - 19      | 2.3          | -                 | -                 | ([24-26])  |
| Hemp                      | 70 - 74         | 17.9 - 22.4         | 3.7 - 5.7    | 0.9          | -                 | -                 | [25]       |
| Eucalyptus bleached kraft | 89              | -                   | 0.5          | -            | -                 | -                 | [31]       |

| Fibers              | Cellulose (wt%) | Hemicellulose (wt%) | Lignin (wt%) | Pectin (wt%) | Extractives (wt%) | Ash content (wt%) | References    |
|---------------------|-----------------|---------------------|--------------|--------------|-------------------|-------------------|---------------|
| <i>Urena Lobata</i> | 73.2            | 10.6                | 16.2         | -            | -                 | -                 | [14]          |
| Coconut             | 36 - 43         | 0.15 - 14.7         | 41 - 45      | 3 - 4        | -                 | -                 | ([25, 26])    |
| RC                  | 65.15           | 7.42                | 16.2         | 3.45         | 5.22              | 0.42              | [32]          |
| OPEFBst             | 35              | 23                  | 28           | -            | 14                | 10.58             | [33]          |
| UL Untreated        | 71.2            | 10.2                | 14.6         | 1.2          | 0.46              | 2.34              | Present study |
| UL H (Conventional) | 70.12           | 11.43               | 13.2         | 1.3          | 1.66              | 2.29              | Present study |
| UL H. H/G           | 74.14           | 11.59               | 13.4         | 0.5          | 0.36              | 0.01              | Present study |

### 3.2. Moisture Content

Determining water content provides critical insights into the moisture absorption properties of fibers. The moisture content of the modified treated, conventionally treated, and untreated *Urena lobata* (UL) fibers is shown in Table 2. The treated UL fibers exhibited the lowest moisture content (5.854%), compared to the conventionally treated fibers (7.028%) and the untreated fibers (9.286%). This moisture content is lower than the values reported for UL fibers by Ghabo et al. (2024) and is also significantly less than that of *Sida stipulata* fibers (8.6%). These results indicate that the hornification treatment with additives plays a substantial role in reducing moisture content. The reduction in moisture content can be attributed to the sensitivity of UL fibers to chemical modifications, specifically the reduction of free hydroxyl groups presents in lignin and hemicellulose ([34, 35]). These hydroxyl groups are responsible for water absorption, and their reduction during the treatment process enhances the hydrophobicity of the fibers. Table 2 also demonstrates that the moisture content values of the studied fibers align closely with those reported in recent literature. For instance, recent studies by [36, 37] have similarly reported significant reductions in moisture content following chemical treatments, further corroborating the effectiveness of hornification in enhancing fiber performance.

### 3.3. Density Measurement

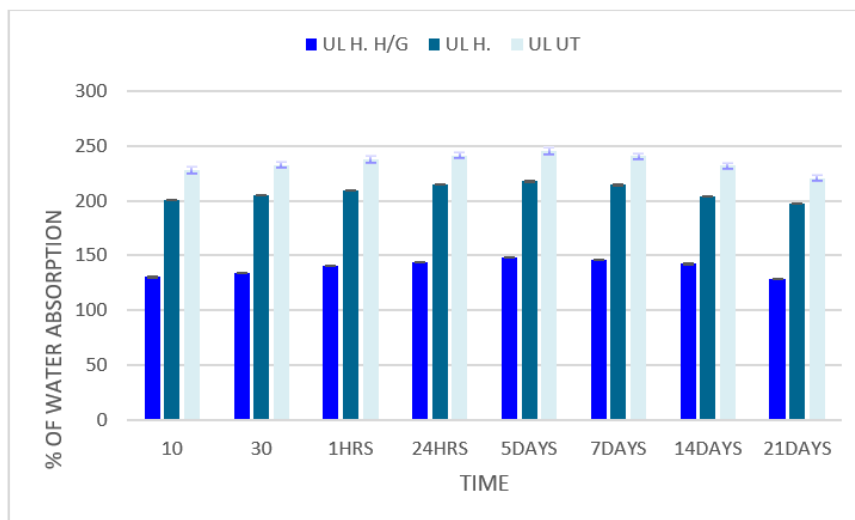
Lignocellulosic fibers are predominantly composed of hemicellulose, with lignin and crystalline cellulose playing critical structural roles. Based on this understanding, the density of untreated, conventionally treated, and additive-treated *Urena lobata* (UL) fibers was measured. The results indicate that the density of modified treated UL fibers increased to 1.83 g/cm<sup>3</sup>, compared to conventionally treated fibers (1.79 g/cm<sup>3</sup>) and untreated fibers (1.72 g/cm<sup>3</sup>). This density is significantly higher than the values reported for

untreated *Urena lobata* and *Sida stipulata* fibers by [38] and is slightly greater than the density of TC fibers noted by [39]. The increase in fiber density is attributed to several factors, including the type of chemicals used, the extraction process, and the developmental zone of the fibers, as suggested by [38]. The specific increase observed in the treated UL fibers is primarily due to the infiltration of hexamine and gallic acid into the cell walls, which densifies the fibers by reducing porosity and increasing material compaction. These findings are consistent with some others studies, such as those by [27, 36], which highlight those chemical treatments significantly improve fiber density by modifying microstructural properties. Enhanced density is a desirable characteristic, as it contributes to improved mechanical performance and durability in composite applications.

### 3.4. Water Content and Water Absorption

The results in Table 3 below detail the effect of wet-dry treatment on *Urena lobata* (UL) fibers. The water absorption of fibers treated with additives was significantly reduced compared to conventionally treated and untreated fibers. The treated fibers showed the lowest water absorption rate (130.37%), followed by the conventionally treated fibers (200.34%), and the untreated fibers (227.79%). The high-water absorption rate of untreated UL fibers, while substantial, is comparable to findings in untreated UL fibers reported by [38] and remains lower than that observed in jute fibers ([34, 40, 41]). The reduction in water absorption of treated fibers can be attributed to the collapse of the fiber wall during the drying process, as well as the closure of pores within the fiber wall due to decreased swelling capacity [42, 43]. Additionally, the modification of the COOH functional group during treatment, as described by [12], increases the hydrophobicity of lignin, further reducing water uptake. The reduction in water retention is also influenced by factors such as fiber shrinkage, semi-irreversible pore closure, and increased crystallinity. Studies by [29, 36] confirm that such chemical and physical changes induced during treatment

significantly enhance fiber hydrophobicity, improving their sorption. suitability for applications requiring reduced moisture ab-



**Figure 6.** Percentages of water absorption of UL fibers.

**Table 3.** Physical properties of *Urena Lobata* fibers compared with some plant fibers.

| Fiber               | Moisture content (%) | Density (g cm <sup>-3</sup> ) | Water absorption (%) | References     |
|---------------------|----------------------|-------------------------------|----------------------|----------------|
| Flax                | 12                   | 1.5                           | 136 ± 25             | [40, 41]       |
| Hemp                | 6.2 - 12             | 1.15 ± 1.45                   | -                    | [34, 44]       |
| Jute                | 12 - 13.7            | 1.3 - 1.41                    | 281                  | ([34, 40, 41]) |
| Urena Lobata        | 10.5                 | 0.23463                       | 228.58               | [38]           |
| Urena Lobata        | -                    | 1.72                          | -                    | [14]           |
| RC                  | 9.37                 | 0.757 ± 0.08                  | 198.17 ± 20.23       | [45]           |
| Sida Stipulate      | 8.6                  | 0.20114                       | 220.95               | [38]           |
| UL Untreated        | 9.286                | 1.72±0.012                    | 227.79±0.05          | Present study  |
| UL H (Conventional) | 7.03                 | 1.78±0.012                    | 200.34±0.05          | Present study  |
| UL H. H/G           | 5.854                | 1.870.04                      | 130.37±0.03          | Present study  |

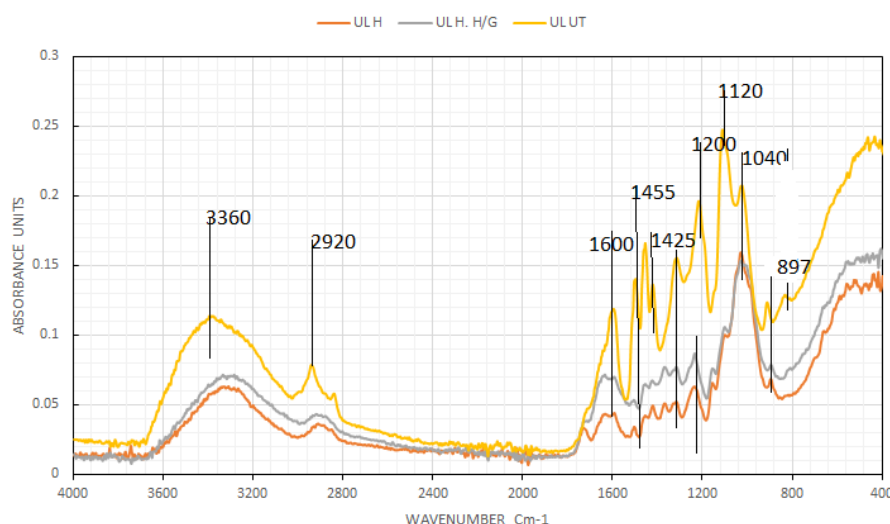
### 3.5. FTIR-ATR Spectrometry Results for Fibers

FTIR spectroscopy was employed to analyze functional groups in *Urena lobata* (UL) fibers, identifying changes in chemical composition resulting from wet-dry treatment and chemical modifications. This technique highlights groups such as hydroxyl, carbonyl, vinyl, and ketone groups, which are vital in understanding fiber behavior (Stark & Matuana, 2004). The spectra (Table 3, Figure 3) illustrate differences among untreated, conventionally treated, and additive-treated fibers. A broad peak between 3500-3300 cm<sup>-1</sup> in all spectra

corresponds to the axial vibration of the hydroxyl group in cellulose ([46-48]). Reduced intensity of this peak post-treatment indicates decreased water content in the fibers. The peak at 2920 cm<sup>-1</sup>, attributed to C-H stretching vibrations of methyl and methylene groups (indicative of lignin), diminishes with increased wet-dry cycles, possibly due to the partial removal of amorphous compounds like lignin during the process [2, 49]. Significant variations in the region between 1800 and 1100 cm<sup>-1</sup> highlight chemical modifications. The absence of a sharp peak around 1600 cm<sup>-1</sup> across all treatments reflects the reduction of C=C aromatic ring vibrations [50, 51]. Peaks at 1455 cm<sup>-1</sup> represent C-H deformation

and CH<sub>2</sub> symmetric bending linked to lignin [51, 52]. Similarly, a peak at 1428 cm<sup>-1</sup> corresponds to the crystalline structure of cellulose [53]. The region from 1320 to 1200 cm<sup>-1</sup> indicates CH<sub>2</sub> wagging, characteristic of crystalline cellulose [54]. Peaks at 1120 cm<sup>-1</sup> and 1040 cm<sup>-1</sup> are associated with C-O-C asymmetric stretching in carbohydrates and C-O bond vibrations, respectively [53, 55]. Peaks between 920 and 890 cm<sup>-1</sup> relate to =CH aromatic ring vibrations and C-H defor-

mation on benzene rings [55]. Post-treatment analysis revealed a rougher fiber surface due to the removal of hemicellulose, pectin, and impurities. This increased the interfibrillar region compared to untreated fibers, which maintained smoother surfaces. The roughened surface improves interfacial adhesion with resin and promotes denser lignin packing, leading to enhanced mechanical properties [29, 36].



**Figure 7.** FTIR spectrum of UL fibres.

**Table 4.** FTIR peak position, corresponding chemical functional group, and composition in the UL fibers.

| UL UNTREATED (cm <sup>-1</sup> ) | UL H. H (cm <sup>-1</sup> ) | UL H. H+G ADDITIVE (cm <sup>-1</sup> ) | ASSIGNMENT                                                                                       | REFERENCE |
|----------------------------------|-----------------------------|----------------------------------------|--------------------------------------------------------------------------------------------------|-----------|
| 3600-3200                        | 3600-3200                   | 3600-3200                              | Stretching of OH bonds (cellulose-hemicellulose)                                                 | [46, 48]  |
| 2950-2900                        | 2950-2900                   | 2950-2900                              | Asymmetry vibration of VAS CH <sub>2</sub> characteristics of hemicellulose cellulose and lignin | [(56-58)] |
| 1600                             | 1600                        | 1600                                   | Aromatic ring vibration                                                                          | [50, 51]  |
| 1506                             | 1506                        | 1506                                   | Symmetrical stretching of C=C of lignin                                                          | [32, 59]  |
| 1455                             | 1455                        | 1455                                   | C-H deformation (lignin) and CH <sub>2</sub> symmetric bending (cellulose)                       | [52]      |
| 1428-1417                        | 1428-1417                   | 1428-1417                              | Relates to the crystalline structure of cellulose                                                | [53]      |
| 1200                             | 1200                        | 1200                                   | CH <sub>2</sub> wagging and it is characteristic of crystalline cellulose                        | [54]      |
| 1107-1090                        | 1107-1090                   | 1107-1090                              | C-O-C asymmetric stretching for carbohydrate and cellulose                                       | [53]      |
| 1040                             | 1040                        | 1040                                   | attributed to vibrations of C-O bonds                                                            | [56]      |
| 896                              | 896                         | 897                                    | Symmetric stretching of the C-H and O-H bonds of cellulose.                                      | [32, 52]  |

### 3.6. Morphological Properties of UL Fibres

The SEM images (Figure 4) depict the surface morphology of untreated, conventionally treated, and additive-treated *Urena lobata* (UL) fibers. The surface of the untreated fibers appears smooth, which is indicative of the presence of impurities or cementing substances such as wax, lignin, and hemicellulose. These substances protect fiber strands from moisture loss, a feature commonly observed in plant fibers [60, 61]. The SEM images of conventionally treated UL fibers exhibit a slightly rougher and more wrinkled surface compared to the untreated fibers. This wrinkling can be attributed to the wet-dry cycles of the hornification process. Such surface modifications are known to enhance fiber-cementitious ma-

trix adhesion and improve the mechanical performance of fibers when used as reinforcement, as also reported by [7, 10, 43, 62]. The additive-treated UL fibers display a significantly rougher surface compared to both untreated and conventionally treated fibers. This pronounced roughness results from the hornification process involving wet-dry cycles with additives such as hexamine and gallic acid. The treatment intensifies the collapse of the fiber lumen and reduces both the specific and internal surface areas of the fibers. The rougher fiber surface promotes mechanical interlocking within the matrix, thereby enhancing interfacial adhesion and potentially improving composite performance. Similar findings have been documented by [7, 62, 63].

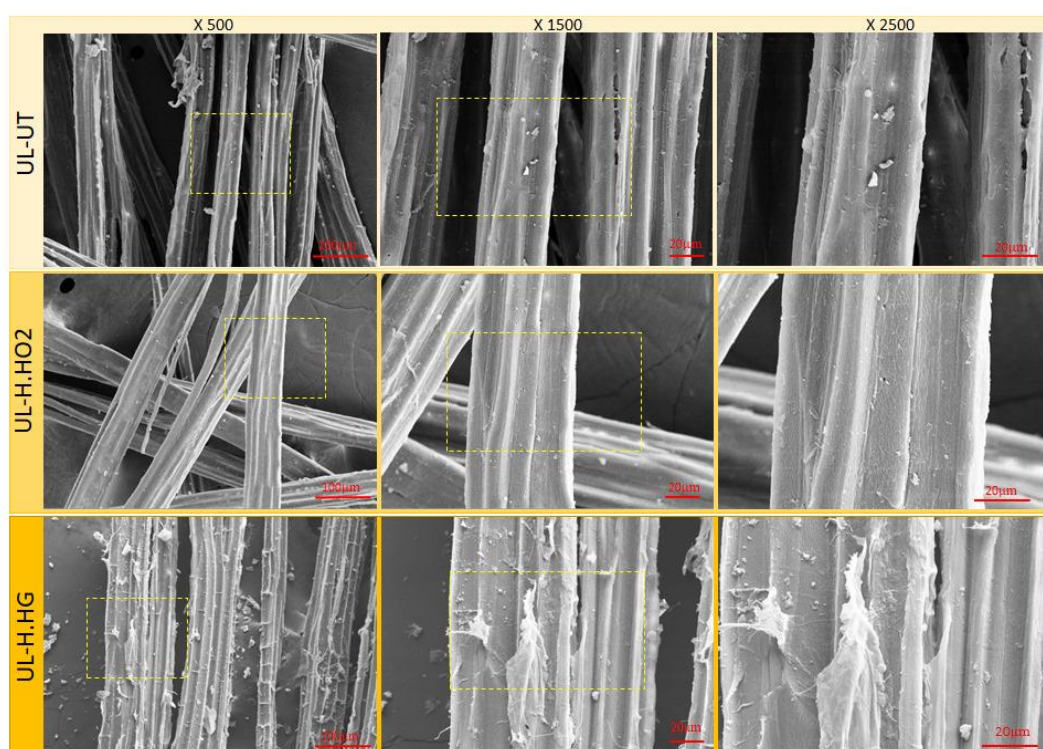


Figure 8. The SEM Image. (a) untreated UL fiber, (b) H. UL fiber, (c) H. H/G UL fiber.

### 3.7. SEM/EDX Analysis

SEM/EDX analysis was conducted to evaluate the elemental composition of *Urena lobata* (UL) fibers treated with hexamine and gallic acid (H/G), the conventional method (H), and the untreated fibers (UL-UT). The micrographs and elemental data revealed the following trends:

- 1) *Treated Fibers (H/G)*: The carbon content increased significantly to 69.26%, suggesting enhanced stiffness due to chemical treatment. The slight reduction in oxygen content compared to untreated fibers indicates decreased hydrophilicity. The presence of minor elements

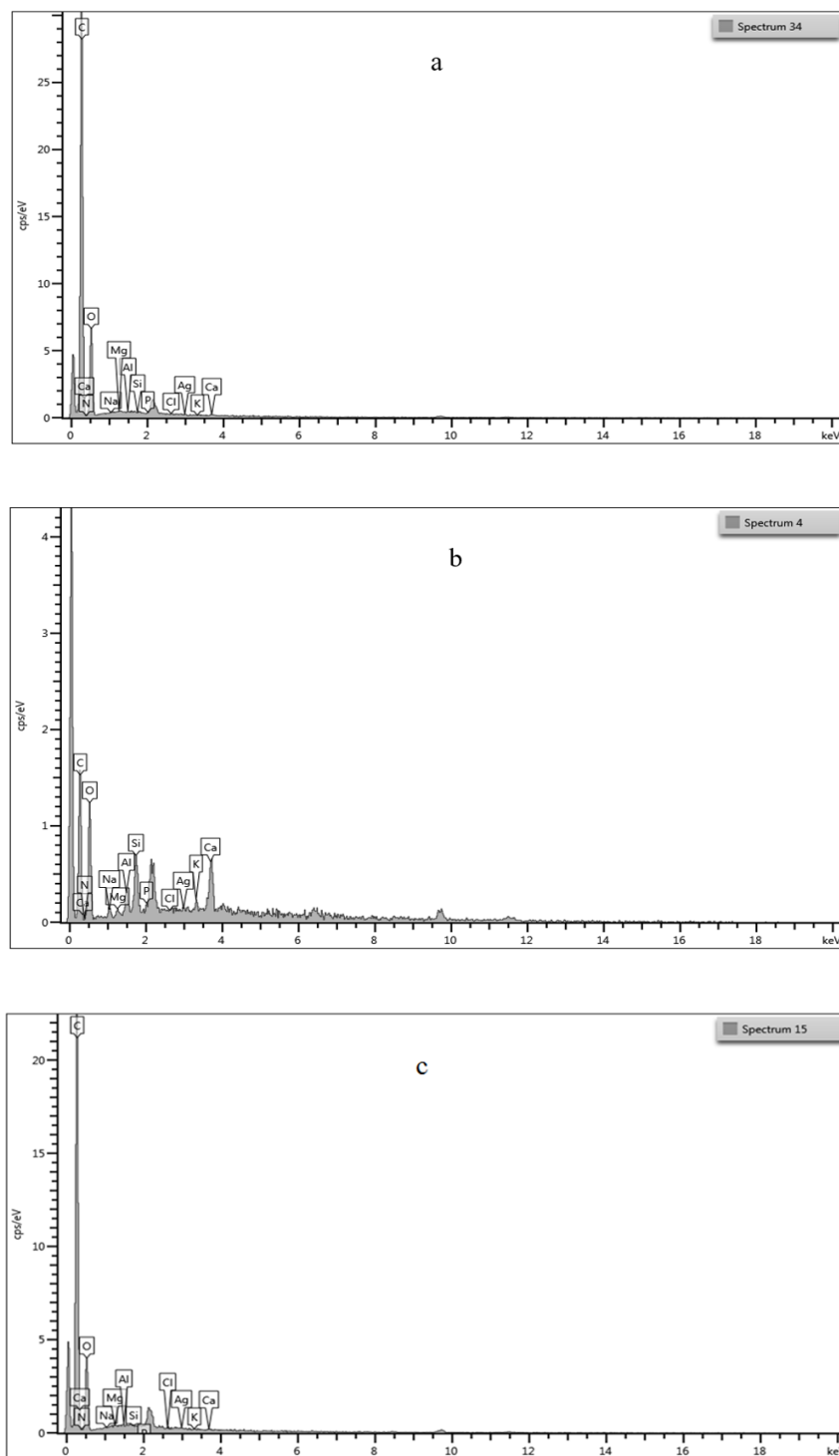
- such as magnesium, aluminum, and chlorine suggest surface modification, likely from the chemical additives, contributing to improved mechanical and water-resistance properties. This finding aligns with recent studies on chemical treatments enhancing fiber properties [61, 63].
- 2) *Conventional Method (H)*: The carbon content was significantly lower at 46.93%, indicating a considerable alteration in chemical composition. An increase in elements such as sodium (44.44%), calcium (4.30%), and minor elements like magnesium, aluminum, and potassium points to the absorption of minerals from the water used during the conventional hornification process.

These changes may enhance specific mechanical properties but could also introduce variability in performance [7, 54].

- 3) *Untreated Fibers (UL-UT)*: The untreated fibers exhibited high percentages of carbon (67.26%) and oxygen (32.43%), representing their natural state. Minimal amounts of other elements were detected, reflecting the absence of chemical treatment. This composition aligns

with typical untreated lignocellulosic fibers [2, 49].

The results confirm that chemical treatments like hexamine and gallic acid significantly modify the fiber surface, improving stiffness and reducing hydrophilicity. Meanwhile, the conventional treatment primarily impacts mineral absorption and alters chemical composition, potentially affecting fiber properties differently.



**Figure 9.** SEM/EDX results. A) Untreated UL fibers, b) conventional UL fibers, c) H. H/G UL fibers.

### 3.8. X-ray Diffraction Result

The X-ray diffraction (XRD) diffractograms of the *Urena lobata* (UL) fiber samples display intensity versus  $2\theta$  patterns, highlighting differences between untreated and treated fibers (Figure 5). The untreated fibers exhibit distinct reflection peaks at  $2\theta$  values of approximately  $35^\circ$ ,  $41.3^\circ$ ,  $50.4^\circ$ ,  $62.9^\circ$ ,  $67.2^\circ$ , and  $74.1^\circ$ , corresponding to the (220), (311), (400), (422), (511), and (440) crystallographic planes, respectively [64, 65]. These peaks indicate the presence of crystalline iron oxide phases without significant impurities. Following hornification treatment, these characteristic peaks are diminished or lost, with minor noise peaks appearing, possibly attributable to calcite formation. A broad peak around  $2\theta \approx 17^\circ$  is observed, likely representing amorphous cellulose or overlapping reflections from the (101) lattice

plane, consistent with prior findings [68, 69]. Notably, a strong, sharp peak at  $2\theta \approx 22.1^\circ$  appears in the additives-treated UL fibers, attributed to the (200) lattice plane reflection characteristic of crystalline cellulose. This enhancement in peak intensity suggests increased crystallinity resulting from chemical treatments affecting cellulose, lignin, and hemicellulose structures [48, 60, 66, 67]. Variations in peak intensities and positions among untreated, conventional, and additives-treated fibers reflect modifications in crystallinity and molecular order. These structural changes corroborate elemental composition differences identified via EDX analysis and explain observed improvements in physical and mechanical properties such as reduced water absorption, increased density, and enhanced stiffness after treatment [49, 61].

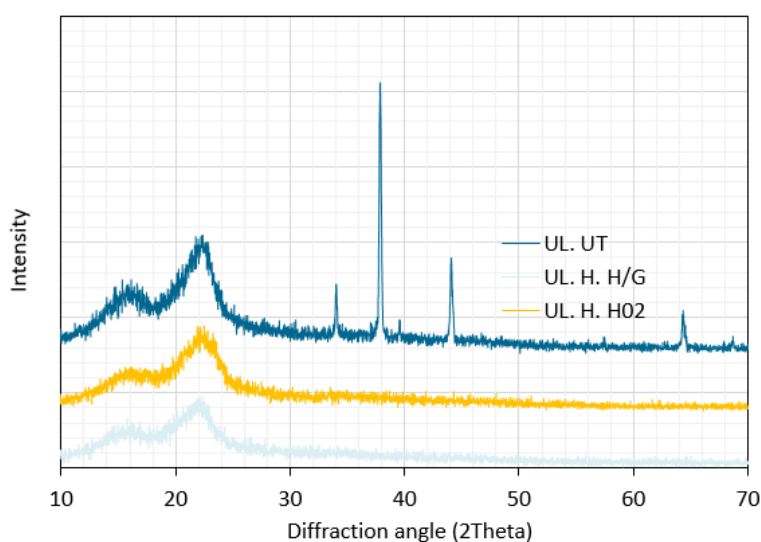
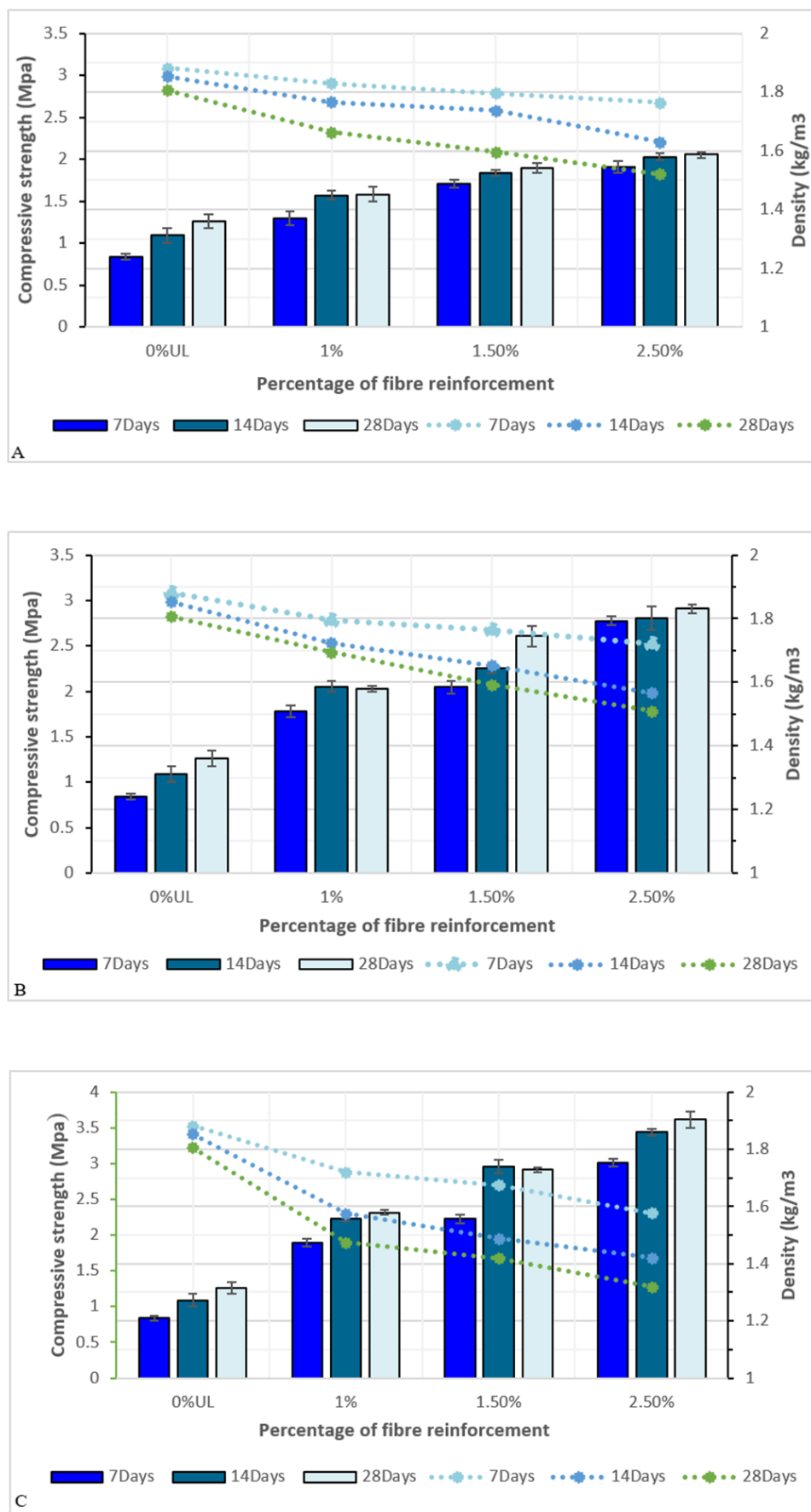


Figure 10. X-ray diffractogram of UL fiber.

### 3.9. Flexural Test

A total of 80 composite samples reinforced with treated, conventional, and untreated *Urena lobata* (UL) fibers were tested for flexural strength after 7, 14, and 28 days of curing. The data analysis revealed that the composites containing treated fibers exhibited a higher average Elastic Modulus after 28 days compared to those with untreated and conventional fibers at both 7 and 14 days. Notably, the flexural strength and stiffness of composites with fibers treated using hexamine and

gallic acid showed a consistent increase over time, suggesting that the chemical treatment significantly enhances the fiber-matrix interaction and overall composite performance. This improvement is likely due to the enhanced interfacial bonding and reduced fiber hydrophilicity resulting from the additives' hornification effects [14]. Similar trends have been reported in some studies, demonstrating that chemical modifications of natural fibers can improve mechanical properties of bio-composites by increasing fiber rigidity and adhesion with the matrix ([49, 70, 71]).

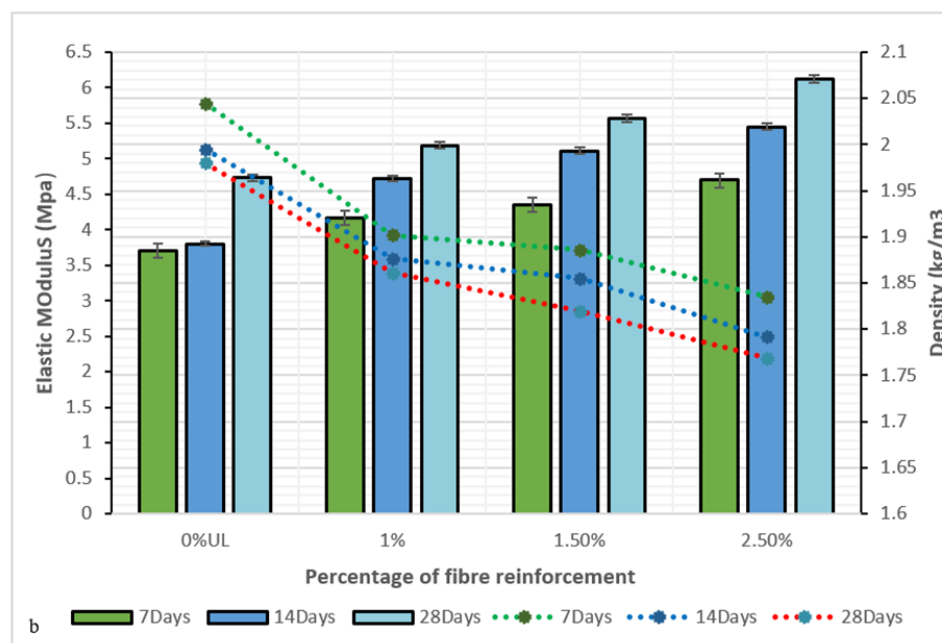
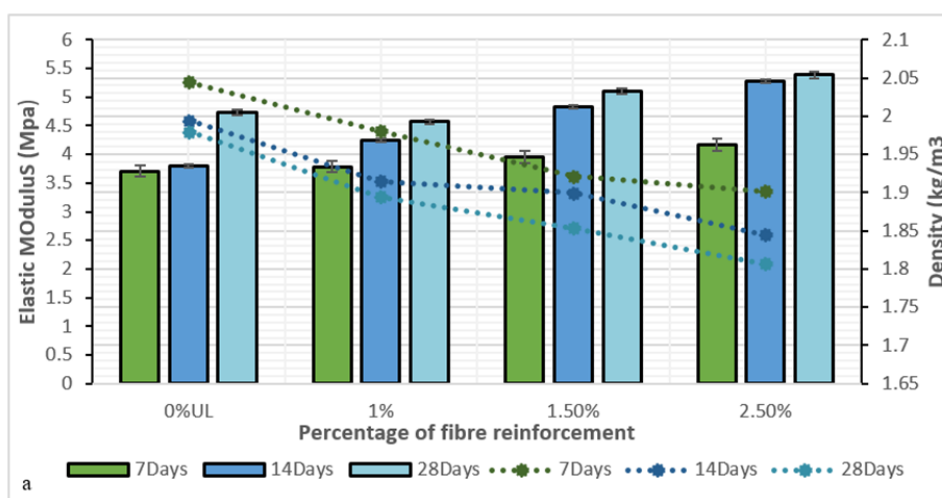


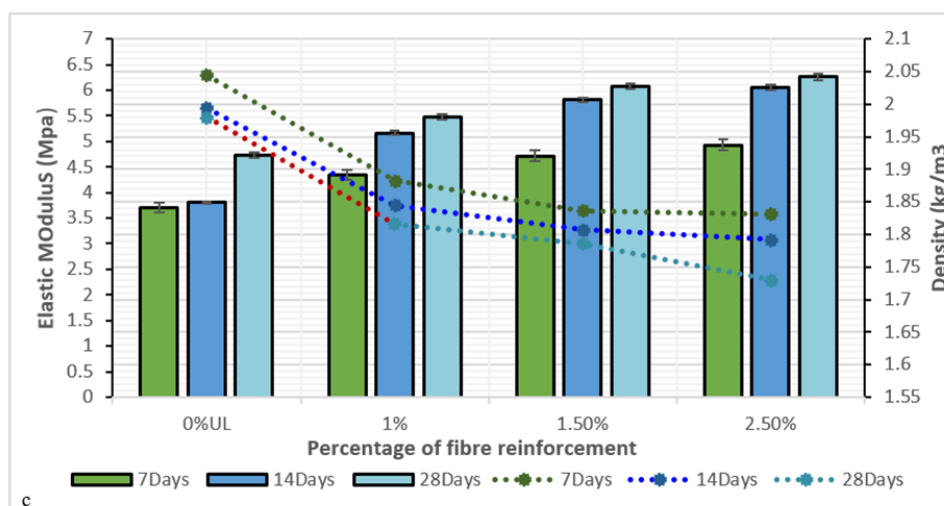
**Figure 11.** Flexural strength of different mixture proportions of fibers cement board after (a) UL untreated fibers composites (b) UL conventional fibers composites and (c) UL treated fibers composites.

### 3.10. Compressive Strength

The compressive strength values of natural fiber cement boards reinforced with *Urena lobata* (UL) fibers were evaluated at 7, 14, and 28 days of curing. The results show that the compressive strength increases with curing time across all samples. Specifically, the boards containing treated fibers at 2.5% loading demonstrated significantly higher compressive strength after 28 days compared to the same boards tested at 7 and 14 days. After 28 days of curing, the compressive strength of the cement boards reinforced with treated UL fibers reached approximately 33.4 N/mm<sup>2</sup>, which is considerably

higher than the 21.5–22.3 N/mm<sup>2</sup> observed for boards with untreated and conventionally processed fibers. This improvement can be attributed to the enhanced fiber-matrix interfacial bonding and the modification of fiber surface properties through chemical treatment, which leads to better load transfer and mechanical reinforcement ([72, 73]). Similar findings have been reported in some studies where chemical treatment of natural fibers improved the mechanical performance of fiber-reinforced cementitious composites by increasing fiber stiffness and reducing moisture absorption [74, 75].





**Figure 12.** Compressive strength and Density of of different mixture proportions of fibers cement board after (a) UL untreated fibers composites (b) UL conventional fibers composites and (c) UL treated fibers composites.

## 4. Conclusion

The effect of the number of wet-drying cycles on the physicochemical, morphological and mechanical properties of UL fibers was extracted and studied. The results reveal an increase in density and elastic modulus for the additives and conventional UL fibers. This increase in density is due to the reduction in the water content and water absorption capacity of the additives UL fibers. The decrease in water retention value is attributed to many factors; including fiber shrinkage, semi-irreversible pore closure, and increased crystallinity. This is consistent with the microstructures of the fiber cross-section and the surface of the fibers seen on the SEM results show a rougher surface for the additives UL fibers compare to the brighter untreated surface over the conventional method. A simple comparison show that UL fibers have properties similar to those in the literature. The physicochemical, morphological and mechanical changes in our fiber properties resulting from the wet-dry process have demonstrated their suitability for use as an alternative to synthetic fibers in composites.

## Abbreviations

|          |                                                                      |
|----------|----------------------------------------------------------------------|
| H/G      | Hexamine gallic acid                                                 |
| H        | Conventional method                                                  |
| UL       | Urena Lobata                                                         |
| UT       | Untreated                                                            |
| SEM      | Scanning Electron Microscope                                         |
| EDX      | Energy Dispersive X-ray Spectroscopy                                 |
| XRD      | X-ray Diffraction                                                    |
| TGA      | Thermogravimetry Analysis                                            |
| FTIR-ATR | Fourier Transform Infrared Spectroscopy-Attenuated Total Reflectance |

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## Author Contributions

**Jerum Biepinwoh Kengoh:** Conceptualization, Funding acquisition, Supervision, Writing – original draft

**Josepha Foba Tendo:** Methodology, Resources, Supervision

**Fabien Betene Ebanda:** Conceptualization, Resources, Visualization, Writing – review & editing

**Yakum Reneta Nafu:** Data curation, Methodology, Writing – review & editing

**Armel Edwige Mewoli:** Data curation, Methodology, Writing – review & editing

**Stanislas Tido Tiwa:** Formal Analysis, Writing – review & editing

## Data Availability

The data used to support the findings of the study are available from the corresponding author upon request.

## Conflicts of Interest

The authors declare no conflicts of interest.

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