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Suitability of Surface-Treated Flax and Hemp Fibers for Concrete Reinforcement

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Abstract: The use of vegetable fibres as a sustainable alternative to non-natural sources of fibres applied for concrete reinforcement has been studied for over three decades. The main issues about plant-based fibres pointed out by other authors are the variability in their properties and concerns about potential high biodegradability in the alkaline pH of the concrete matrix. Aiming to minimise the variability of flax and hemp fibres, this research compares a range of chemical surface treatments, analysing their effects on the behaviour of the fibres and the effects of their addition to concrete. Corroborating what has been found by other authors, the treatment using NaOH 10% for 24 h was able to enhance the properties of hemp fibre-reinforced concrete and reduce the degradability in alkaline solution. For flax fibres, a novel alternative stood out: treatment using 1% of stearic acid in ethanol for 4 h. Treatment using this solution increased the tensile by 101%, causing a minor effect on the elastic modulus. Concrete mixes reinforced with the treated flax fibres presented reduced thermal conductivity and elastic modulus and increased residual tensile strength and fracture energy.

Keywords: production of vegetable fibre reinforced concrete (FRC); extraction of flax and hemp fibres; surface treatment of vegetable fibres; degradability of plant fibres into the alkaline matrix; mechanical properties of FRC; fracture energy of natural fibre reinforced concrete

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1. Introduction

Natural fibres are widely studied to be used as an ecological replacement for non-natural fibres for the reinforcement of composites. Their applications include, among many, the automotive, polymer, and construction industries and they can be classified according to their source of extraction, usually as animal, mineral and vegetable [1–4].

The advantages of vegetable fibres over others include abundant sources of raw materials and reduction of CO_2 emissions, low density, good mechanical and physical properties, low cost of production and higher biodegradability [5,6]. On the other hand, their biodegradability can also be considered an issue regarding the application of vegetable fibres in composites produced to offer a longer lifespan, such as concrete [7,8]. Considering their availability in Ireland, flax and hemp fibres were the vegetable fibres selected for this study. As mineral fibres, basalt fibres were also studied. To avoid possible animal cruelty, fibres from animal sources were not included.

Hemp is becoming a common source of fibres in the construction industry. Hemp fibre-reinforced concrete (HFRC) is a concrete matrix with fibres incorporated into the mixture. Another similar application is hempcrete, a lime-based binder, with the addition of modified or unmodified hemp fibres to enhance properties such as heat insulation, weight and embodied carbon reduction, etc. [9–11]. Shang and Tariku (2021) [11] estimated the environmental impact of composites using hemp fibres. They experimentally found that the thermal mass of hemp concrete increases the heating energy consumption and reduces the cooling energy consumption due to smaller solar heat gain. According to the authors, for mild and cold climates, most of the cooling energy savings compensated

the increase in heating energy consumption. Compared to the savings on the embodied carbon emission, the difference in the energy consumption was small, showing that the emissions of the hemp concrete building with a lifespan of 50 years would be reduced by 23.2% and 9.9% for cooling and heating energy consumption, respectively. For regions with well-defined seasons, it would be a good alternative, but for countries that require more heating than cooling, the use of hemp fibre-reinforced concrete (HRFC) would not be as effective for thermal insulation isolated [11]. For that reason, it was decided to compare the mechanical properties of hemp with flax fibres as a possible alternative for countries that require more heating energy than cooling.

Flax fibres are part of the stem on the flax bast plant, they are largely available in Europe, and their use is already common in the automotive industry. Like cotton, flax fibre is a cellulose polymer but with a more crystalline structure, which makes it stronger and harder, but also more brittle and prone to wrinkling [12,13].

Considering the well-known positive outcome obtained by the enlisted authors using hemp fibres for cementitious composites and the promising mechanical properties obtained by the scientific community for flax fibres, as well as their large availability in Ireland and UK, this research aims to compare different surface treatments on flax and hemp fibres, analyse their effects on the degradability of the fibres, and verify the impact of their use as reinforcement of concrete or filler material, comparing their values with basalt and more well-established polypropylene fibres.

Following the initial characterisation of the virgin natural fibres, the most efficient surface treatment for each fibre was selected, aiming to increase the tensile strength and/or reduce the elastic modulus of the material. More pertinently, the variability in fibre properties and their long-term resistance to aggressive environments was addressed. To reduce biodegradability and variability and to improve the physical and mechanical properties of vegetable fibres, different surface treatments were suggested by Sood and Dwivedi (2018) [14]. The reduction of biodegradability occurs because the chemicals remove oils, wax, and impurities from the external layers of the fibres, exposing layers of hemicellulose and pectin, allowing their decomposition and improving the mechanical properties of the fibres as the cellulose reminiscent is responsible for the tensile strength [15]. However, treatments longer than 36 h allow the depolymerisation of secondary layers of the fibres, reducing their mechanical performance [16].

Different surface treatments can be conducted to remove the hemicellulose, waxes, and pectin while keeping the cellulose and the lignin, the structural parts of the plant. Page et al. (2021) [17] affirm that another advantage of certain surface treatments is that they can create a barrier of protection against the high pH of the matrix, such as concrete. The normal pH of concrete goes from 12.5 to 13, and it tends to get reduced with deterioration [18]. As an alkaline environment, it can be highly aggressive to untreated vegetable fibres [19]. In order to ameliorate the deterioration caused by such an aggressive environment, different chemicals have been used for surface treatment. A drawback to many of these, however, is that they can be hazardous to the wider environment, both in their production and use. Seeking a sustainable alternative, different researchers have been studying less hazardous chemicals. Recently, Page et al. (2021) [17] verified the use of a fatty acid could work to protect flax, where they used linseed oil and found that this treatment can also improve the workability and flexural strength of concrete containing treated fibres.

The chemicals used in this study and associated hazards are represented in Table 1. As shown, among the solutes, stearic acid is the only chemical that does not present a clear hazard (the hazard is associated with the solvent, ethanol). Similar to stearic acid in ethanol, the solution containing EDTA also presents fewer hazards but does present a health hazard with the possibility of causing respiratory irritation when inhaled. Nonetheless, after performing a risk assessment of the treatments, it is possible to safely proceed with the evaluation of the properties of the fibres.

The characterisation of mechanical, chemical, and physical properties of materials is important to understand and predict their behaviour for the purpose of use. Density,

diameter, and physical appearance are physical properties to be easily assessed from the fibres, while tensile strength, elongation, and elastic modulus represent relevant mechanical properties to be verified. To estimate the chemical composition, different analyses can be adopted, including FTIR. From the peaks obtained on the spectrums, it is possible to assess the presence of cellulose, hemicellulose, lignin, waxes, etc. [14,19–21].

Table 1. Treatment solutions suggested by M. Sood and G. Dwivedi [14,22].

Treatment/Solute	Hazard (s)	Solvent	Hazard (s)	Concentration
Alkali (NaOH)	None	Water	None	5%, 10%, 15%
Potassium Permanganate ($KMnO_4$)		Acetone		0.05%
Stearic Acid ($C_{18}H_{36}O_2$)		Ethanol		1.0%
EDTA ($C_{10}H_{16}N2O_8$)		Water		5 g/L

Dai and Fan (2017) [23] correlated the presence of vegetable fibre compounds with their vibration regarding the peak of transmittance at certain wavenumbers, as presented in Table 2.

Table 2. Main infrared transition for hemp fibre [23].

Wavenumber [cm^{-1}]	Vibration	Sources
3336	OH stretching	Cellulose, Hemicellulose
2887	C–H symmetrical stretching	Cellulose, Hemicellulose
1729	C=O stretching vibration	Pectin, Waxes
1623	OH bending of absorbed water	Water
1506	C=C aromatic symmetrical stretching	Lignin
1423	HCH and OCH in-plane bending vibration	Cellulose
1368	In-the-plane CH bending	Cellulose, Hemicellulose
1317	CH2 rocking vibration	Cellulose
1246	C=O and G ring stretching	Lignin
1202	C-O-C symmetric stretching	Cellulose, Hemicellulose
1155	C-O-C asymmetrical stretching	Cellulose, Hemicellulose
1048	C-C, C-OH, C-H ring and side group vibrations	Cellulose, Hemicellulose
1019	C-C, C-OH, C-H ring and side group vibrations	Cellulose, Hemicellulose
995	C-C, C-OH, C-H ring and side group vibrations	Cellulose, Hemicellulose
896	COC, CCO and CCH deformation and stretching	Cellulose
662	C-OH out-of-plane bending	Cellulose

This research is an extension of a study previously published by the authors [24–26] where a preliminary characterisation of different fibre FRC mixes was performed, and now, the continuation of works is detailed. Flax and hemp fibres were the vegetable fibres selected after a viability study considering their availability in Ireland. As a benchmark, basalt and polypropylene were included. Initially, steel fibres were also adopted, but the behaviour of vegetable fibres tends to be more similar to the synthetic fibres, and steel FRC was not included in the continuation of the study.

Therefore, this research aims to compare the effects of different chemical surface treatments on the properties of flax and hemp fibres, untreated and treated, and the differences in performance of various properties of fibre-reinforced concrete. The degradation of untreated and treated fibres in an alkaline solution was also evaluated.

2. Materials and Methods

Hemp fibres were acquired as ropes (Figure 1a), manually separated, and cut into 40 mm lengths (Figure 1b). The flax fibres were bought as retted straws (Figure 1c) and were also manually extracted, separated, and chopped into 40 mm lengths (Figure 1d).

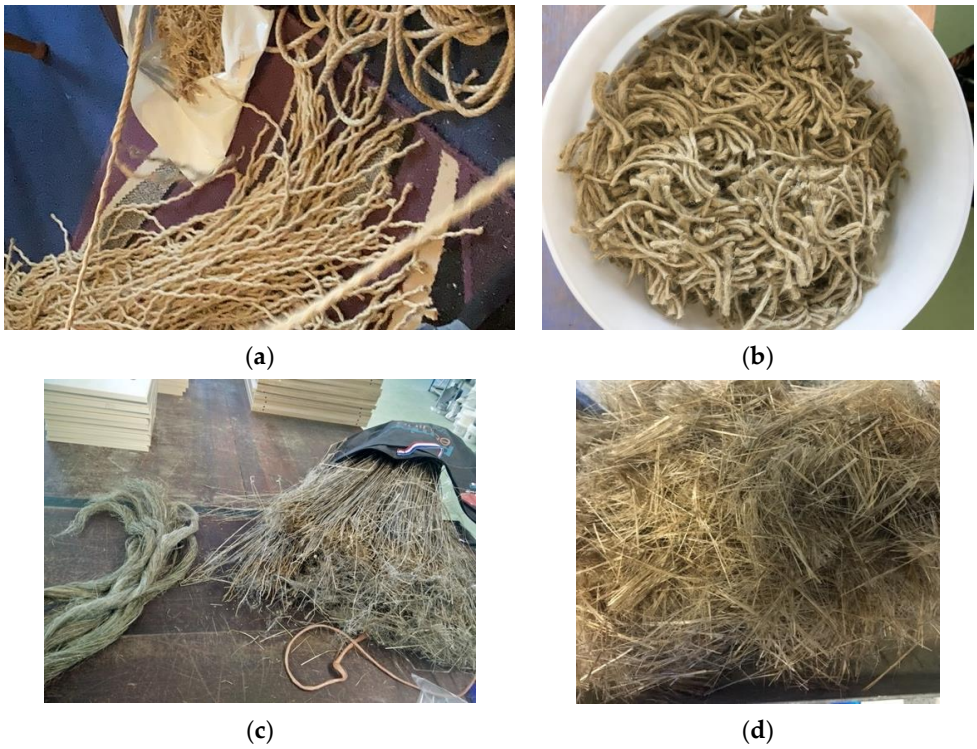


Figure 1. Fibres during separation phase: (**a**) hemp, (**c**) flax. Fibres chopped (**b**) hemp, (**d**) flax.

2.1. Vegetable Fibres

This section details the methodology adopted to submit the selected vegetable fibres for chemical surface treatment and to have some of their properties evaluated. To be able to discuss and compare the results in the following section, the preliminary study was conducted [26], and the fundamental content for this analysis is summarised in the following section.

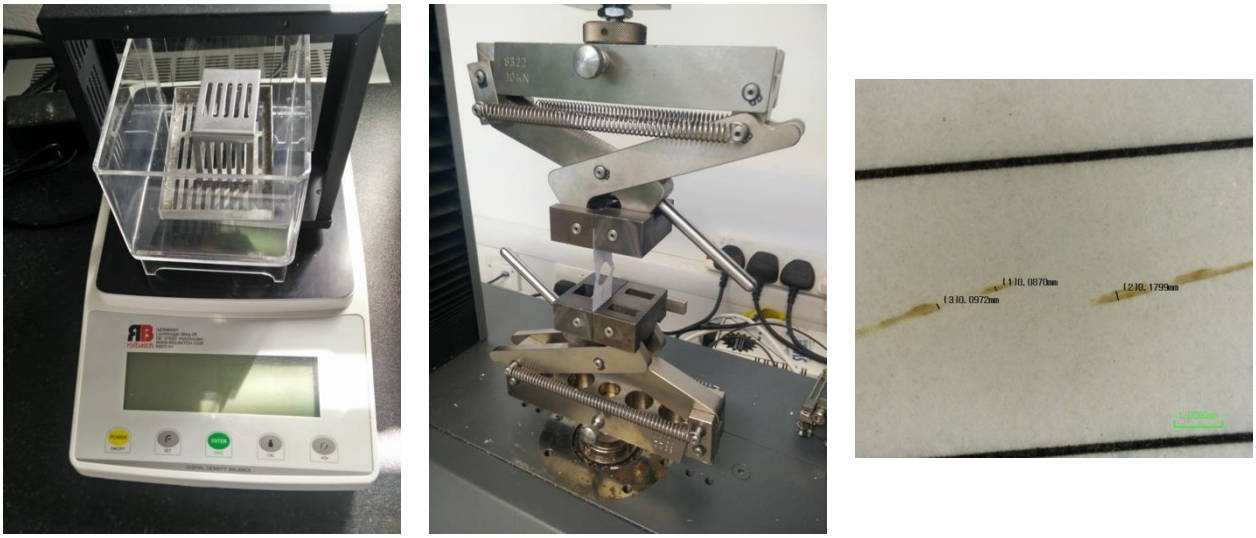
2.1.1. Untreated Fibres Analysis: Preliminary Study

Once ready, representative samples of the fibres were separated, and their densities were measured using a digital balance (rolbatch RBDT-01). Secondly, specimens of hemp and flax fibres were made, following the parameters given by the Standard Test Method for Tensile Strength and Young's Modulus of Fibres (ASTM C1557) [27]. Each sample containing 40 specimens was tested by attaching a single 40 mm long fibre into a paper. Using a tensiometer machine, the stress and elongation of each specimen were tested until it broke. The digital balance and a tab being tested are shown in Figure 2a,b, respectively [26].

Following the rupture, the diameters of the fibres were measured more than once close to the broken part, as recommended by ASTM C1557, using a digital microscope (Figure 2).

As the vegetable fibres presented significantly lower properties in comparison to basalt, surface treatment can be considered as an option to enhance their behaviour.

The individual mean value was adopted to calculate the tension at the breaking point, Young's modulus (elastic's modulus) (E), and elongation at break (ε%), and the results are presented in Table 3. They will be considered when analysing the effects of the surface treatments on the flax and hemp fibres.



(**a**) (**b**) (**c**)

Figure 2. (**a**) Digital balance (**b**) specimen in between grips during the tensile test (**c**) diameter of fibres measured after rupture.

Table 3. Mechanical and physical properties of natural fibres [26].

Material	Diameter [μm]	Density [g/cm^3]	Tensile Strength [N/mm^2]	E [GPa]	ε%
Basalt	14.0	2.2	2546.00	136.18	1.67%
Flax	82.4	1.1	865.96	40.78	2.09%
Hemp	73.0	1.1	262.68	22.44	1.47%

2.1.2. Initial FRC Mixture

The initial control mix was designed following the ACI method, and the quantity of each material is detailed in Table 4. The target compressive strength was 32 MPa (including 8% of standard deviation and 2.5 N/mm^2 of margin, slump 10–30 mm).

Table 4. Concrete mix designed for the preliminary characterisation.

Material	Quantities in kg Per m^3 (to the Nearest 5 kg)	Fibre	% of Volume
Cement	385	Basalt	1.0%, 0.5%
Water	170	Flax	0.5%
Fine aggregate	585	Hemp	1.0%, 0.5%
Coarse aggregate (10 mm)	415	Polypropylene	1.0%, 0.5%
Coarse aggregate (25 mm)	830	Steel	0.1%, 0.05% and 0.025%

2.2. Surface Treatment and Mechanical Properties of the Fibres

From a list of surface treatments for vegetable fibres suggested by Sood and Dwivedi [14], different types of solutes and solvents were selected for a broad view of their effects. Among all the options mentioned, the following solutions were chosen: an acid saline (EDTA), an acid oxide (KmnO4), a fatty acid (stearic acid), and an alkali solution (NaOH) in different concentrations. Each solution was produced as presented in Table 1. To avoid damage to the internal layers of the structure of the fibres, as pointed out by Nayak (2020) [16], the samples of hemp and flax fibres were treated for 4 h, 6 h, 10 h, 15 h, and 24 h.

Once all the solutions were prepared, individual samples of the fibres were fully submerged into separate beakers (Figure 3a,b). When each time studied was reached,

the solutions were neutralised, washed (Figure 3c), and left to dry in the oven at 60 °C (Figure 3d) for 24 h. Once dry, the fibres were separated, and new samples were made containing 17 specimens of single fibres of 40 mm length and had their mechanical properties evaluated following the same procedure previously described.

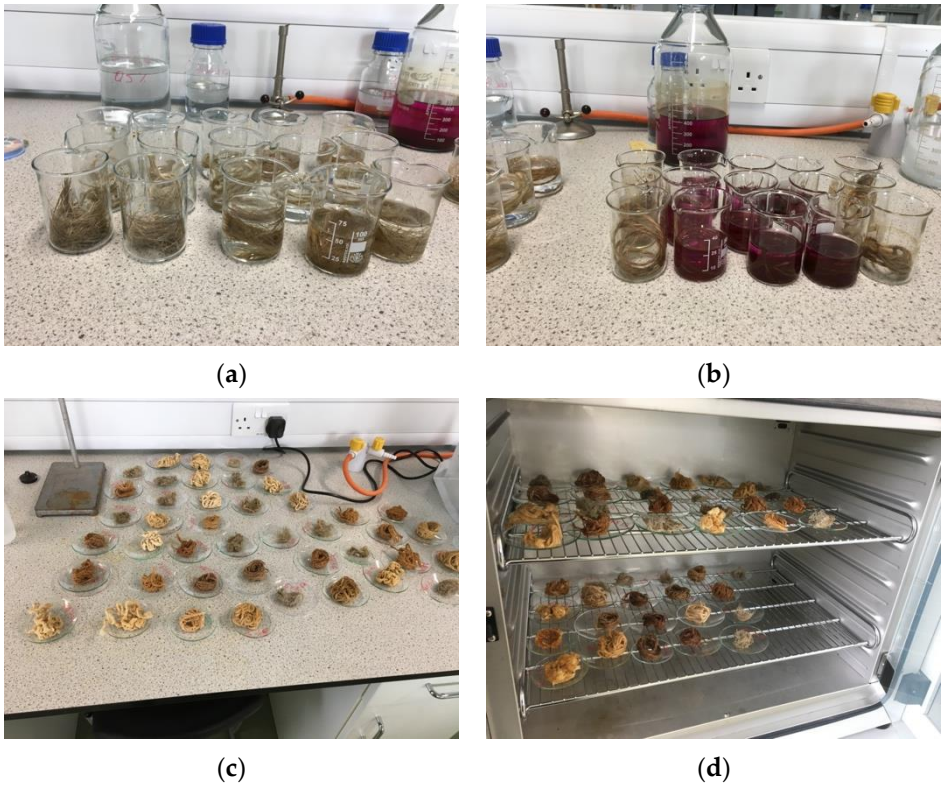


Figure 3. Surface treatments conducted on the fibres: (**a**) Fibres under surface treatments, (**b**) Fibres under surface treatments, (**c**) Fibres neutralised and ready to dry and (**d**) Fibres in the oven.

The results obtained are presented in the next section. The fatty-acid solution with stearic acid was adopted considering the study from Page et al. (2021) [17] and for being a chemical present nature. Analysing the effects of all treatments, it was then possible to select the surface treatments for those fibres to be used as concrete reinforcement. The parameters adopted are also explained in Section 3.

2.3. Degradability Test of Fibres in Alkaline Solution

To better understand the behaviour of the treated fibres in the concrete mixture, an experiment was carried out to evaluate their degradability in an alkali environment. As no specific standard procedure for this test was found, it was decided to adapt the methodology described by the ASTM D6942-03 (2019) "Standard Test Method for Stability of Cellulose Fibers in Alkaline Environments" [28]. Since the whole fibres were the object of interest, instead of working with the pulp of fibres, 10 g of the whole fibres were used, and 23.3 g of 1N NaOH (pH 14) was added. Corresponding to a consistency of 30%, 10 g fibres was used, totalising 33.3 g.

Samples were immersed in the alkaline solution for 3, 7, 14, 21, and 28 days, respectively. Upon completion of their respective immersion periods, the samples were removed, and their tensile strength was measured. In addition, those immersed for 28 days were examined using SEM prior to tensile testing.

2.3.1. FTIR

To estimate the effects of each treatment on the composition of the fibres, they were analysed using a PerkinElmer Spectrum One FTIR Spectrometer, as shown in Figure 4. Results and discussions are provided in the following sections of this paper.

Figure 4. FTIR Spectrometer.

2.3.2. SEM Images

To be able to compare the aspects of flax and hemp fibres in the natural and degraded forms as well as the effects of the surface treatment on them, SEM images were made and are presented in Section 3.

2.4. Vegetable Fibre Reinforced Concrete

From the initial study, it was observed that having the maximum size of aggregate as 25 mm would limit the thickness of the structures produced, not allowing a possible usage for hollow concrete blocks of thinner panels, so for this part of the study, a new mix was designed where the coarse aggregate size was limited to 10 mm.

The mix studied here is an optimisation of a previous study (mix 1) [26] using a concrete mixture containing 1:1.53:1.08:2.16:0.44 [cement]:[fine = aggregate]:[coarse aggregate 10 mm]:[w/c ratio].The objectives were to reduce the maximum nominal size of the coarse aggregate and to increase the mix workability by adopting a higher w/c factor.

The concrete mixture (mix 2) was designed following the ACI method. As in the initial experiment, the target compressive strength was again 32 N/mm^2 at 28 days, with a maximum size of coarse aggregate of 10 mm. Choosing a smaller coarse aggregate size, the mixture could have different applications depending on their behaviour, including the production of masonry blocks, thinner precast, and 3D printed structures. The water/cement ratio was reduced from 0.8 to 0.5 to avoid shrinkage and cracking [29,30]. The proportion calculated was 1:1.78:2.16:0.5 [cement]:[fine aggregate]:[coarse aggregate 10 mm]:[w/c ratio].

The designed slump was 30–60 mm, as it was expected that the fibres would absorb moisture from the mixture.

Following the preliminary part of this work as a reference for starting quantities, the initial proportion of fibres per volume was 0.5%. Different authors suggest the addition of 1% or less of natural fibres into a concrete mix [21,26,31,32]. For flax fibres, the same amount was also adopted. However, the workability was strongly affected. Therefore, mixes containing 0.35% and 0.25% of flax fibres were made, focusing on obtaining a homogeneous, workable, and mouldable mixture.

The initial mixtures produced were:

i. Control, no addition of fibres
ii. Basalt fibres (48 mm)—0.5%
iii. Hemp Fibres—0.5%
iv. Flax fibres—0.5%
v. Flax fibres—0.35%
vi. Flax fibres—0.25%

Aiming for the optimisation of the workability of a flax-reinforced mixture, two other mixes were produced. As the mix containing 0.25% presented a satisfactory slump, the mix was repeated to verify their compressive strength at both 7 and 28 days.

As a benchmark, mixtures containing polypropylene and basalt fibres were also tested for compressive strength. The flexural strength of the polypropylene FRC mixes was reported elsewhere [24] and is used again here for comparison.

2.4.1. Compressive Strength (f_{cu})

Following the standards EN 12390-1/2/3 [33–35], 6 cubes were moulded measuring 100 × 100 × 100 mm for each mixture, 3 to have their compressive strength tested at 7 days and 3 to be tested at 28 days.

2.4.2. Fracture Energy (Gf), Young's Modulus€ and Residual Flexural Tensile Strength(f)

Although metallic fibres were not part of this study, the parameters from the standard EN 14651 (2012) Test method for metallic fibre concrete—Measuring the flexural tensile strength (limit of proportionality (LOP), residual)" [36] were followed as recommended by the IS EN 14845-2:2007 "Test Methods For Fibres In Concrete—Part 2: Effect On Concrete" [37].

Hence, 6 beams measuring 15 × 15 × 550 mm were produced to have their flexural tensile strength tested at 7 and 28 days. From the following methodology, it was possible to experimentally measure from the EN14651 the flexural tensile strength and calculate the fracture energy (Gf), Young's modulus € and residual flexural tensile strength (f) as described above.

To better understand how these properties are calculated, the measurements adopted on each equation are detailed in Figure 5, and the fracture energy (Gf) can be calculated through Equation (1) and the elastic module (E) through Equation (3).

$$G_f = \frac{W_0 + mg\delta}{A} \quad (1)$$

where W_0 is the area below the curve on the Load [N] × Diffraction [m] diagram, m is the mass of the specimen (kg), g is the gravity acceleration [m/s^2], δ represents the deflection at the final failure of the specimen [m], and A the area [m^2] calculated by Equation (2).

$$A = b(d - a_0) \quad (2)$$

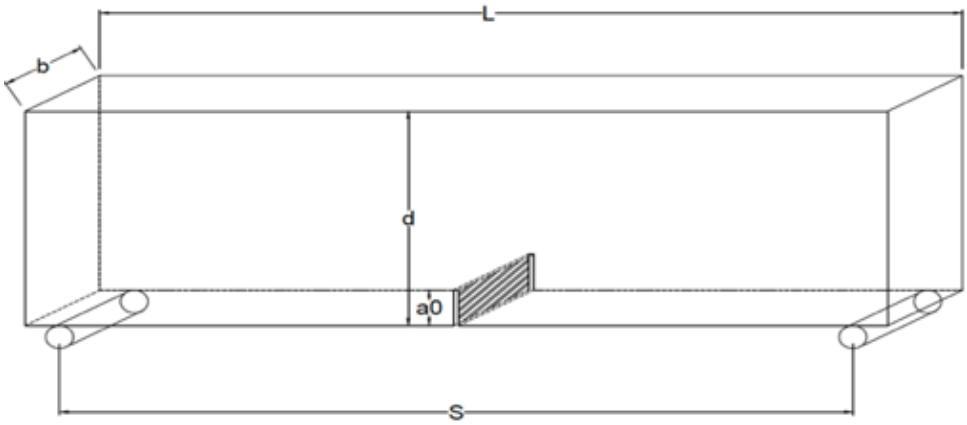


Figure 5. Details of notched beam specimen.

The elastic modulus, or Young's modulus, represents the stiffness of materials. Higher values indicate more brittleness, while lower values represent ductile materials.

$$E = \frac{6Sa_0V_1(\alpha)}{Cib(d^2)} \tag{3}$$

where V_1 and α can be obtained from Equations (4) and (5)

$$V_1(\alpha) = 0.76 - 2.38\alpha + 3.87\alpha^2 - 2.04\alpha^3 + \frac{0.66}{(1-\alpha)^2} \tag{4}$$

$$\alpha = \frac{a_0}{d} \tag{5}$$

Flexural Strength (F_L), (F_n) [MPa]

Following EN14651, the limit of proportionality (LOP) is calculated from Equation (6). For the peak strength (F_n), F_L is the value of the peak load [N], and for the residual strength at net deflections, F_j is the correspondent load [N] at specific deflection δ_j(N) points and used to calculate the residual flexural tensile strength ($f_{R,j}$) as shown in Equation (7).

$$F_n = LOP = \frac{3F_LS}{2b(d-a_0)^2} \tag{6}$$

$$F_{R,j} = \frac{3F_jS}{2b(d-a_0)^2} \tag{7}$$

where S is the span, b is the width measurements from Figure 5 and peak strength (F_n), F_L is the value of the peak load [N], and for the residual strength ($F_{R,j}$) at net deflections, F_j is the correspondent load [N] at specific j deflection points.

Values of F_L and F_j were extracted from the load vs deflection points obtained from the 3-point bending tests; a_0, b, and d from measurements taken using a Vanier calliper for each specimen, and values for flexural strength at the peak load and the residual strength were calculated for net deflections j, from Table 5.

Table 5. Correlation between CMOD and deflection [36,38].

j	CMOD (mm)	δ (mm)
1	0.05	0.08
2	0.1	0.13
3	0.2	0.21
4	0.5	0.47
5	1.05	1.32
6	2.5	2.17
7	3.5	3.02
8	4.0	3.44

The residual strength was calculated following the NF EN 14651, and as mentioned, the diagrams used were load vs deflection instead of CMOD, respecting the parameters established on the standard and following the procedure previously detailed in the literature review.

2.4.3. Thermal Conductivity

To assess and compare the impact caused by the fibre reinforcement in the heat transfer, a slab measuring 50 × 300 × 300 mm for each mixture was studied. The procedure followed was the ISO 8301 (1991) [37] and the manual for "Thermal Conductivity of Building Materials" [39]. Each slab was tested for at least 3 h through the steady boxes method.

Thermal resistance was calculated based on readings taken every minute throughout the test.

Figure 6 shows the equipment used to obtain the thermal conductivity by the boxes method works according to Bala and Gupta (2021) [40]. On the top of the box, there is a hot plate and, on the bottom, a cold plate. Their temperatures are controlled by an electric heater, and the cold plate uses water to keep its temperature steady.

Figure 6. Equipment used to measure thermal conductivity.

Thermal conductivity represents how heat is transferred from one surface of the specimen to the other. Each specimen was placed in between 2 plates, and the temperature was increased on one side of it. The temperature difference was measured by the equipment on the opposite plate every minute.

Having the thickness (l_s) [m] of each specimen measured, the thermal conductivity (λ) $[W/m^2K]$ is calculated using Equation (8).

$$\lambda = fe\frac{l_s}{\Delta T} \tag{8}$$

where f is the equipment calibration factor and e is the heat flow meter output $[mV/m^2]$. Results are presented in Section 3.

2.4.4. Water Penetration

To measure water penetration depth, 3 cubes measuring 150 × 150 × 150 mm were moulded and cured for 28 days. The standard method followed was the I. S. E. 12390-8:2009 "Testing hardened concrete—Part 8: Depth of penetration of water under pressure" [41].

The cubes were placed into the equipment, where pressurised water was forced into one surface of the cubes for 72 h. After this period, the cubes were split in two, using a compressive one-point load applied on the centre of each cube, as shown in the sketch from Figure 7a. After having them divided, the water penetration depth (p) was measured using a vernier calliper Figure 7b.

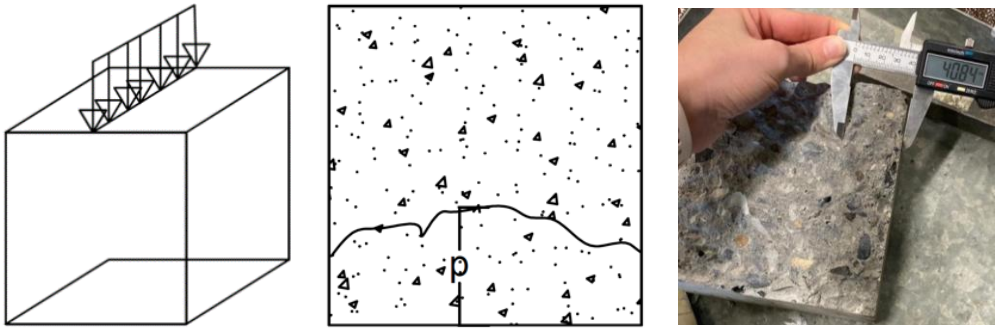


Figure 7. (**a**) Load distribution model used to split the cubes, (**b**) Highest line of water penetration (**c**) water penetration measurement.

2.4.5. SEM Images

To analyse the microscopic differences throughout the time on the internal aspect of the concrete surface of the proposed mix containing 0.25% of flax fibres, SEM images were made from samples of it after 28 days and over 90 days.

3. Results and Analysis

3.1. Vegetable Fibres

To compare the effects on mechanical properties of the fibres on the different surface treatments adopted, results obtained for maximum tensile strength (MPa), elastic modulus (GPa), elongation at break, and diameter (mm) are detailed and discussed ahead.

3.1.1. Chemical Surface Treatment

Each sample treated was tested under the same methodology described previously. Values for maximum tensile strength and elastic modulus are presented in Figures 8 and 9 for flax fibres and Figures 10 and 11 for hemp fibres. In each graph, the line parallel to the *x*-axis represents the values for untreated fibres from Table 3.

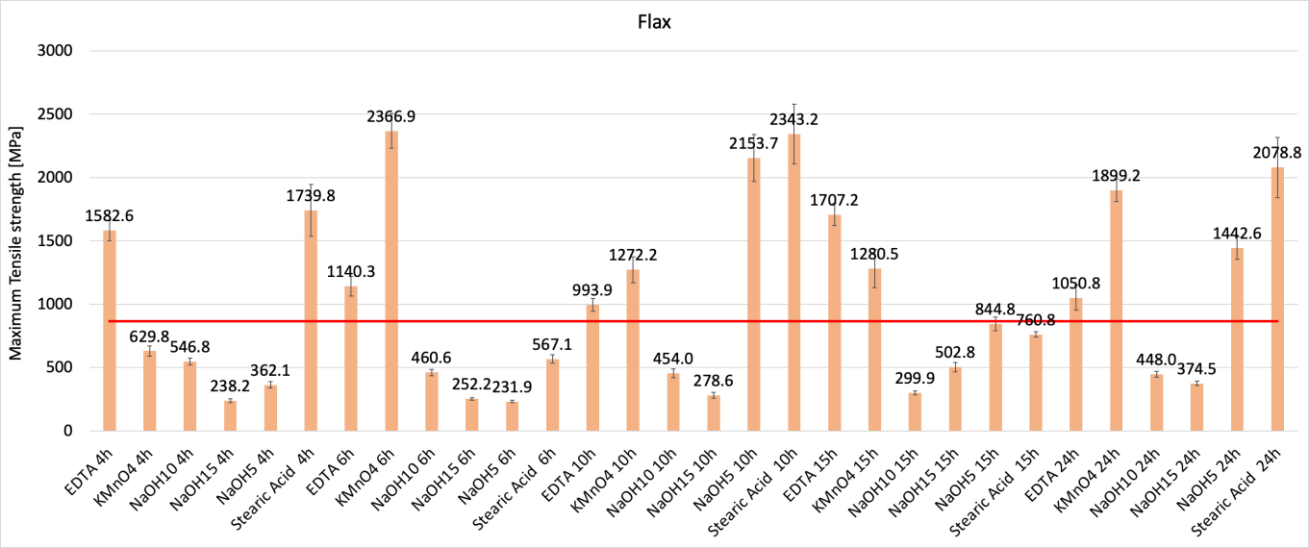


Figure 8. Maximum tensile strength obtained for each treatment on flax fibres.

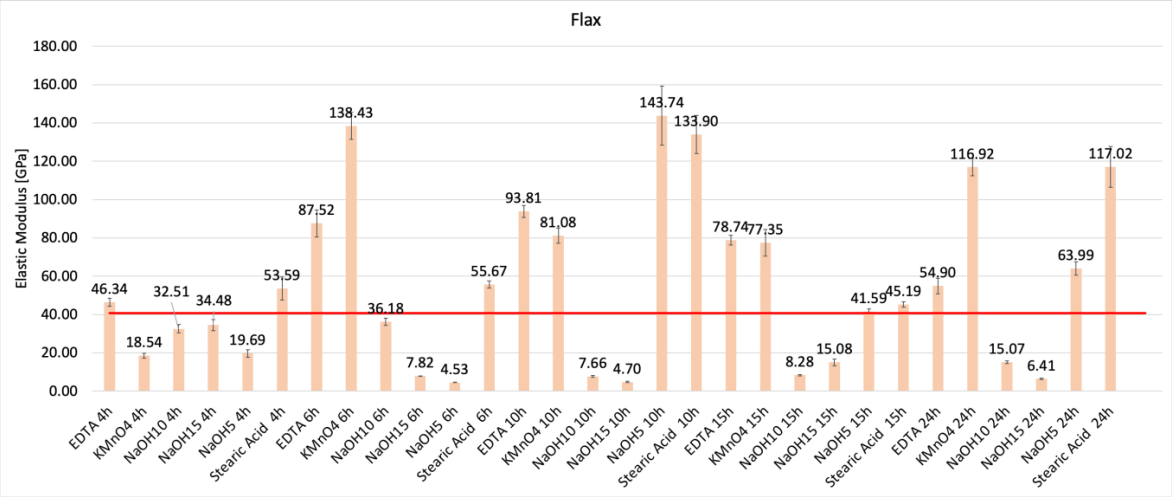


Figure 9. Elastic modulus obtained for each treatment on flax fibres.

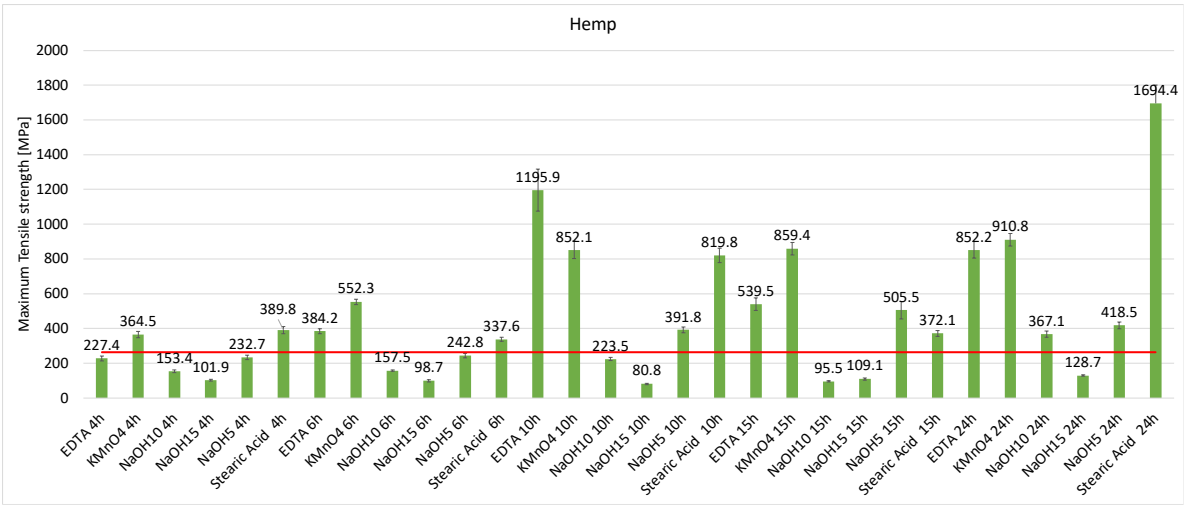


Figure 10. Maximum tensile strength obtained for each treatment on hemp fibres.

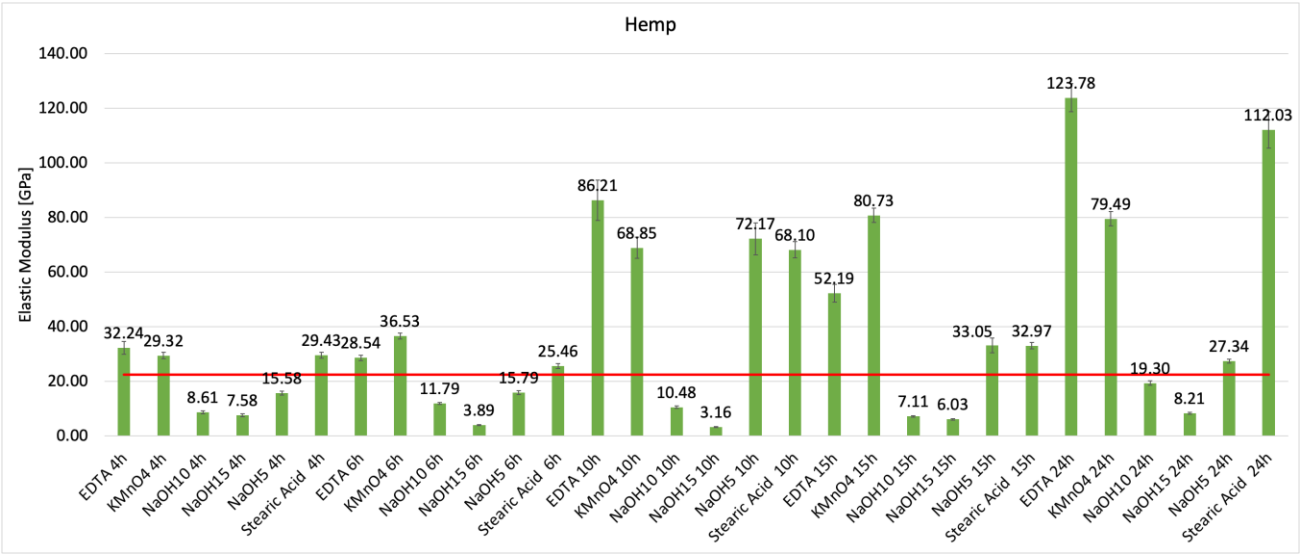


Figure 11. Elastic modulus obtained for each treatment on hemp fibres.

Analysing the results from flax fibres from the first graph, it is possible to see that the treatments with EDTA (4 h), stearic acid (4 h), EDTA (6 h), $KMnO_4$ (6 h), EDTA (10 h), $KmnO_4$ (10 h), NaOH 5% (10 h), EDTA (15 h), $KmnO_4$ (15 h), EDTA (24 h), $KmnO_4$ (24 h), NaOH 5% (24 h), and stearic acid (24 h) increased the tensile strength of the fibres.

Compared to Figure 9, none of these treatments reduced the elastic modulus. The two treatments that enhanced the tensile strength with a lower increase in the elastic modulus were the treatments with EDTA (4 h) and stearic acid (4 h).

EDTA (4 h) increased the tensile strength by 82.7% and the elastic modulus by 13.6%, while stearic acid (4 h) doubled the tensile strength value (200%), increasing the elastic modulus by 31.3%.

Regarding their hazardousness, going back to Table 1, EDTA presents an exclamation mark and health hazard, while stearic acid presents none. However, the second requires ethanol as a flammable solvent substance also with an exclamation mark. As treatments presented similar behaviour, the study conducted by Page et al. (2021) [17] using linseed oil was taken into consideration, and stearic acid was selected for being a fatty-acid chemical, with use potentially positive for the intended application.

In a similar analysis, but for hemp fibres, the following surface treatments improved the tensile strength: $KMnO_4$ (4 h), stearic acid (4 h), EDTA (6 h), $KMnO_4$ (6 h), stearic acid (6 h), EDTA (10 h), $KMnO_4$ (10 h), NaOH 5% (10 h), stearic acid (10 h), EDTA (15 h), $KMnO_4$ (15 h), NaOH 5% (15 h), stearic acid (15 h), EDTA (24 h), $KMnO_4$ (24 h), NaOH 10% (24 h), NaOH 5% (24 h), stearic acid (24 h).

Among those, only the treatment using NaOH 10% 24 h was able to increase the tensile strength and reduce the elastic modulus. Treatments using NaOH are commonly suggested by authors, as seen in the literature review, and for that reason, this treatment was selected for hemp fibres.

Therefore, the treatment using NaOH 5% 15 h was selected, as it increased the original tensile strength by 92% while affecting the elastic modulus by just 2.5% compared to the EDTA option.

For flax fibres, EDTA 4 h also presented similar effects from the sample containing hemp fibres. However, Stearic Acid 4 h stood out in this analysis as it increased the tensile by 101%, causing an increase in the elastic modulus of 31%. Apart from that, it is important to mention that a stearic acid solution is also a greener option as it is a natural saturated fatty acid [42].

Spectroscopic analysis is suggested by authors to estimate the chemical composition of vegetable fibres and FTIR was the test method adopted in this research. From the spectrums obtained, it was possible to identify the presence of cellulose, hemicellulose, lignin, and waxes according to the peak of their transmittance at a certain wavenumber [14,20,30].

Analysing the effects of the two selected treatments (stearic acid 4 h for flax and NaOH10% 24 h for hemp) on the chemical structure of the fibres, it was possible to observe an increase in the presence of cellulose and hemicellulose on the wavenumber 3336 cm^{-1}, representing an OH stretching according to Table 2. For flax, there was a reduction in pectin, waxes, water, and lignin, while for hemp all the compounds investigated were reduced except for pectin and waxes. For that same reason the elastic modulus also is affected, as treated fibres lose lignin, waxes, and hemicellulose that offer them elasticity.

Confirming what was suggested by Sood and Dwivedi [14] and Nayak (2020) [16], certain treatments were able to significantly increase the tensile strength of vegetable fibres by removing organic compounds and coating the layers of cellulose. Results obtained could improve the tensile strength, but they were still significantly lower than results found by Nayak (2020), who obtained tensile strength values for treated singular flax fibres up from 5705 MPa to 14,103 MPa.

Figure 12 brings the spectrums obtained for flax and hemp fibres before and after the selected surface treatments.

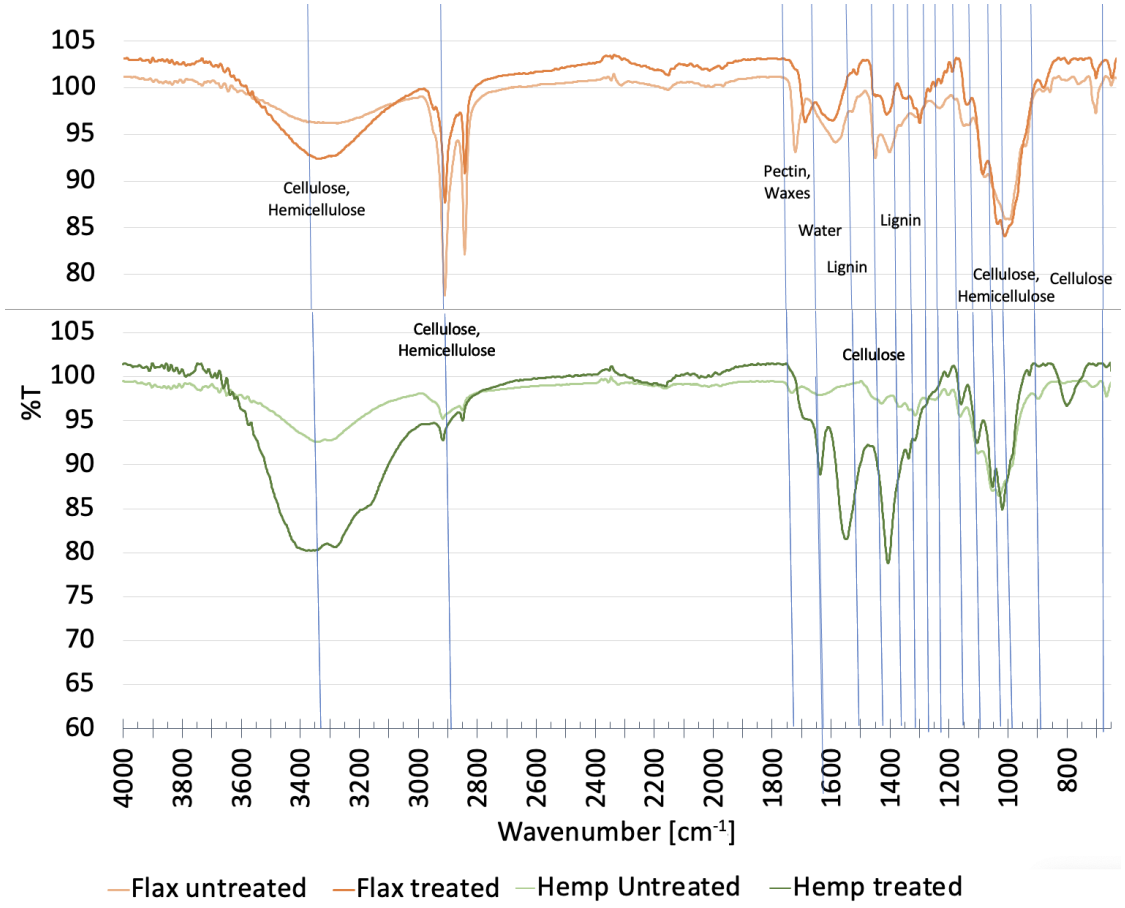


Figure 12. FTIR spectrums obtained for untreated and treated flax and hemp fibres.

3.1.2. Degradation

The degradability in an alkaline environment was estimated for samples containing 15 specimens of treated and untreated fibres. The results are presented in Figures 13 and 14.

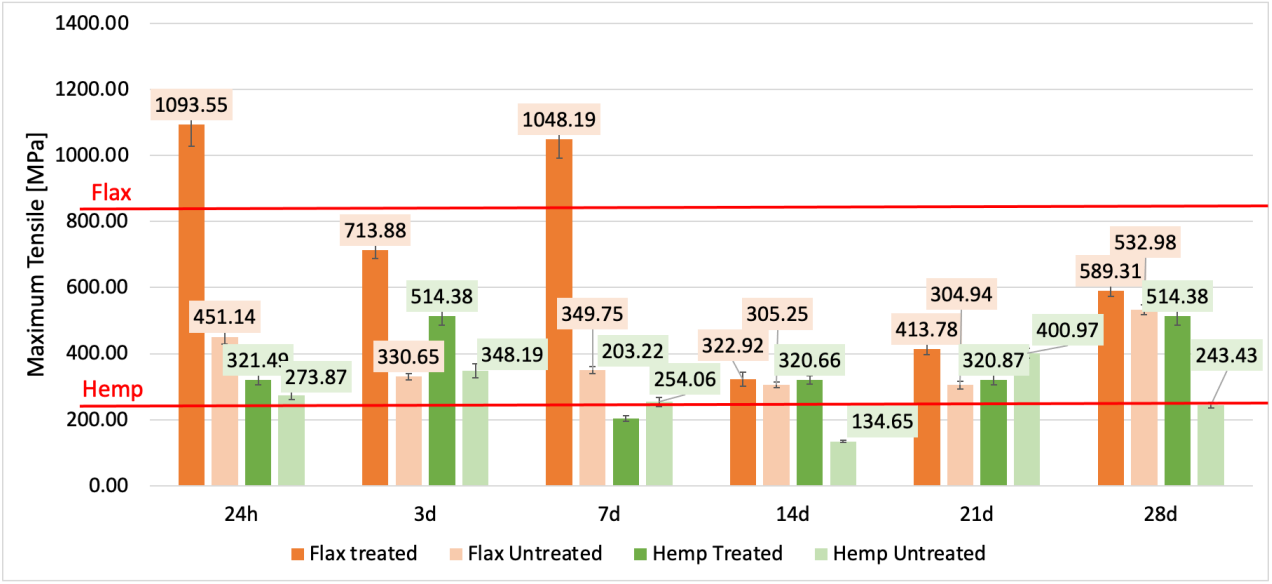


Figure 13. Effects of the degradation at an alkaline solution on the tensile strength.

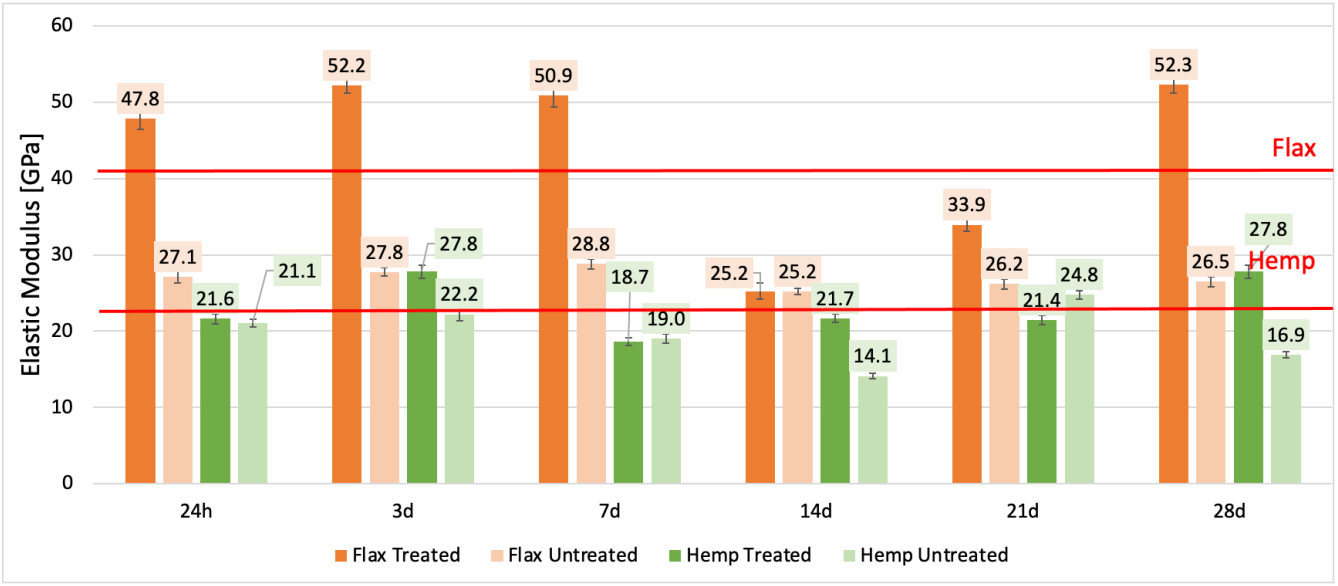


Figure 14. Effects of the degradation at an alkaline solution on the elastic modulus.

It was observed that treated flax fibres had their maximum tensile strength increased during the first 24 h, reduced after three days, and increased again after seven days. It dropped by 70% after 14 days and gradually increased again for the following two weeks. However, when compared to the untreated natural fibres from (indicated line parallel to the *x*-axis), it had a reduction of 32%. In comparison with the untreated fibres degraded, the treatment showed an enhancement in the tensile strength during the whole period considered.

For hemp fibres, no pattern could be found for enhancement of the tensile strength through time (non-linear behaviour), but compared to the strength of natural untreated hemp fibres, after 28 days of degradation, the treated fibres were 95% stronger than the untreated natural fibres from Table 3. Untreated fibres reduced their tensile strength by 8.4%.

In terms of the elastic modulus, untreated flax fibres did not change significantly during the experiment, with E reduced from 40.78 GPa to values from 25.2 to 28.8 GPa. However, treated and degraded flax fibres had their elastic modulus increased in the first 24 h, gradually decreased during the first two weeks, and higher again during the final 14 days of the experiment, which could indicate an increase in their stiffness through time.

For treated hemp fibres, the variability of the elastic modulus was smaller in comparison to flax. Results indicate that treated fibres presented increased elastic modulus after 3 and 28 days of the experiment, while degraded untreated fibres presented lower elastic modulus throughout the test except for the samples tested at 21 days. These results could represent that treatment using NaOH 10% for 24 h reduced the variability of the properties changes of fibres when placed into an alkaline environment. Meanwhile, the treatment with stearic acid could have coated the fibres, allowing chemical changes in an alkaline matrix.

3.1.3. SEM Images

Using scanning electron microscopy (SEM), it was possible to visualise the changes on the surface of the fibres in both natural and treated states before and after the degradation test.

As shown in Figure 15, both flax and hemp fibres treated presented better coating than untreated fibres. After the degradation test, the untreated fibres were damaged, and their internal layers were exposed, while the treated samples presented structures similar to the natural and untreated samples, indicating that the treatment could efficiently reduce the degradability of hemp or flax fibres in the alkaline pH of concrete.

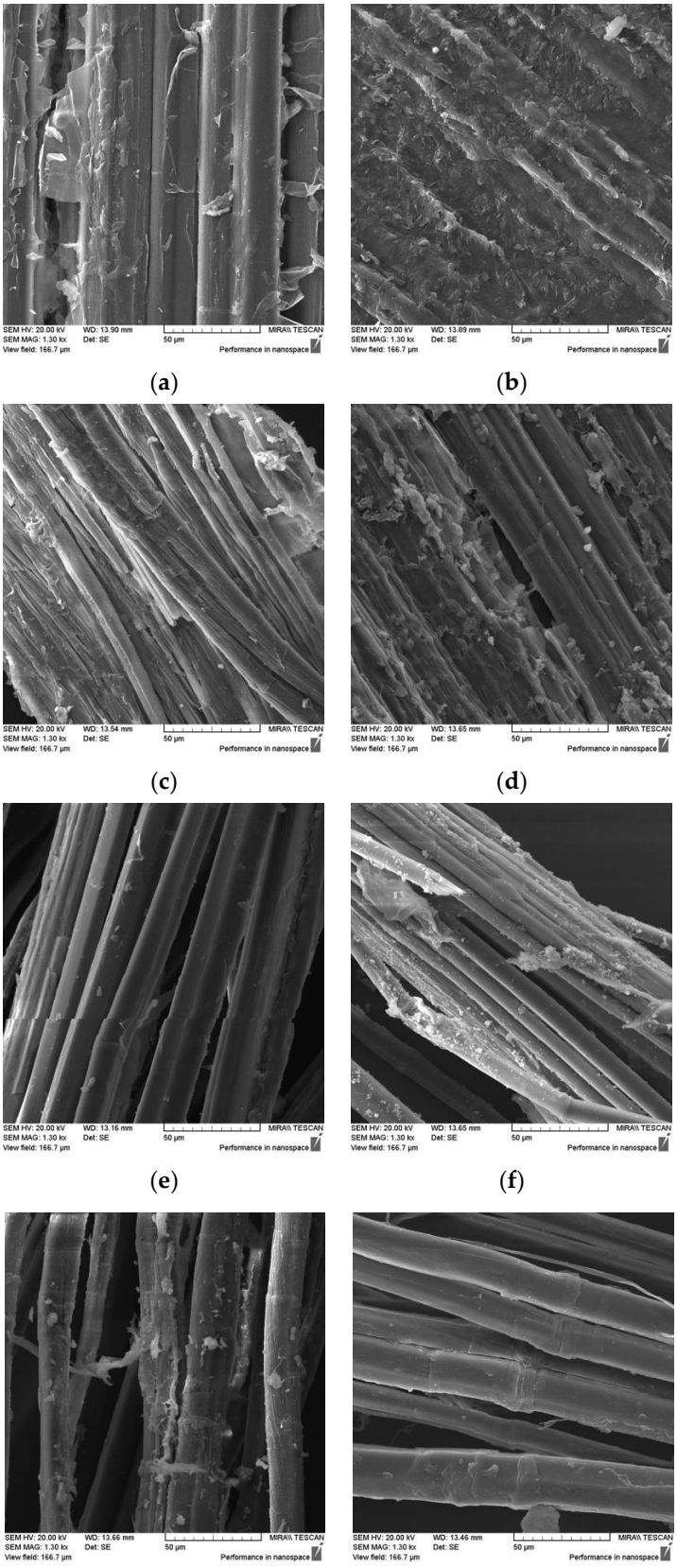


Figure 15. SEM images of untreated and treated flax and hemp fibres before and after the degradation test. (**a**) Natural and untreated flax fibres. (**b**) Flax-treated stearic acid 4 h. (**c**) untreated flax fibres degraded. (**d**) treated flax fibre degraded. (**e**) Natural and untreated hemp fibres. (**f**) Hemp fibres treated NaOH10% 24 h. (**g**) untreated hemp fibres degraded. (**h**) treated hemp fibre degraded.

3.2. Vegetable Fibre Reinforced Concrete

After evaluating the effects of surface treatment on hemp and flax fibres, concrete mixtures were made and tested to have their properties assessed. As the target of this study is to propose a satisfactory mixture of flax fibre-reinforced concrete, the concentration of these fibres was optimised according to the results obtained.

As mentioned, the control mixture was redesigned after the previous study [26], aiming for the reduction of thickness and higher workability to increase the possibilities of applications for the new mix. The results obtained are presented and discussed ahead in Section 3.

3.2.1. Compressive Strength (f_{cu}), Density and Slump

Firstly, two different samples of the control mix were made to verify the reproducibility of the values adopted in comparison with the obtained for the fibre-reinforced concrete mixes. Values for compressive strength, densities and slump obtained for each mix are presented in Table 6.

Table 6. Mechanical properties.

Mix	f_{cu} [MPa] 7 Days	f_{cu} [MPa] 28 Days	Density [g/cm^3] 28 Days	Slump [mm]
Control 1.1	37.4	49.7	2.411	35
Control 1.2	36.2	51.3	2.396	38
Polypropylene 0.5%	11.1	21.1	2.256	5
Polypropylene 0.25%	26.4	27.9	2.267	12
Hemp 1.1 0.5%	21.5	28.2	2.318	0
Hemp 1.2 0.5%	23.6	30.8	2.333	0
Flax 1 0.5%	19.6	29.2	2.297	0
Flax 2 0.35%	17.5	19.5	2.324	0
Flax 3.1 0.25%	24.8	32.4	2.348	20
Flax 3.2 0.25%	25.8	35.2	2.332	23
Basalt 1.1 0.5%	23.3	25.6	2.297	10
Basalt 1.2 0.5%	21.0	26.7	2.301	10

To visually interpret these values, Figure 16 graphically brings the results of compressive strength [MPa] obtained after seven and 28 days and their respective specific gravity [kg/m^3].

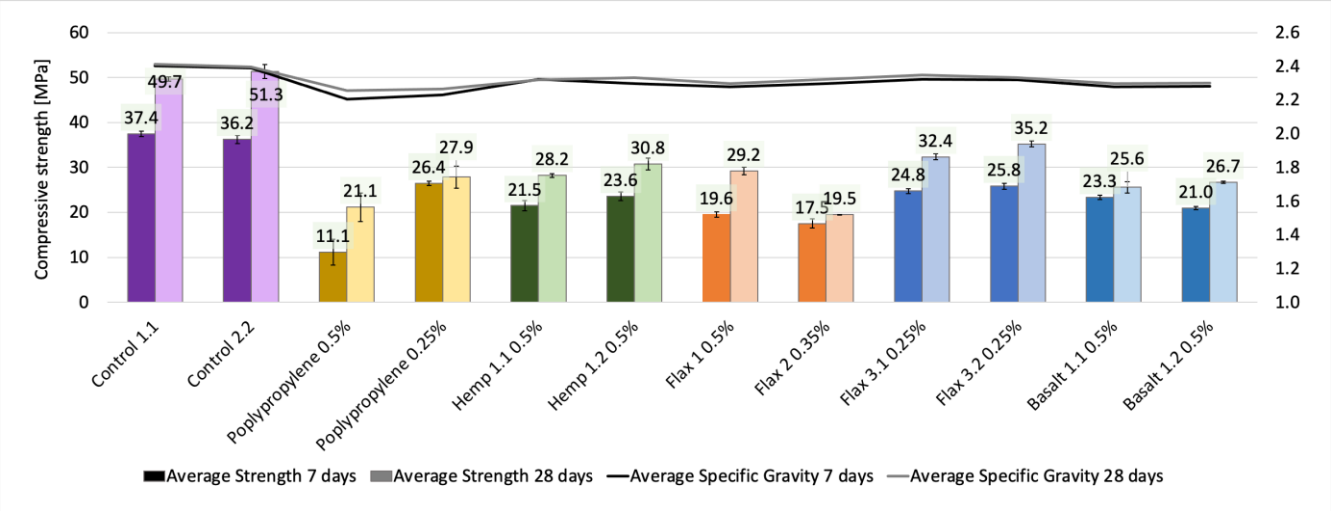


Figure 16. Compressive strength [MPa] and specific gravity [g/cm^3].

Similar to the results obtained in the preliminary stage of this study, the target f_{cu} on the mix design was 32 MPa. As the w/c ratio was adjusted from 0.82 to 0.5 as explained, there actual compressive strength obtained for the control mix after 28 days represented a C50, a high strength concrete. However, when the fibres were incorporated into all the mixtures, the compressive strength was reduced to less than 32 MPa. As the target of this study is to propose a satisfactory mixture of flax fibre-reinforced concrete in comparison to hemp, the concentration of flax fibres was optimised according to the results obtained.

By reducing the % by volume of treated flax from 0.5% to 0.25%, it was possible to achieve the compressive strength designed (C32) for the duplicate mixes produced, achieving higher compressive strength than the other FRC studied, representing over 70% of the f_{cu} obtained for the plain control concrete.

In terms of density, the incorporation of fibres reduced the density of all the mixes when compared to the control sample. The property was slightly reduced from samples tested at seven in comparison to those tested at 28 days.

3.2.2. Fracture Energy (Gf), Young's Modulus (E) and Flexural Tensile Strength (F)

Results presented in Table 7 indicate that the addition of fibres increased the fracture energy and reduced the elastic modulus of the mixtures in comparison to the control mix at seven days, representing a reduction of the concrete brittleness as suggested by Liu et al. (2019) [43]. Starting this analysis from the vegetable fibres, the mix containing flax 0.5% reached fracture energy 88% higher than the control mixture, a value greater than those obtained for mixes reinforced with basalt 0.5% and polypropylene 0.5% (177% and 171%). Hemp fibres presented the highest increase in fracture energy among the mixes reinforced with 0.5% of fibres, surpassing the mix reinforced with hemp at 1.0%.

Table 7. Fracture energy and elastic modulus from the three-point bending test.

Mixture	Gf [N/m]	Gf [% of Control]	E [GPa]	E [% of Control]
Control	180.31	100%	48.33	100%
Basalt 0.5%	319.86	177%	30.02	62%
Basalt 1.0%	489.42	271%	29.17	60%
Flax 0.5%	338.63	188%	23.65	49%
Hemp 0.5%	368.35	204%	38.62	80%
Hemp 1.0%	356.57	198%	17.34	36%
Polypropylene 0.5%	308.84	171%	20.76	43%
Polypropylene 1.0%	406.35	225%	37.54	78%
Steel 0.05%	193.87	108%	47.01	97%
Steel 0.1%	190.49	106%	44.94	93%
Steel 0.15%	415.56	230%	47.30	98%
Steel 0.2%	596.74	331%	40.61	84%

Mixes containing less than 0.15% of steel fibres did not have results significantly different from the control mixture, and for those containing 0.15% or 0.2%, results were considerably higher.

The addition of 0.5% of flax fibres reduced the elastic modulus by 49%, representing an increase in the concrete ductility and a reduction in the brittleness. This result was the third smallest obtained in the study, only higher than the mixes containing hemp by 1.0% and polypropylene by 0.5%. As expected, all fibre-reinforced mixes presented reduced elastic modulus. Reduction of the maximum aggregate size also provided a reduction in this property [44]. The reduction in this property can possibly be associated to the fibre-matrix adhesion, as the aggregate size also directly affects the Young's modulus.

The addition of basalt fibres by 0.5% and 1.0% obtained respectively 62% and 60% of the result found for the control mix. Mixes containing steel fibres only slightly reduced the elastic modulus. Results do not indicate a proportional correlation between the percentage of fibres and the elastic modulus.

Table 8 shows the average values obtained and calculated from the three-point bending test for fracture energy (Gf) and Young's modulus (E) for the optimised mixes of FRC. In comparison to the values obtained from the preliminary analysis presented in Table 7, the reduction of the maximum aggregate size increased the fracture energy of mixes fibre reinforced, except for the mix containing 0.35% of flax fibres tested at seven days. However, after 28 days, the same mixture presented an increase of 53% in comparison to the control mix.

Table 8. Fracture energy and elastic modulus of FRC.

Mixture	Gf [N/m]	Gf [% of Control]	E [GPa]	E [% of Control]
Control 7 d	198.77	100%	27.67	100%
Basalt 0.5% 7 d	1296.88	652%	11.14	40%
Flax 0.25% 7 d	129.80	65%	25.13	91%
Hemp 0.5% 7 d	285.61	144%	19.43	70%
Control 3 28 d	146.31	100%	45.30	100%
Basalt 0.5% 28 d	909.16	728%	36.95	82%
Flax 0.25% 28 d	191.58	153%	32.44	72%
Hemp 0.5% 28 d	316.74	254%	32.59	72%

The mixture containing hemp fibres presented, at 7 days, an increase in the Gf of 44% and after 28 days the percentage rose to 254% of the control.

In terms of elastic modulus, all the reinforced concrete mixes presented lower values. The modulus increased for both basalt and hemp fibres comparing the tests conducted at seven days to those conducted at 28 days, contrary to the mix containing flax fibres that at seven days presented an elastic modulus proportional to 91% of the control and at 28 days the value was reduced to 72%, equivalent to the mix reinforced with 0.5% of hemp fibres.

Flexural Tensile Strength (f_n) and Residual ($f_{R,j}$) [MPa]

Table 9 show the results obtained during the preliminary study.

Table 9. Flexural tensile strength (f_n) and ($f_{R,j}$) [MPa], j values from Table 5.

Mixture	f_n	$f_{R,1}$	$f_{R,2}$	$f_{R,3}$	$f_{R,4}$	$f_{R,5}$	$f_{R,6}$	$f_{R,7}$	$f_{R,8}$
Control	4.71	0.67	1.11	1.99	2.15	-	-	-	-
Basalt 0.5%	4.29	0.31	0.42	0.68	2.35	0.51	0.21	-	-
Basalt 1.0%	5.29	0.26	0.34	0.53	1.71	1.27	-	-	-
Flax 0.5%	3.21	0.43	0.63	1.00	2.58	0.65	0.35	-	-
Hemp 0.5%	4.95	0.40	0.69	1.23	3.81	0.90	-	-	-
Hemp 1.0%	3.60	0.30	0.41	0.62	1.50	1.94	1.19	-	-
Polypropylene 0.5%	3.81	0.45	0.68	1.08	2.44	1.25	-	-	-
Polypropylene 1.0%	4.51	0.50	0.75	1.26	3.88	1.80	-	-	-
Steel 0.05%	4.65	0.64	1.04	1.84	1.98	-	-	-	-
Steel 0.1%	4.62	0.53	0.84	1.59	2.93	-	-	-	-
Steel 0.15%	4.87	0.64	1.03	1.80	2.62	1.32	-	-	-
Steel 0.2%	4.14	0.44	0.74	1.43	3.33	1.28	1.16	-	-

Before comparing the results from Table 10 to the values obtained in the first mixture (Table 9), as advised by the standard EN14651, results were tested for possible instability and the mixture containing basalt fibres tested at 28 days presented $f_{R,4}$ (CMOD = 0.5) less than 30% of f_n ($CMOD_{FL}$). As the results presented a similar behaviour to the mix tested at 7 days, the values were still considered in this analysis.

Table 10. Flexural tensile strength (f_n) and ($f_{R,j}$) [MPa] obtained from the optimised mixes.

Mixture	f_n	$f_{R,1}$	$f_{R,2}$	$f_{R,3}$	$f_{R,4}$	$f_{R,5}$	$f_{R,6}$	$f_{R,7}$	$f_{R,8}$
Control 7 d	3.95	0.30	0.40	0.57	1.48	0.11	-	-	-
Basalt 0.5% 7 d	6.02	0.35	0.49	0.75	1.73	5.13	5.92	-	-
Flax 0.25% 7 d	3.07	0.38	0.55	0.90	2.30	0.97	0.44	0.21	0.15
Hemp 0.5% 7 d	3.66	0.41	0.63	1.04	2.68	3.06	1.95	1.39	1.54
Control 28 d	4.03	0.79	1.29	2.16	0.17	-	-	-	-
Basalt 28 d	9.15	0.60	0.97	1.67	4.34	7.17	3.07	-	-
Flax 0.25% 28 d	4.23	0.46	0.73	1.23	3.03	1.00	0.34	-	-
Hemp 0.5% 28 d	4.60	0.59	0.97	1.73	4.09	3.66	2.08	-	-

Comparing these results to the preliminary analysis, the second control mix achieved lower values of flexural tensile strength at peak load, indicating that by reducing the maximum aggregate size, this property is also slightly decreased, however, the residual tensile strength obtained for the mixes reinforced with flax and hemp fibres at seven days was increased, presenting residual strength for deflections j = 7 and j = 8. After 28 days, all the mixes presented higher values than those obtained at seven days in this part of the study, except for the mix reinforced with 0.25% of treated flax fibres that at 28 days presented a residual tensile strength equivalent to the obtained for the mix containing 0.5% of untreated fibres during the preliminary analysis.

3.2.3. Thermal Conductivity

To evaluate the insulation related to each mixture, their thermal conductivity was measured following the procedure previously detailed. As seen in the literature review, thermal conductivity is the inverse of thermal resistivity, where lower values represent enhanced thermal insulation [40].

Table 11 shows the values experimentally obtained. In a similar study, Bala and Gupta (2021) [40] studied this property on concrete reinforced with the waste of tile rubber and the values obtained for the vegetable fibres are close to their mixes using up to 40% of tile rubber as a sand replacement: 0.96 to 0.85 [W/mK].

Table 11. Thermal conductivity (λ).

Mix	λ [W/mK]	Mix	λ [W/mK]
Control	0.98	Hemp (0.5%)	0.89
Polypropylene (0.5%) FRC	0.84	Flax (0.5%)	0.80
Polypropylene (0.25%) FRC	0.87	Flax (0.35%)	0.83
Basalt (0.5%)	0.81	Flax (0.25%)	0.93

To compare the effects of the addition of fibres on the thermal conductivity of concrete, Figure 17 presents the percentage of control obtained from each sample tested.

Values of density from Table 6 were also included, allowing a comparison between the density and the thermal conductivity.

As expected, adding fibres reduced the thermal conductivity of all samples compared to the control mix. The two mixtures that presented better results were the one reinforced with basalt fibres, a material already used by the construction sector for insulation, and the mix containing 0.5% of flax fibres. Corroborating the literature review, the mixes containing a higher volume of fibres presented lower density, confirming that the samples presented a direct relation between density and thermal conductivity.

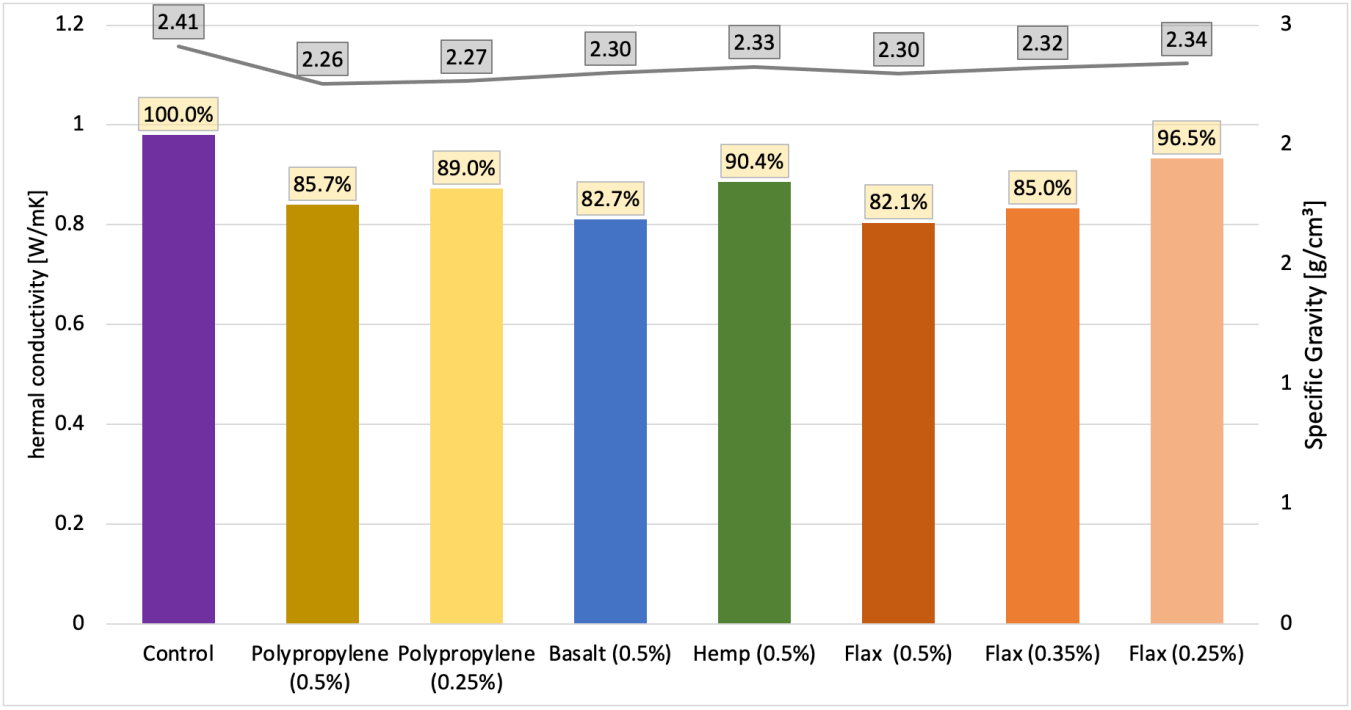


Figure 17. Thermal conductivity [W/mK] and Specific Gravity [g/cm^3].

3.2.4. Water Penetration

Figure 18 shows results obtained from the experiment led to measuring the water penetrability on the hardened concrete mixtures produced. In comparison to the control mix, the presence of fibres, in general, increased the water penetration. However, the addition of 0.25% of flax fibres presented values virtually similar to the control mix, indicating that, at this proportion, the cement paste would be able to coat the fibres, making it difficult for the water to penetrate the matrix.

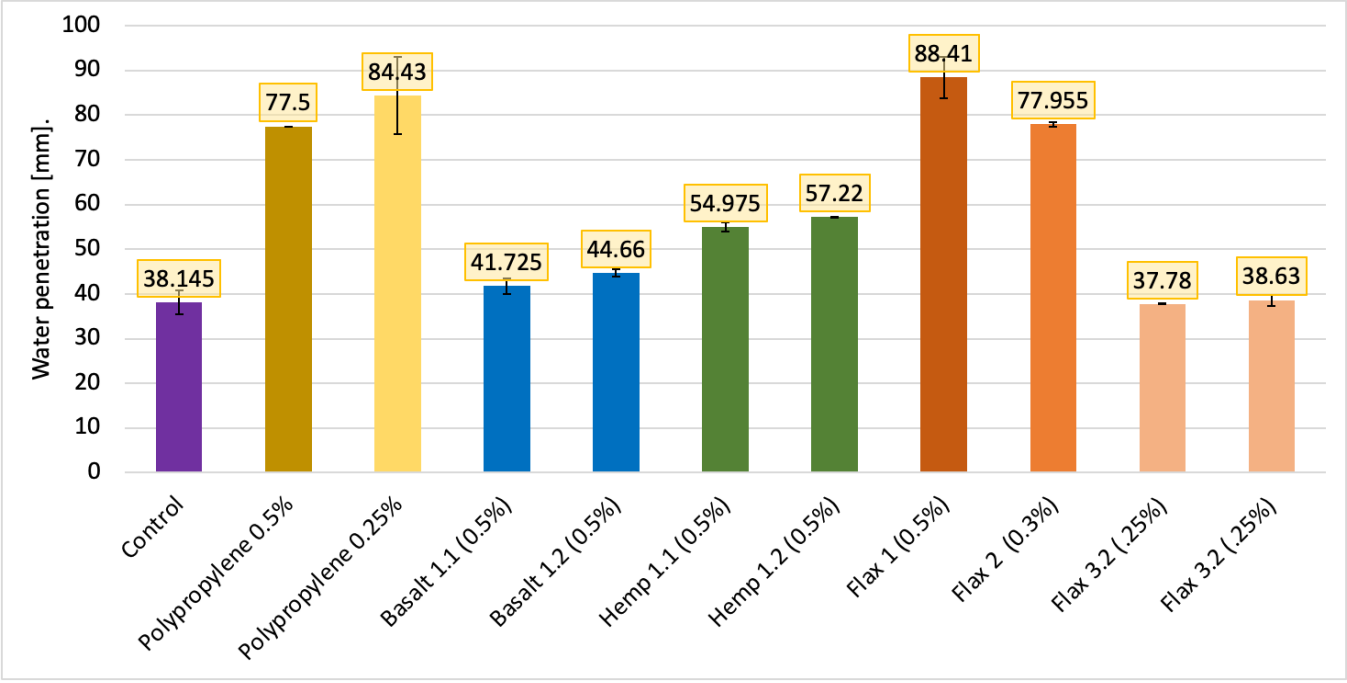


Figure 18. Water penetration [mm].

The mixes containing hemp fibres presented up to 50% higher penetration levels in comparison to control and flax 0.25%.

Surprisingly, the mixes containing polypropylene, a synthetic material, presented higher levels of water penetration, which could indicate lower fibre-matrix adhesion.

3.2.5. SEM Images

Comparing the SEM images from 30 days and the same mixture after 90 days (Figure 19), it was possible to observe the strong presence of fibres on both samples of HFRC and FFRC at 30 days with a fibrous aspect, and after 90 days the cementitious bond seems much closer and the presence of fibres is reduced, which could indicate degradation of the fibres associated with the concrete strengthening.

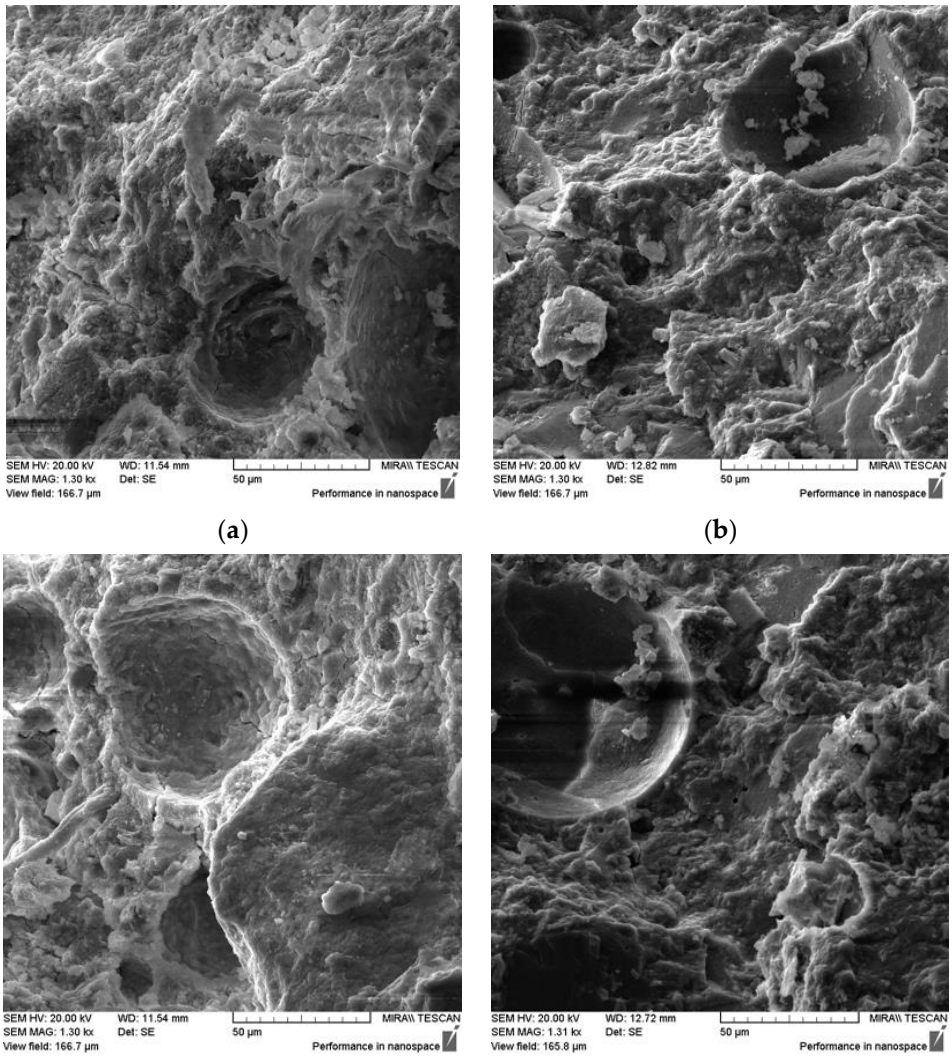


Figure 19. (**a**) treated hemp fibre-reinforced concrete at 30 days, (**b**) treated hemp fibre-reinforced concrete after >90 days, (**c**) treated flax fibre-reinforced concrete at 30 days, (**d**) treated flax reinforced concrete after >90 days.

It was not possible to evaluate the change in the percentage of voids caused by fibre degradation and concrete ageing in this study. However, as suggested by the literature, the removal of organic compounds, such as hemicellulose, lignin, and waxes, affects the structure of the fibres, allowing the cement paste from concrete to penetrate areas that

would be degraded with time, possibly turning into voids. This behaviour increases the adhesion between fibre and matrix, affecting the mechanical properties of concrete.

4. Limitations

The manual extraction and fibre chopping could not guarantee the exact length of the fibres.

Although the plain mixture was designed to be a C32, as the w/c ratio was adjusted from 0.8 to 0.5, the obtained compressive strength was proportional to a high-strength concrete C45/C50. Finally, the concrete specimens with SEM images evaluated were tested at 90 days. Formation of portlandite was not observed at this age.

5. Conclusions

From the study described in this research paper, it was possible to conclude that:

- Basalt fibres, as mineral fibres, presented superior results when compared to flax and hemp fibres as concrete reinforcement, consistent with what is already known by the research community.
- Most of the surface treatments studied increased both the tensile strength and elastic's modulus of hemp and flax fibres.
- Although the surface treatment using stearic acid for 4 h was selected for the flax fibres, treatment using EDTA for 4 h also presented interesting results. It might require an additional treatment step, according to Le Troedec et al. [45], and this can be suggested as a topic for future research work for both flax and hemp fibres.
- Corroborating results found by other authors, using NaOH to treat hemp fibres reduced their variability, including under an alkaline environment.
- All FRC mixes presented increased fracture energy and reduced elastic modulus, with the mixes containing 0.5% of treated hemp fibres and 0.5% treated flax fibres outstanding compared to the others.
- Adding treated flax fibres by 0.5% could reduce the thermal conductivity by 4% more when compared to the same mixture reinforced with polypropylene.
- While the water penetrability was inversely reduced by adding synthetic fibres into concrete, vegetable fibres presented a proportional increase with a more significant percentage of fibres. This was possibly due to the higher adhesion between the fibre and matrix caused by the high hygroscopicity of plant fibres, while polypropylene fibres would present less permeability.

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Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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