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Evaluation of Hydrothermally Treated Wood Fibre Performance in Cement Mortars

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Abstract: Biofibres' wide application in mortar enhancement has thus far been restricted by factors related to their chemical composition and hygroscopic nature. Their hydrophilic behaviour increases the water demand of mortar mixtures and diminishes their affinity to the matrix, while further moisture-related fibre degradation issues may arise. Additionally, natural fibres seem to be susceptible to degradation caused by exposure to alkaline environmental conditions such as those experienced by cement mortars, restricting their utilisation in the construction industry. Therefore, the current study investigates the potential of fibre modification through treatments that would permanently alter their structure and chemical composition to improve their performance. In this study, wood fibres of black pine and beech species were exposed to mild thermal treatment (140 °C 2 h, under a steam atmosphere), characterised in terms of the physical and chemical properties and incorporated in cement mortars, applying the proportion of 1.5% v/v in the mortar, in order to assess their performance as reinforcement material. The mortars' workability (at a fresh state) was examined, as well as other physical, hygroscopic, thermal, and mechanical characteristics of the mortars at the ages of 28, 90 and 365 days and weathering performance, by subjecting them to different artificial ageing environments (freeze–thaw cycles or outdoor exposure). The results revealed the beneficial role of the treated fibres in dimensional stability, flexural strength, thermal insulation properties and capillary absorption of the mortar specimens, especially during the ageing process, with the black pine fibres showing the greatest improvement. The hydrothermally treated wood fibres seem to help maintain the integrity of cement mortars under all ageing conditions, proving that they could provide low-cost and eco-friendly mortar enhancement pathways.

Keywords: bio-fibre; cement; composites; lignocellulose; mortar; natural fibre; treatment; wood

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1. Introduction

Wood, a natural resource and valuable building material has been used in countless structural applications since ancient times. The continuous population growth and the subsequent increase in wood demands have resulted in the constantly increasing woody biomass residue generation derived mainly from logging, timber mechanical processing, demolitions, wood recovery from old furniture/structures, etc. Various industries appear to have a strong interest in the utilisation of this raw material, such as those of paper and pulp, wood-based composite materials, bioenergy, biofuels (pellets, briquettes), etc. Nevertheless, the most beneficial uses among these options are the ones that maintain the carbon content absorbed in the material mass for as long as possible, such as the long service-life-duration building materials and structures [1]. Furthermore, substituting plastic or other energy-consuming materials with wooden elements is one strategy used to create eco-friendly building materials with a low environmental impact. Currently, several types of synthetic fibres are used to transform a brittle cement matrix into a ductile and tougher one [2]: flexible fibres, such as polyvinyl alcohol, polypropylene and polyethylene, and stiff

fibres, such as glass or steel, are mainly used. On the other hand, artificial fibres require considerable amounts of energy and could be economically replaced by natural fibres [3]. Natural fibres, such as those of wood, reed and straw, have been utilised for thousands of years to reinforce inorganic materials in bricks and mortars [4]. Since wood fibres of both hardwood and softwood species are an abundant, lightweight, low-cost and less energy-intensive alternative to synthetic fibres, they are becoming more and more popular in the fibre-cement building product market [5]. In particular, incorporating wood fibres into mortars could provide good indoor air quality (since they act as moisture-buffering materials), good thermal insulation and acoustic absorbency [1,6–8]. Flexural strength, fracture toughness and dimensional stability all tend to improve with an increase in wood fibre length and a decrease in fibre diameter for a range of fibre contents [9]. A fibre content higher than 3% results in low-density mortars with improved insulating properties, as well as lower mean static bending strength [10–12].

In general, the moisture content, density, chemical composition and morphological characteristics/geometry of the fibres, fibres content, the matrix cohesion, the interface between the reinforcing fibre and matrix [13], as well as the water–cement ratio in the matrix, curing conditions and age, among others, constitute critical factors that affect the strength and almost all properties of the wood–cement composites [5].

Unfortunately, wood fibres also demonstrate lower density and comparatively worse mechanical properties compared to synthetic fibres, which also applies to the resultant composites [13]. As an organic, hygroscopic material, wood fibres are prone to dimensional instability and micro-organism catastrophic action. Their hydrophilic nature increases the water demand during mortar-mixture preparation, adversely affecting the workability and homogeneity of the fibres' dispersion [1] and decreasing their adhesion to the matrix [14,15]. Additionally, natural fibres appear to be susceptible to degradation when exposed to alkaline environmental conditions, such as those of cement mortars, restricting their utilisation in the construction industry [16].

It is well known that wood consists of lignin, cellulose, hemicelluloses, extractives and ash. Hemicelluloses are mostly made up of glucose, mannose, galactose, xylose and arabinose, as well as a few water-soluble monosaccharides [4,17]. When cement hydrates, these molecules tend to combine to create calglucon, which is beneath the cement's surface and blocks both cement and water. The cement hydration reaction rate slows down, the film gradually thickens, and the cement hardening rate gradually prolongs as the reaction goes on [4]. Furthermore, effective approaches for resolving the incompatibility of wood fibres and cement are critical to their successful utilisation as mortar reinforcement.

Treatments for wood fibres focus on increasing the fibres' dimensional stability against the changes in relative humidity that tend to cause swelling and shrinkage of the wood fibres, and the prevention of this phenomenon results in micro-cracking of the matrix during the cement hydration process [18]. In order to eliminate the described drawbacks of wood fibres, three main wood fibre modification methods have been used so far, i.e., aqueous extraction, alkaline hydrolysis and coating with substances on the fibre surface (retention treatment) [4]. Although these treatments effectively improve the performance of cement settings on wood surfaces, each one acts in a very different way. Aqueous extraction, mainly in hot water, removes inhibitory water-soluble compounds but demonstrates relatively low leaching efficiency and improvement [4]. The commonly used method of alkaline hydrolysis degrades hemicelluloses and sugars into non-inhibitory substances by exposing the wood fibres to a NaOH solution in order to dissolve 'out' the monosaccharides. This is considered an intensive treatment and is responsible for the destruction of both hemicelluloses and cellulose, highly deteriorating the wood fibres' mechanical strength [1]. In the retention treatment, a thin coating is formed on the wood surface, and inhibitory substances are not released to the setting medium; nevertheless, the final mechanical properties of the resulting composites do not appear to be satisfying [4].

Hydrothermal modification constitutes a non-biocidal, environmentally friendly and low-cost wood protection method applied to enhance the behaviour of wood and wood

fibres and some properties critical for its service life expectancy and utilisation perspectives. In the presence of steam, heat induces changes to the chemical constituents of wood, as well as altering its physical, hygroscopic and mechanical properties. This treatment may cause a decrease in the density and, therefore, in the mechanical strength of wood fibres, especially when intense conditions are applied (high temperature, pressure or duration), though it seems to limit the hygroscopic nature of the material because of the gradual thermal degradation of the hemicelluloses and amorphous parts of the cellulose [19], and enhances the natural durability of wood [17], providing a chance (especially for those species characterised by low water resistance, dimensional stability and high susceptibility to bio-degradation factors) to be adequately utilised, participating in a much wider range of applications [20]. Additionally, by restricting the hygroscopicity of the wood fibres, the workability of these fibres in cementitious matrices is being improved [1,18,21]. The depolymerisation of wood fibre hemicelluloses could also effectively improve the cement curing properties of the wood–cement mortar to some extent [4]. During the treatment, some volatile extractives that demonstrate a retarding effect on cement hydration are being released into the atmosphere. Additionally, the thermal degradation of chemical components may slightly increase the surface roughness of the wood fibres, contributing to higher adherence and affinity to the matrix [19].

Although lignocellulosic fibres have been employed so far in several experimental studies involving a cement matrix, a great lack of information has been detected in the literature concerning the potential utilisation and performance of modified wood fibres in a cement matrix and cementitious mortars, especially those treated under a mild hydrothermal modification process. Therefore, in the present contribution, residual wood fibres of black pine and beech wood species (two commonly used and abundant species) were incorporated in wood–cement composites (applying the proportion of 1.5% v/v fibres in mortar) to assess their performance as reinforcement materials after their exposure to a mild hydrothermal modification. This mild treatment is expected to induce the necessary changes in the fibre's chemical composition in order to eliminate the drawbacks of wood fibres and improve the wood–cement composites' performance. Therefore, the chemical composition of treated and untreated wood fibres has been assessed. The mortars' workability (at a fresh state), as well as their physical, hygroscopic, thermal and mechanical characteristics at the ages of 28, 90 and 365 days and their weathering performance by subjecting them to different artificial ageing environments (freeze-thaw cycles or outdoor exposure) were examined to provide a thorough insight into this field of study.

2. Materials and Methods

2.1. Materials

2.1.1. Wood Fibres and Hydrothermal Treatment

For the purposes of this study, two forest species of Greek origin were used, black pine (*Pinus nigra* L.) and beech (*Fagus sylvatica*), grown in the Kalampaka region. The wood material was transported to the laboratory in the form of boards, where it was cut and crushed into small pieces of fibrous particles (1–2 cm long) using a hammer mill to let their moisture content easily reduce and incorporate them into cement mortars. The wood fibres remained for about 2 months in an air-conditioned chamber under stable conditions (temperature 20 ± 2 °C, 60 ± 5% relative humidity) until a constant weight was achieved. Their equilibrium moisture content (EMC) was measured to be 10.73% for beech and 11.44% for black pine wood [22].

Then, the wood fibres were subjected to a mild hydrothermal treatment at 140 °C in an atmosphere of saturated steam at the low pressure of 1.5 atm in a closed chamber with a steam-generating unit in a laboratory custom-made device for the duration of 2 h from the moment of the stabilisation of the final condition in the chamber (temperature and pressure). At the end of the treatment, the wood fibres were cooled in glass desiccators and then weighed and stored in a conditioning chamber (20 ± 2 °C, 65 ± 3% relative humidity).

Fourier transform infrared spectroscopy-attenuated total reflectance (FTIR-ATR) was performed at room temperature on the surface of treated and untreated wood fibres of the two studied wood species to detect any potential chemical changes induced in these lignocellulose materials by the mild hydrothermal treatment. The measurements were implemented in the range of 500–4000 cm^{-1} in the respective FTIR-ATR device (Agilent technologies, Tokyo, Japan, Cary 630 FTIR ATR), and the software of FTIR "MicroLab" was used for the spectra processing and visualisation.

2.1.2. Chemical Analysis of Fibres

Even though in this study there is no reference to untreated wood fibres, a comparative analysis of treated and untreated fibres' chemical composition was performed to understand the role of hydrothermal modification on their properties. According to the spectroscopic analysis of unmodified and thermally modified black pine and beech wood fibres derived from FTIR-ATR spectroscopy, an intense variation in the anatomical characteristics, chemical compounds, chemical bonds and structural characteristics between the untreated and treated black pine (Figure 1) and beech (Figure 2) wood fibres were not detected (neither derived from the treatment nor from the different species). The peaks of spectra were found to be fairly typical, indicating that wood bears the same functional groups and wood bond types among species, which result in various vibrations upon contact between the wood fibre and the light bundle. Although the different bands that correspond to different characteristics of wood groups (aromatic rings, double-bonds, carbon chains, carbonyls, hydroxyls, absorbed moisture, etc.) were examined, differences between the spectra of treated and untreated wood are challenging to identify and complicated to assess, as there are numerous interactions in wood constituents that occur simultaneously during the thermal treatment process [23].

According to the spectra of both species, a slight increase in the carbonyls (C=O) of hemicelluloses has been identified in the spectra band of 1700–1730 cm^{-1}, which is true for both of the two different studied wood fibre species. The mild hydrothermal treatment appeared to induce low-intensity changes, in addition to changes in the spectral lines in those regions that correspond to the wood's chemical components. There is a slight difference between the untreated and treated samples within the bounds of 1462–1425 cm^{-1} and 1384–1346 cm^{-1}, in which the key wood components of cellulose and hemicelluloses are expressed, supporting the hypothesis that even such a mild hydrothermal treatment may affect the level of wood components.

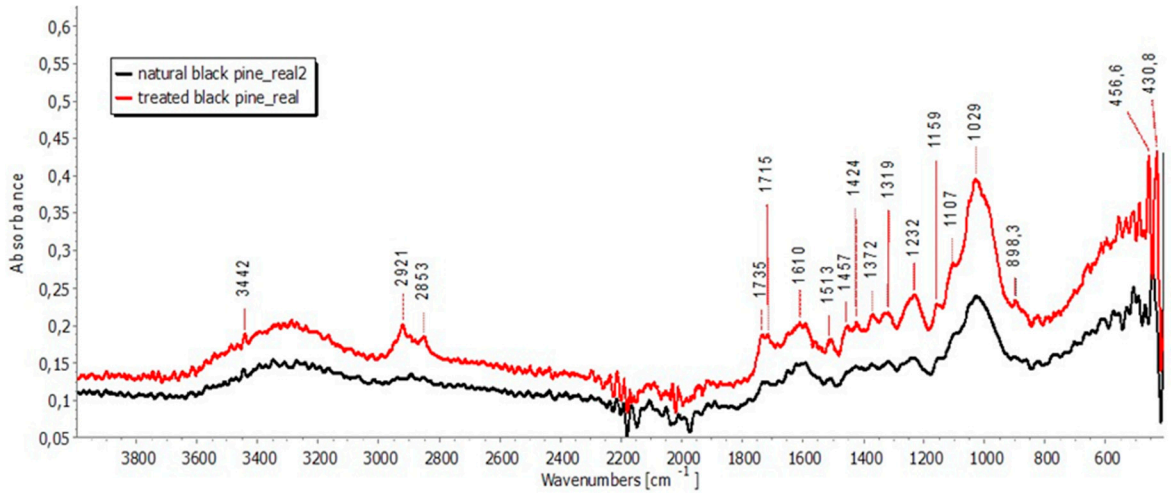

Figure 1. FTIR-ATR chemical analysis spectra of untreated and thermally treated black pine fibres over the range of 4000–500 cm^{-1}.

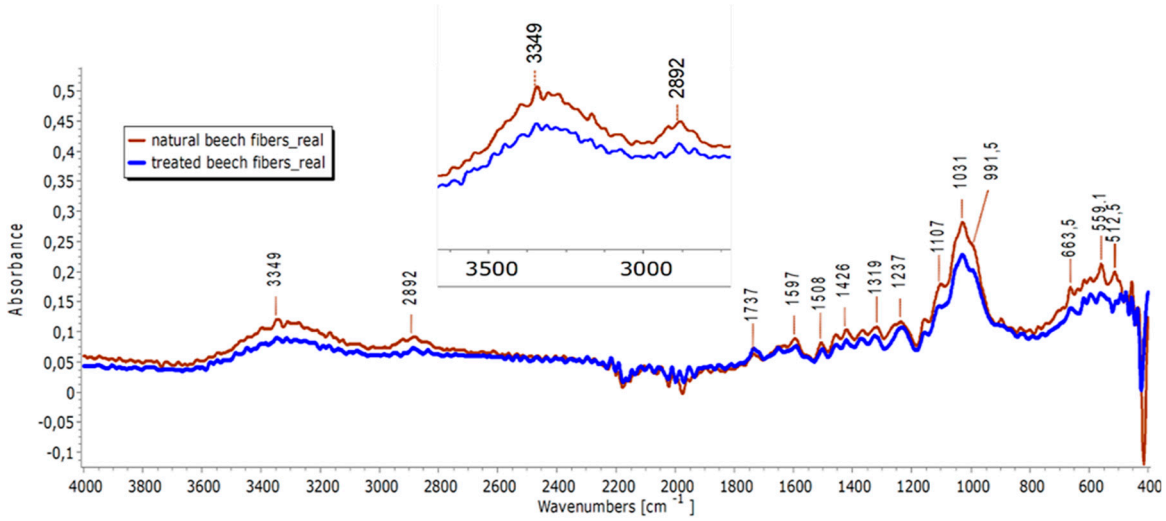

Figure 2. FTIR-ATR chemical analysis spectra of untreated and thermally treated beech fibres over the range of 4000–400 cm^{-1}.

In the case of black pine, the spectra reveal that the 3430 cm^{-1} band, which represents the O-H stretching vibrations of alcohols (3600–3300 cm^{-1}) and carboxylic acids (3300–2500 cm^{-1}) as well as involving lignin and polysaccharides evenly, did not significantly change, while in the case of beech wood, slight changes have been detected, corresponding to O–H stretch vibrations, revealing a slight decrease in polysaccharides in the mass of wood. Concerning both species of wood fibres, the hydroxyl groups were found to decrease slightly by the hydrothermal treatment. The asymmetric stretch vibrations of the $–CH_2–$overlap to form the two bands at 2900 cm^{-1} and 2800 cm^{-1} (the band around 2921–2853 cm^{-1}) and $–CH_3$ (the band around 2970–2950 cm^{-1}), along with the symmetric stretch vibrations of the $–CH_2–$overlapping band at 2865–2845 cm^{-1} and $–CH_3$ (2880–2860 cm^{-1}). In the specific area of bands, a minor shift is depicted. According to the literature, it is expected that thermally treated wood would present a decrease in this band, especially in mild treatments [24,25]. Beech demonstrated a shift and an increase in the 1735 cm^{-1} band.

Furthermore, the spectra of the peaks at 1460 cm^{-1} and 1420 cm^{-1} showed a notable difference. Based on the literature, it is expected that the peaks of absorption found at 1460 cm^{-1} and 1420 cm^{-1} will rise after thermal modification [26].

Concerning the pine fibres, the band of 1600–1750 cm^{-1} displayed a slight variation between the C=O bonds of carbonyls and C=C bonds of alkenes of the hydrothermally modified and reference unmodified fibres.

In the regions of 1457–1424 cm^{-1} and 1372–1345 cm^{-1}, within which the fundamental structural elements of wood are illustrated, there is a minor variation between the untreated and treated pine wood fibres, just as with beech wood, which strengthens the belief that the hydrothermal modification slightly affected the contents of these structural components, mainly the hemicelluloses, which were slightly degraded due to hydrothermal modification. Within the bands of 1505–1512 cm^{-1}, a slight reduction in methoxyl groups was detected, with the subsequent increase in mass loss during the treatment. In the area of 3600–3300 cm^{-1}, which reflects the O-H stretching vibrations of alcohols, slight variations were detected in the spectra of treated and untreated fibres, as well as in the area of 3300–2500 cm^{-1}, which corresponds to carboxylic acids, concerning mainly polysaccharides.

Therefore, several minor modifications and changes in the FTIR-ATR spectra of both species of hydrothermally modified wood fibres demonstrated that the components of wood had been slightly influenced by such a mild thermal treatment. The free hydroxyl groups were decreased because of the slight and gradual thermal depolymerisation of hemicelluloses. The extracts of pine and beech wood fibres exhibited a decrease since

the volatile ones have been evaporated, while some new extracts were formed by the hemicellulose degradation products. In general, according to the FTIR-ATR findings, the fibres were found to be of enhanced properties compared to the untreated fibres without being intensively thermo-degraded by the treatment, which increases their utilisation potential as raw materials in cement-based mortars and composites, enhancing these products' properties and performance as well.

The mean EMC values of the treated beech and black pine wood fibres were measured to be 8.36% (10.73% before treatment) and 9.24% (11.44 before treatment), respectively. Further data that resulted from the characterisation of the specific natural/unmodified fibres (reference fibres) and their performance in respective cement–wood fibre composites, without applying any modification, are provided in a recently published research study by our team [8].

2.1.3. Preparation of Mortars

Three cement mortars (C'-) were produced, including a reference-unreinforced mixture (R) and two wood fibre-reinforced mixtures. For the enhancement of the cement (type CEM I42.5) mortars, hydrothermally treated black pine (pn) and beech (fs) fibres (mean EMC of 8.36% for treated beech and 9.24% for black pine, respectively) of a 1–2 cm length were applied in the proportion of 1.5% *v*/*v* (fibres volume per mortar volume) (Figure 3). This specific percentage was determined based on previous experience and study of old mortars, where often the number of fibres included was not exceeding this value [27]. Concerning the remaining mortar components, natural siliceous sand (0–4 mm) of river origin was utilised as the aggregate, and the water/binder ratio remained stable at 0.45. The binder/aggregate ratio was stable (1/ 2.5 by weight) in all cases. It should be mentioned that a small portion (% *w*/*w*: weight of additive per weight of binder) of superplasticiser (aqueous polycarboxylate solution) was added to the bio-reinforced mortars, aiming to achieve the expected workability (11 ± 1 cm) based on EN 1015-3 [28]. The detailed mortar composition, together with their succeeded workability, is displayed in Table 1. Evaluating these results, it seems that the mild hydrothermal treatment minimises the hydrophilic character of wood fibres since, with the incorporation of a low superplasticiser amount, a similar workability to the reference sample has been accomplished.

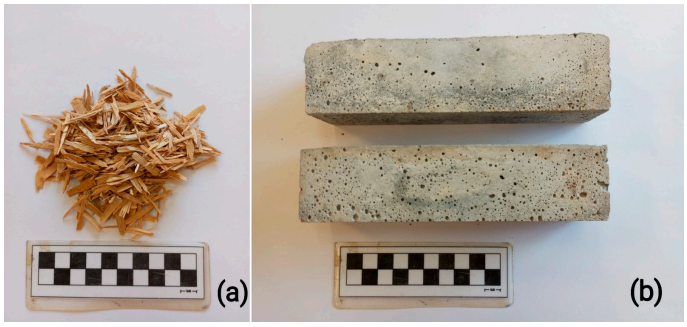

Figure 3. Wood fibres of beech species (*Fagus sylvatica* L.) (**a**) and the respective specimens of wood–cement mortars prepared for the static bending strength test (**b**).

Table 1. Composition of cement mortars (in proportion by weight).

Mortars	Cement	Aggregates	Wood Fibres (% *v*/*v*)	*w*/*c*	Superplasticiser (% *w*/*w*)	Workability (cm)
C'R	1	2.5	-	0.45	-	12.0
C'pn	1	2.5	1.5	0.45	0.25	12.0
C'fs	1	2.5	1.5	0.45	0.25	12.0

For the implementation of the experimental tests, prismatic specimens (4 × 4 × 16 cm^3) and flat (slab) (20 × 20 × 2.5 cm^3) specimens were prepared. After demoulding, all samples

(except for those intended to be employed for the determination of volume stability and water vapour permeability) were placed in a climatic chamber under the stable conditions of 20 ± 2 °C and 95% ± 5% relative humidity and remained under these curing conditions until the time of the testing process (namely for 28, 90 and 365 days of hardening).

2.2. Methods

Physical, mechanical and artificial ageing tests were performed using the prismatic specimens, while hygroscopic and thermal property measurements were performed on the flat specimens, respectively. In particular, at all ages of specimens, experiments were conducted, including the determination of the mortar's apparent specific gravity; absorption and open porosity values (according to RILEM CPC11.3 [29]); capillary absorption (based on EN 1015-18 [30]); drying rate, indicating the first 24 drying hours (according to EN 16322 [31]); mechanical properties in terms of the compressive and flexural strength (based on EN 1015-11 [32]); and the dynamic modulus of elasticity (based on EN 12504-4 [33]). The investigation of the composites' physical properties was completed with the evaluation of their volume stability. The specific assessment was achieved by placing the prismatic mortar samples (after demoulding) in a climate chamber in which the stable conditions of 20 ± 2 °C and 50 ± 5% rh prevailed, as well as measuring their dimensions in accuracy on a daily basis using a digital vernier calliper until the achievement of volume stabilisation, which usually occurs concerning the cement mortars during the first 28 ageing days [34].

Regarding the mortars' thermal properties, the thermal conductivity coefficient (λ) was assessed using a heat flow metre apparatus (HFM 100 Series-Thermtest), conforming to EN 12667 [35], at all samples maturing times (as previously mentioned) at two mean heating temperatures (10 °C and 20 °C). In order to establish these final temperatures, a difference of 10 °C was required to be set between the two heating–cooling plates of the instrument. Hence, aiming to perform the test at 10 °C (mean temperature), the lower plate was set at 15 °C and the upper at 5 °C, while for 20 °C (mean temperature), the lower plate was set at 25 °C and the upper at 15 °C. Prior to the measurements, the specimens were subjected to a drying-smoothing procedure to avoid the moisture content's influence and meet the surface quality requirements of the device manufacturer. This process involved their placement into a climate chamber under the conditions of 20 ± 2 °C and 50 ± 5% rh until constant mass (the determination of the mass changes was carried out by applying successive weight measurements) and then sanding of the flat sample surfaces until the achievement of smooth and parallel/flat sides. For each specimen category, three flat samples were prepared and characterised.

In regards to the specimens' hygroscopic performance investigation, water vapour permeability tests were performed based on the methodologies described in EN 1015-19 [36] and EN ISO 12572 [37]. The methodology included the storage of the flat samples (after demoulding) for 2 days in moist conditions (a climatic chamber of 20 ± 2 °C and 95 ± 5% rh) and for 26 days in dry conditions (20 ± 2 °C and 50 ± 5% rh) as a primary step prior to testing. Afterwards, the test set-ups were formed by sealing the mortars over cylindrical test cups with hot glue silicone, which contained an air gap of 2.5 cm and a saturated solution of potassium nitrate (KNO_3). This solution was provided within a set-up of 97% rh at 21 °C. Aluminium foil was used for the mortar peripheral covering. After that, the set-ups were placed in the climatic chamber at 20 ± 2 °C and 50 ± 5% rh and daily weighted until the achievement of a steady-state vapour transmission rate. The calculation of the mortars' water vapour permeability Wvp involved the multiplication of the water vapour permeance Λ (described below) with the slab thickness. Then, the water vapour diffusion-resistance factor μ (dimensionless quantity) was defined as the ratio of the air-water vapour permeability to the mortar-water vapour permeability. The water vapour permeance equation in [kg/m^2*s*Pa] is as follows:

$$\Lambda = \frac{1}{A\Delta_P/(\Delta G/\Delta t) - R_A}$$

where A describes the area through which the water vapour diffusion occurs (m^2), Δ_P is the partial water vapour pressure difference between the set-up air and the climate chamber air (Pa), and, finally, R_A defines the set-up air-gap water vapour resistance (0.048×10^9 Pa $\times$ m^2 $\times$ s/kg per 10 mm air gap).

Finally, the mortars' weathering performance was evaluated by subjecting the 90-day prismatic samples to different artificial ageing environments, including exposure to freeze–thaw cycles and outdoor conditions. In correlation with the first case, the mortars were submitted to 60 freeze–thaw cycles based on ASTM C666/ C666M-03 [38], incorporating freezing in the air (-18 ± 2 °C) for 4.5 h $\pm$ 0.5 h and thawing in water (specimens immersed in water) at 4 ± 2 °C for 19.5 $\pm$ 0.5 h. During this durability procedure, the wet mortars' dimensions (utilising a digital vernier calliper) and dynamic moduli of elasticity (based on [33]) were recorded. The choice of the specific cycle number (which usually in the literature is over 300 for cement compositions) was taken in the frames of avoiding the mortars' visible deterioration and achieving their mechanical property measurements [39]. The specimens remained exposed to exterior environment conditions for 8 months. During this experiment, the volume changes and the dynamic moduli of elasticity of the specimens were evaluated. For each ageing technique, 3 samples of each specimen category were tested. At the end of the weathering exposure, the mortar porosity, dynamic modulus of elasticity (in a dry state) and compressive and flexural strength were assessed (according to the standards mentioned previously).

3. Results and Discussion

3.1. Physical Properties

Evaluating the mortars' volume deformations, presented in Figure 4, it seems that the addition of treated wood fibres in cement mortars improved their volume stability. This observation could be justified by the decreased volume changes in the reinforced mortar samples in comparison to the reference samples. In general, during hardening, the cement materials tended to shrink due to moisture loss. This water movement originated from the chemical reactions occurring inside the cement structure (hydration process) or the interactions with exposure to environmental conditions (drying-water evaporation). Considering these, the volume stabilisation of the bio-reinforced cement mortars is probably affected by the fibres' water absorbency, which leads to moisture release in the cement matrix [40,41]. Considering the different wood fibre performances, the black pine fibres demonstrated the most stable performance during maturing, which is in accordance with their higher mechanical strength, higher lignin content and higher release of moisture in the cement matrix compared to the beech fibres [8,25]. In all cases, the mortar dimensions tended to stabilise after the age of 24 days.

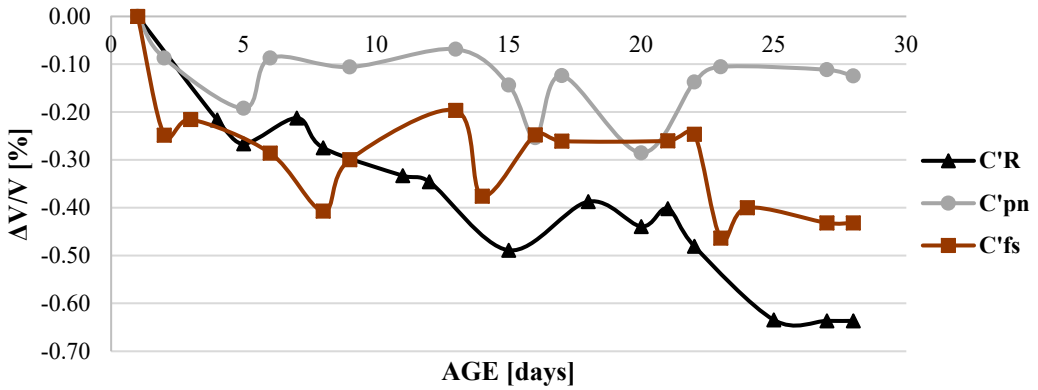

Figure 4. Volume deformations of the produced mortars (C'R: reference/unreinforced mixture, C'pn: reinforced mixture with black pine fibres, C'fs: reinforced mixture with beech fibres).

In regard to the characterisation of the mortars' physical properties, presented in Table 2, the presence of the biofibres increased the water absorbency and the porosity values of the specimens, while the samples became slightly lighter. The wood fibres' low density influenced the specific gravity of the final-reinforced cementitious composites [42]. Comparing the two wood species' fibres impact, the highest differences from the reference sample property levels were recorded for beech fibres (with a 97% absorption increase at the age of 28 days or 37% after 365 hardening days and an 89% porosity increase at the age of 28 days or 32% after 365 hardening days). However, the fibre-reinforced mortars displayed similar specific gravity values over time, with the difference from the reference composite samples extending about −4.1% at the age of 365 days. Evaluating the mortars' physical properties over time, the unreinforced samples presented an increase in water absorption capacity from 28 to 90 days of ageing. Then, the stabilised values were recorded for 365 days, while the pn-fibre-reinforced mortars showed stable physical properties through the maturing process (with low variations). Finally, the fs-reinforced specimens displayed a decline in their porosity-absorption values from 28 to 90 hardening days and then a stabilisation process until the age of 1 year. In addition, the composites preserved their specific gravity rates during hardening (with low variations). A possible explanation of this specific phenomenon lies in the formation of micro-shrinkage cracks due to ageing in the pure cement structure and their absence from the fibre-reinforced composite cases due to the fibres swelling (this complies with the mortar volume deformations presented in Figure 4). Also, the fibres' moisture release promoted the hydration process of the non-hydrated calcium silicate compounds [43] with their products to fill the existing voids.

Table 2. Absorption, porosity and specific gravity of the produced mortars at the ages of 28, 90 and 365 days (28 d, 90 d and 365 d).

Sample	Absorption (%)			Open Porosity (%)			Specific Gravity [g/cm^3]		
	28 d	90 d	365 d	28 d	90 d	365 d	28 d	90 d	365 d
C′R	1.77	2.43	2.33	3.91	5.29	5.20	2.21	2.18	2.20
C′pn	2.74	2.79	2.80	5.93	6.04	6.18	2.16	2.16	2.14
C′fs	3.48	2.65	3.19	7.38	5.78	6.85	2.12	2.16	2.15

The capillary absorption of the specimens is affected both by the addition of biofibres (depending on the wood species) and their hardening time. According to Figures 5 and 6, it appeared that the reference mortars maintained their tendency towards a stable capillary absorption over time (referring to the first and final measurements), although from 28 to 90 days, there was an increase of about 82% in the capillary water absorption coefficient (C). This increase is probably related to the porosity increase and the formation of the shrinkage cracks mentioned previously, which amplifies the communication of the internal micropores. This tendency was balanced during maturing when the hardening products reduced the voids and diminished their interconnection [44]. Examining the fibres' performance, the two reinforced mortars behaved differently. As presented in Figures 5 and 6, the C′pn case displayed a decreasing absorption inclination through ageing, with lower water absorption values than those of the reference case at the age of 365 days (a −50% variation of the C value). In contrast, the C′fs case presented a rising trend through maturing, recording shorter capillary curves than the reference composition between 28 and 90 days of hardening. Apparently, the voids/pores connection system was affected by the mortar's porosity and hydration process of cement since their interaction was impaired in the pn case (the porosity remained stable, but the hydration products minimised the pores' connections) and improved in the fs-type (the porosity was decreased, but interconnected micropores may be formed). A rational explanation probably arises from the fibre structure, which affects the interaction zone of the fibre–cement matrix and contributes to the formation of the mortar capillary systems.

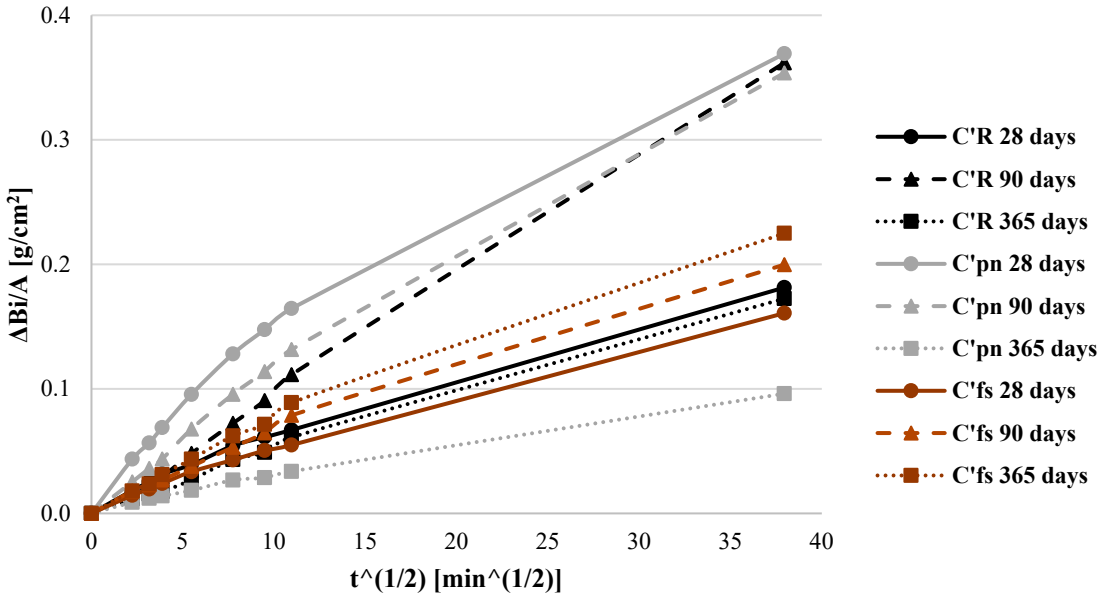

Figure 5. Capillary absorption of the cement mortars at the ages of 28, 90 and 365 days.

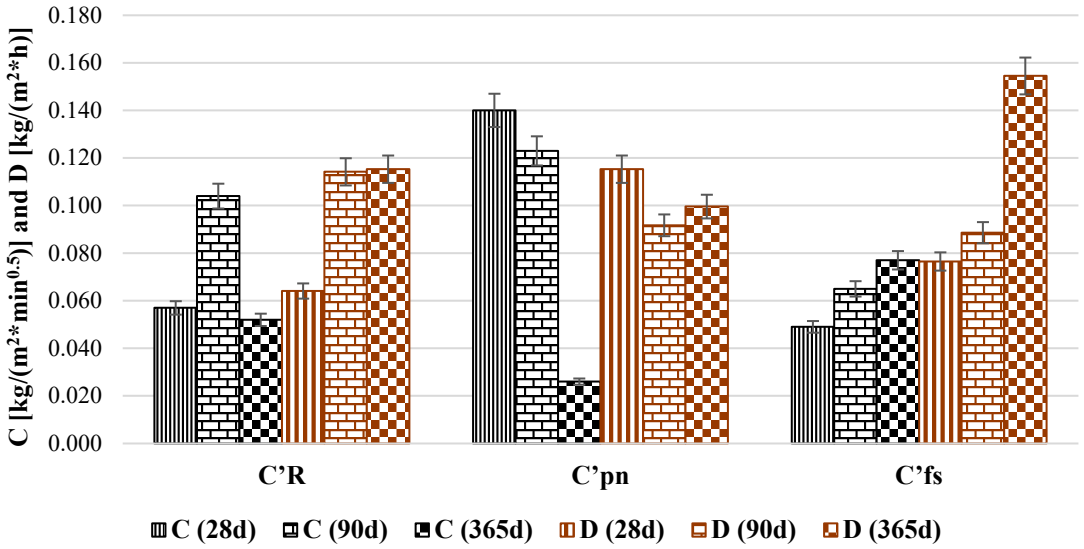

Figure 6. Capillary water absorption coefficient (C) and drying rate for the first 24 h of drying (D) of the cement mortars at the ages of 28, 90 and 365 days (28 d, 90 d and 365 d).

Finally, by examining the specimens' drying curves (Figure 7), it was observed that all different material samples became less dense during maturing as the drying phases became longer. The specific behaviour was related to the porosity values of the C'R and C'pn cases and to the capillary absorption tendency of the C'fs composite category. Concerning the effect of the wood fibres in the mortar drying procedure, it appeared that at early ages (28 days), the presence of fibres boosted the drying action of the cement mortars (with the reference case being the densest), while after 365 hardening days, the C'pn mortars became the densest (which displayed the lowest capillary absorption coefficient at this age). On the other hand, after 365 maturing days, the C'fs case showed the highest drying rate, which corresponded to an increase of about 34% compared to the reference composite category (Figure 6).

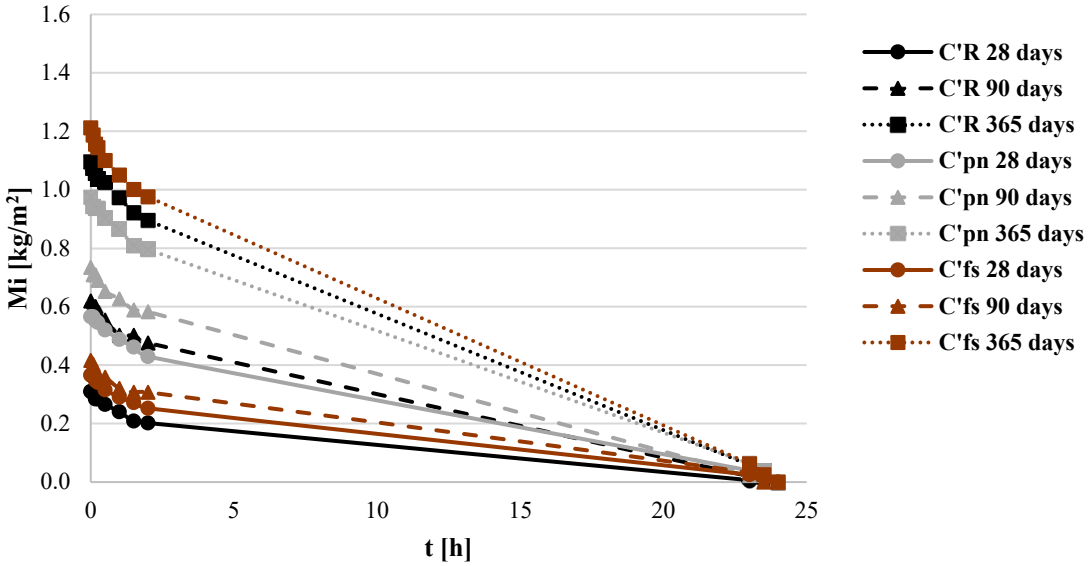

Figure 7. Drying curves of the produced mortars after 28, 90 and 365 hardening days.

3.2. Mechanical Properties

During the maturing process, the mechanical properties of all composite samples were found to be enhanced, marking a gradual improvement (Figure 8). More specifically, regarding the strength improvement in the mortars from 28 to 365 days, the flexural strength was enhanced by about 25%, 27% and 22%, and the compressive strength was improved by approximately 20%, 30% and 20% for the C'R, C'pn and C'fs samples, respectively. However, this uniform behaviour was interrupted concerning the flexural strength of all sample categories from 28 to 90 ageing days, when a decrease of about 3%, 9% and 1% was observed (for C'R, C'pn and C'fs mortars, respectively). The specific improvement in mortar mechanical properties could be attributed to the hydration process of cement and the curing conditions, as the wet environment provides the humidity required for the hydration of the non-hydrated compounds [45,46]. Moreover, the flexural strength reduction from 28 to 90 days is probably associated with the corresponding porosity increase in the samples (Table 2).

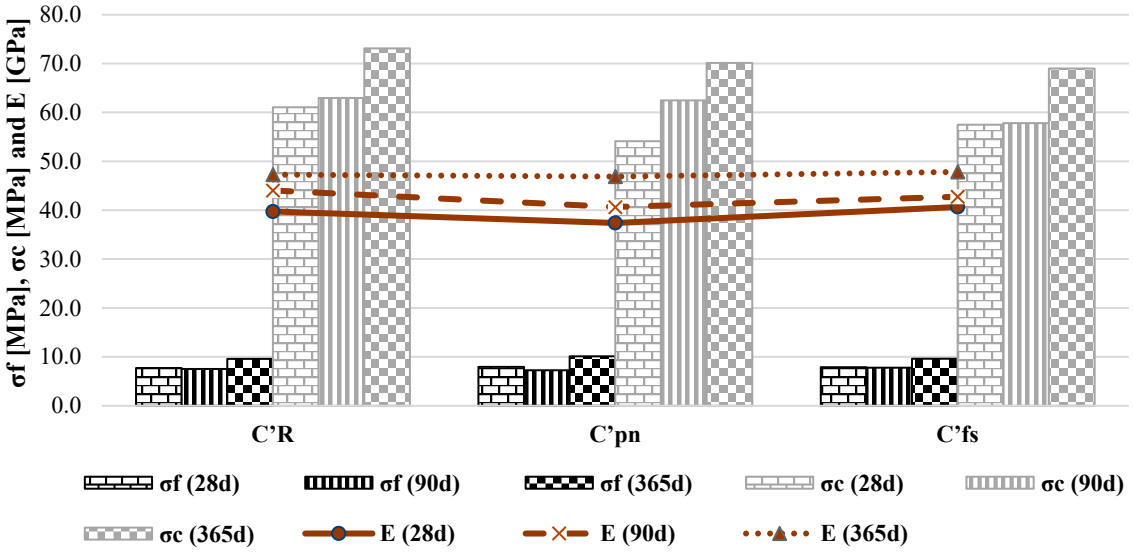

Figure 8. Flexural strengths (σ_f), compressive strengths (σ_c) and dynamic moduli of elasticity (E) of the produced mortars at the ages of 28, 90 and 365 days (28 d, 90 d and 365 d).

The treated wood fibres incorporated into a cement matrix provided enhanced flexural strength to the mortar specimens and a slightly lower compressive strength (Figure 8) during hardening in comparison to the unreinforced specimens. The cement mortar's flexural behaviour for each hardening time (28, 90 and 365 days) was mostly benefited by an increase of about 3.3% (pn fibres), 3.6% (fs fibres) and 5.4% (pn fibres), respectively. On the other hand, concerning the compressive strength, the lowest reduction was about 5.8% (with fs fibres), 0.8% (with pn fibres) and 4.0% (with pn fibres) for the previous hardening ages, which could be justified by the higher porosity values of the bio-enhanced samples. The performance of treated wood fibres in the cement mortar's mechanical properties confirmed the results presented in the literature, revealing that biofibres tend to reinforce the low flexural strength of cement compositions by bridging the cracks through the pull-out procedure [18]. Meanwhile, the presence of biofibres usually adversely affects the compressive strength of cement mortars due to the destruction of hydrogen bonds (formed between the matrix and fibres) caused by water absorption during hardening in a wet environment [34,47]. High differences in the response of the samples reinforced with the two different wood species fibres were not detected.

Finally, Figure 8 displays the samples' dynamic moduli of elasticity (E) throughout one year of ageing. The results revealed an improvement in the mortars' rigidity during hardening, which responded to the compressive strength evolution [48]. The specific increase was equivalent to 19%, 25% and 16% from 28 to 365 days for the cases of C'R, C'pn and C'fs, respectively. Furthermore, the addition of black pine fibres contributed to a reduction in the mortars' E values, while as time passed by, this differentiation was normalised until the 0.8% decrease at the age of 365 days. In contrast, the beech fibre-reinforced samples presented similar values to the reference composition through all maturing times. This behaviour of fibre-reinforced mortars was probably affected by the fibres' E value and the composite structure. More specifically, it has been proved that the saturated fibres' E value is a scale lower from the corresponding dry state, which negatively affected the mortar's modulus of elasticity. Moreover, the existence of flaws, namely voids and microcracks, in the mortar structure also reduces its stiffness [49].

The fibres' performance through 365 days of ageing designated their durability and satisfactory functionality in alkaline environments. This statement can be documented by the fact that the weakening of the fibres would lead to a mechanical strength impairment, as mechanisms like fibre mineralisation or cellulose alkaline hydrolysis could occur [47,50].

3.3. Hygrothermal Properties

According to the composite specimen's thermal behaviour, displayed in Table 3, it seems that cement mortars maintained their high thermal conductivity values during ageing. There was a slight decrease of about 0.8–4.3% between 28 and 90 days of hardening and a higher reduction of about 2.8–14.9% for all samples at both temperatures tested, probably due to the porosity rise and the micro-cracking formation [51]. Furthermore, the presence of wood fibres in the cement matrix induced a reduction in λ values of about 32.7–36.2% for the C'pn samples case (compared to the reference of all ages) and a corresponding decrease of about 10.1–27.5% for the C'fs specimens. The explanation of this behaviour lies in the higher porosity and lighter structure records of the reinforced samples compared to the reference (Table 2) and in the thermal insulation nature of the biofibres. According to the Technical Guideline T.O.T.E.E. 20701-2/2017 of the Technical Chamber of Greece (TGG) [52], the pine wood λ value is 0.14 W/(m*K), and the beech wood value is 0.17 W/(m*K), which are an order of magnitude lower than the pure cement matrix material (C'R case).

Table 3. Thermal conductivity coefficient (λ) at the ages of 28, 90 and 365 days and water vapour resistance factor (μ) of the produced mortars.

Samples	λ [W/(m*K)]						μ
	28 Days		90 Days		365 Days		
	10 °C	20 °C	10 °C	20 °C	10 °C	20 °C	
C′R	1.2597	1.3008	1.2050	1.2586	1.1101	1.0723	57
C′pn	0.8093	0.8302	0.8032	0.8474	0.7440	0.7210	44
C′fs	0.9251	0.9426	0.9164	0.9158	0.9421	0.9635	51

In regards to the samples' hygroscopic characteristics (Table 3), the water vapour permeability of the cement mortars was improved through the use of biofibres, especially in terms of the buildings good indoor air quality [1,7]. More specifically, the pn fibres contributed to a 22.8% decrease in the water vapour resistance factor (μ) compared to the reference case, while fs fibres were conducive to a corresponding 10.5% decline, attributed to the tortuosity of the pore [53]. Hence, the fibres-reinforced mortars, of higher porosity values than the reference specimens, were found to demonstrate an advanced ability for moisture transfer inside their structure, leading to a regulation of the interior living conditions in the case of inhabited edifices in accordance with the literature [54].

3.4. Weathering Performance

Evaluating the E and volume deformation curves of the cement mortars during artificial ageing (Figures 9 and 10), similar modification trends of the slope were observed between the reference and the fibre-reinforced samples under both weathering conditions. Extensive analysis revealed that, at the freeze–thaw cycles, the C′R and C′fs samples presented similar E values, with the C′pn values being the lowest ones. Nevertheless, in relation to the volume deformation, the C′R and C′pn compositions displayed the highest dimensional stability. The gradual increase in the specimens' dynamic moduli of elasticity indicated the evolution of the cement hydration process at these first stages of freeze–thaw cycles, which overcame the freeze–thaw effect due to the phase change in the water inside the material's voids [55]. Moreover, the fibres' role was probably not significant yet since the deterioration process was not fully developed [34]. According to the curves of the mortars for the outdoor exposure, the fs-reinforced samples case displayed the most stable E behaviour regarding the changes amongst subsequent time moments. Furthermore, regarding the dimension deformations, the C′R and C′fs presented higher volume stabilities than the C′pn. Under specific severe exposure conditions, where constant temperature, humidity, ultraviolet (UV) radiation, CO_2 subjection and air changes interacted [47,56], it seemed that the porosity values and the void connections affected the material properties. Hence, the fs-fibre-reinforced mortars, with a similar porosity to the R samples case and the lowest capillary absorption at the age of 90 days (the beginning of the cycles), improved the cement mortar behaviour, while the humidity absorption and movement were probably minimised.

Confirmation of the above formulations was achieved through the examination of the mechanical property values presented in Figure 11. The mortars that were subjected to freeze–thaw cycles displayed, in most cases, advanced E and mechanical properties at the end of the experiment (due to cement hydration), while the porosity decreased due to a reduction in pores/voids [55]. On the other hand, outdoor exposure led to a porosity increment and E reduction, likely as a result of microcrack formations. Nevertheless, the mechanical properties were improved, probably due to the chemical reactions that followed. The moisture increase, as a result of rain during that period, probably contributed to this direction. The highest flexural enhancement was found in the C′pn sample case (30.6%). Concerning the fibre impact, at the end of the freeze–thaw cycles, both reinforced types of composites displayed enhanced flexural strength compared to the C′R composite case, in contrast to outdoor exposure, where only the pn-reinforced samples showed a

corresponding improvement. Furthermore, at the end of both weathering exposures, the reinforced samples showed a lower compressive strength compared to the reference case, which could be attributed to their higher porosity values. Overall, the performance of the fibres through extreme weathering conditions with long-term testing indicated their durability and suitability for their utilisation as cement mortar reinforcements.

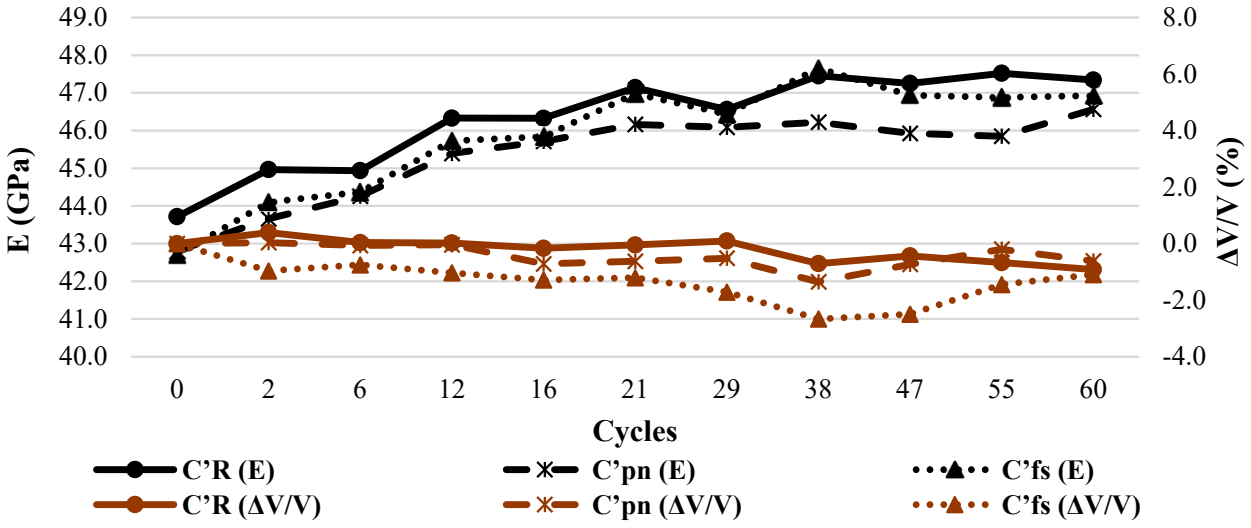

Figure 9. Dynamic moduli of elasticity (E) and volume deformations ($\Delta V/V$) of the produced mortars during freeze–thaw cycles.

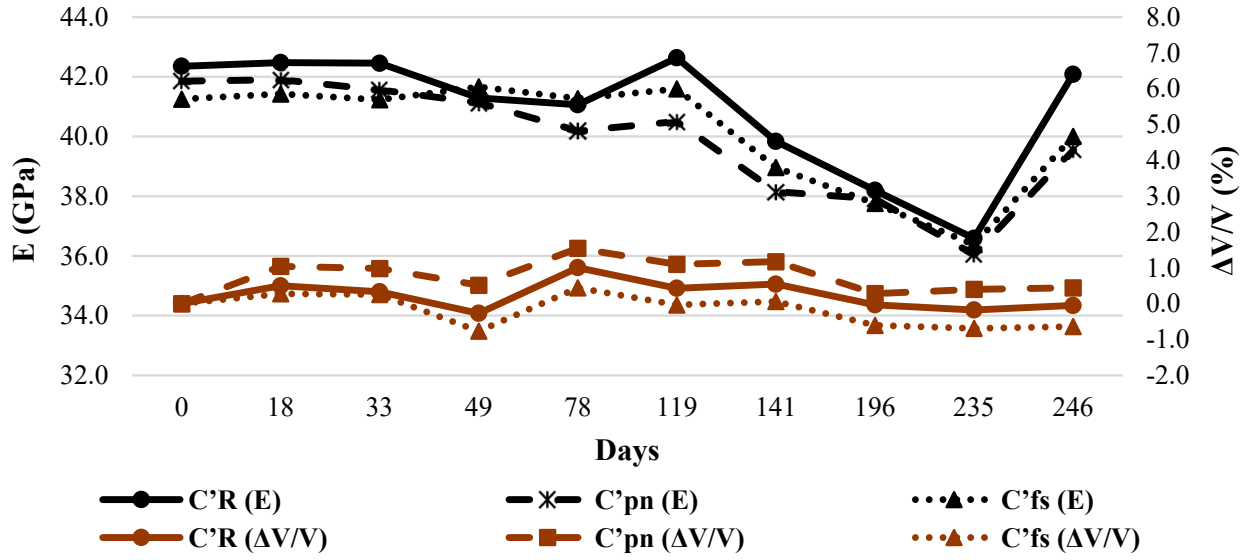

Figure 10. Dynamic moduli of elasticity (E) and volume deformations ($\Delta V/V$) of the produced mortars at days of exposure to outdoor conditions.

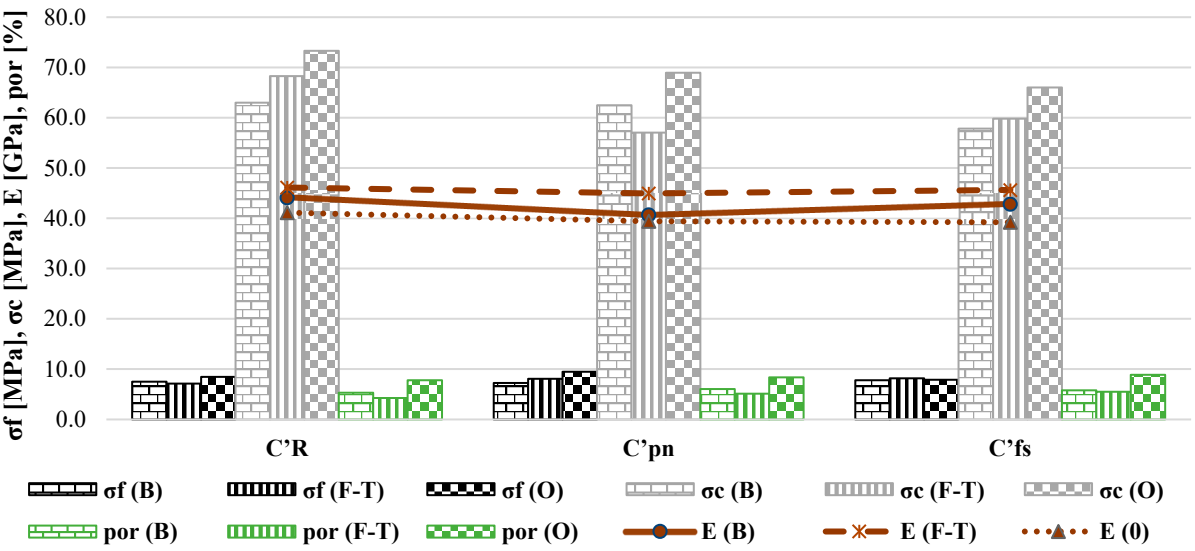

Figure 11. Flexural strengths (σ_f), compressive strengths (σ_c), dynamic moduli of elasticity (E) and porosity (por) of the produced mortars before the start of cycles (B) and at the end of the freeze–thaw cycles (F–T) or outdoor exposure (O).

4. Conclusions

In this current study, black pine and beech wood fibres were exposed to mild hydrothermal treatment and were incorporated into cement mortars. The evaluation of their performance at different ages (after 28, 90 and 365 days of hardening) indicated their beneficial role in dimensional stability, with the black pine fibres presenting the lowest dimensional change, specific gravity and weight. The wood fibres promoted the tendency towards voids formation, with the pn-fibre-reinforced samples maintaining their stable porosity during ageing (in contrast to the other two composite categories). Furthermore, the capillary absorption tendency of the mortars through maturing differed, with the most beneficial mixture being the C'pn at the age of 365 days. The drying curves comparison revealed that all mortar samples became less dense through ageing, and the R and C'pn samples were the densest after 28 and 365 days of hardening, respectively.

Concerning the mechanical properties of the mortars, all specimens presented improved flexural and compressive strength through ageing, and their rigidity improved as well. In regard to the fibres' impact, they provided enhanced flexural strength and contributed to a slightly decreased composite compressive strength. The pn fibres revealed the highest flexural strength improvement (about 5.4%) at the age of 365 days and the lowest compressive reduction from 90 to 365 days of hardening. Moreover, the moduli of elasticity of all specimens was found to be similar after one year.

The specimens preserved their high thermal conductivity values through ageing, and the presence of fibres induced a λ decrease of about 35% (at all maturing times) for the black pine samples and 19% for the beech samples. Furthermore, the wood fibres improved the water vapour permeability of the cement mortars considering the breathability of the material, with the C'pn composite samples displaying the highest μ reduction (about 22.8%).

Under artificial ageing, the wood fibre-reinforced samples displayed a similar change in the E and volume deformation trends, with the reference mortars, the black pine fibres and beech fibres improving the volume stability of the cement mortars under freeze–thaw cycles (and outdoor exposure, respectively). In most cases, at the end of artificial ageing, the mechanical properties of the mortars were enhanced, and their porosity values were decreased in the freeze–thaw cycles and increased under outdoor conditions. Meanwhile, the reinforced mortars presented improved flexural strength (except for the fs case during outdoor exposure) and slightly reduced compressive strength compared to the reference samples.

To sum up, the treated wood fibre-reinforced composites seem to remain integrated, and the fibres performed satisfactorily in alkaline environments, such as those of the studied composites, through all hardening conditions. Considering the minimisation of the building energy consumption and the breathability boost of the structures, the black pine fibres proved to be the most beneficial choice among the two wood species examined in the current study. The FTIR spectra of the fibres revealed a slight decrease in extracts— hemicelluloses and hydroxyl groups that have been induced by the mild hydrothermal treatment—which justifies the beneficial role of the fibres in the cementitious mortar performance. Hence, their utilisation in the construction sector could provide green, sustainable, and low-cost solutions for mortar reinforcements.

Further investigation of the different wood fibre treatments' impact, as well as in terms of the microstructure of the final wood–cement composites and the mortar-substratum adhesion, could contribute to a thorough understanding of the treated wood fibres' role and impact on cement mortars. More research efforts are considered required to identify the optimal fibre treatment conditions that would ensure the enhancement of some critical properties of the fibres while avoiding the deterioration of their mechanical strength, contributing in this way to an enhanced performance of cementitious fibre-reinforced composite materials.

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