TECHNOLOGICAL UNIVERSITY OF THE SHANNON: MIDLANDS MIDWEST

# THE SUITABILITY OF VEGETABLE FIBRES FOR THE REINFORCEMENT OF CONCRETE

being a thesis submitted for the fulfilment of the requirements for the degree of

**DOCTOR OF PHILOSOPHY**

by

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Based on a research carried out under the supervision of

Dr Paul Archbold

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# Declaration

I certify that this PhD dissertation which I now submit for examination is entirely my own work and has not been taken from the work of others save and to the extent that such work has been cited and acknowledged within the test of my work. The dissertation was prepared according to the regulations for postgraduate study of the Technological University Of The Shannon: Midlands Midwest and has not been submitted in whole or part for an award in any other Institute or University.

The work reported on in this dissertation conforms to the principles and requirements of the Institute's guidelines for ethics in research.

Signed: ______________________________

Student No.: A00257970 Date: ______________________________

*“Seja qual for o seu problema*

*Fale com Deus, ele vai ajudar você.*

*Após a dor vem a alegria*

*Pois Deus é amor e não te deixará sofrer.*

*Deus te trouxe aqui*

*Para aliviar o seu sofrimento*

*É Ele o autor da Fé,*

*Do princípio ao fim,*

*Em todos os seus tormentos.*

*E ainda se vier noites traiçoeiras*

*Se a cruz pesada for, Cristo estará contigo*

*O mundo pode até fazer você chorar*

*Mas Deus te quer sorrindo.”*

*Simone Telésforo, Carlos Papae*

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# Abstract

The use of vegetable fibres as a sustainable alternative to non-natural fibres for reinforcing concrete was investigated in this research. For plant-based fibres, previous studies have identified issues such as the variability in properties and biodegradability in the alkaline pH of concrete.

The study was divided into four different sections along with the literature review. First, the properties of untreated flax and hemp fibres were assessed. Second, the fibres were subjected to surface treatments using NaOH (at concentrations of 5%, 15%, and 15%), KMnO4, stearic acid, and EDTA solutions for various durations (4, 6, 10, 15, and 24 hours) to minimize variability and degradation caused by the alkaline pH of concrete. The effects of each treatment were evaluated, and the treatments that yielded the most satisfactory results were selected. For flax fibres, the 4-hour stearic acid treatment, which was a novel treatment in this study, was chosen. For hemp fibres, the 24-hour NaOH 10% treatment was selected, which provided original results for the reinforcement of concrete. Thirdly, the effects of an alkaline environment on the tensile strength and elastic modulus of treated and untreated fibres were assessed.

Lastly, the effect of long fibres (40 ± 5mm) on certain properties of concrete was investigated. The addition of treated fibres increased the peak flexural tensile strength and reduced the elastic modulus of the concrete by at least 50% after 28 days. The mixes containing treated fibres exhibited significantly lower fracture energy values than the basalt fibre reinforced blend. Both types of fibre reinforced concretes using surface-treated fibres showed higher values for residual tensile strength. Additionally, the addition of fibres improved the thermal conductivity of the concrete, and a reduction in density was observed based on the volume of added flax fibres.

However, the addition of fibres increased the levels of water penetration, with the exception of the mix containing 0.25% flax fibres, which exhibited similar results to the control blend and reduced water penetration compared to the other FRCs tested. Overall, the results suggest that, although additional research is still required, the surface treatment of vegetable fibres can enhance the properties of FRC and contribute to their use as potential and sustainable alternative to non-natural fibres for concrete reinforcement.

**Keywords:** Natural fibre reinforced concrete, flax fibres, hemp fibres, fracture energy, mechanical properties, surface treatment.

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# LIST OF ABBREVIATIONS

| *Initials* | *Meaning* |
|---|---|
| CMOD | Crack Mouth Opening Displacement |
| EC | Carbon Emission |
| EE | Embodied Energy |
| FRC | Fibre Reinforced Concrete |
| FRP | Fibre Reinforced Polymer |
| FTIR | Fourier-transform infrared spectroscopy |
| GBTs | Green Building Technologies |
| GFRC | Glass Fibre-Reinforced Concrete |
| GHS | Globally Harmonized System of Classification and Labelling of Chemicals |
| JFRC | Jute fibre reinforced concrete |
| LCA | Life-Cycle Assessment |
| LOP | Limit of Proportionality |
| NFRC | Natural Fibre Reinforced Concrete |

| | |
|---|---|
| NFRP | Natural Fibre Reinforced Polymer |
| PC | Plain Concrete |
| PPFRC | Polypropylene fibre reinforced concrete |
| SEM | Scanning Electron Microscope |
| SFRC | Steel Fibre Reinforced Concrete |
| SNFRC | Synthetic Fibre-Reinforced Concrete |
| VFRC | Vegetable Fibres Reinforced Concrete |

# Chapter One

# Introduction

# Chapter 1: Introduction

Fibre reinforced concrete is a cementitious composite reinforced with fibres in random orientation. The term composite was defined by R.P.L. Nijssen (2015) as *"a material structure that consists of at least two macroscopically identifiable materials that work together to achieve a better result"*. The author explains that combining materials in a composite form is relevant to obtaining a new product with enhanced properties to satisfy the requirements needed for a defined application that isolated, none of the materials would behave so well. From that concept, it is possible to understand that cementitious composites are produced from the combination of one or more materials into a cementitious matrix, and that concrete is also a cementitious composite.

In the construction industry, concrete is defined by the English Oxford Living Dictionary Online (2019) as *"a building material made from a mixture of broken stone or gravel, sand, cement, and water, which can be spread or poured into moulds and forms a mass resembling stone on hardening"*. Figure 1.1 shows the main ingredients of a general concrete mix according to their approximate proportions designed to resist compressive stresses with decades or centuries of durability. (Tariq et al. 2021)

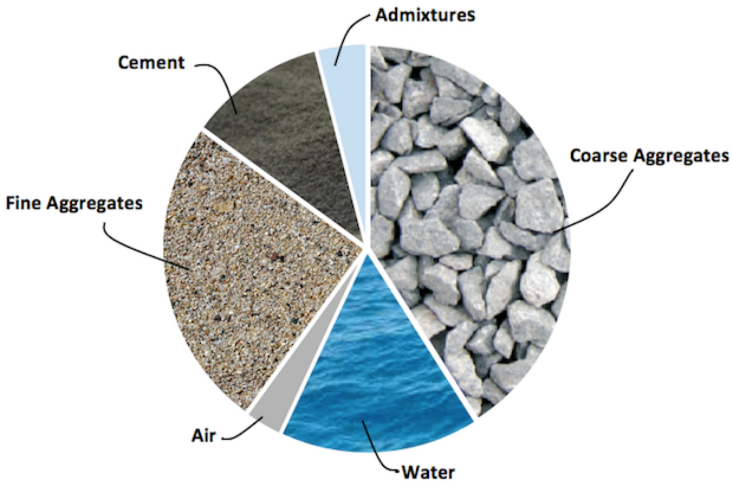



*Figure 1.1– Composition of concrete(*Concrete & Its Ingredients (%) – Civil-Ideas *[no date]).*

Plain concrete mixes are widely known for their excellent behaviour under compressive stresses. Their applications range from in-loco moulded concrete structures to precast and modular pieces. Fragile at an early age and when submitted to high impacts or tensile stress, still after hardening, concrete is a brittle material with no post-cracking residual tensile strength due to its ductility. (Rahimi et al. 2022a)

Depending on the application, reinforcement is commonly required. Reinforcing bars are added to give the concrete the capacity to resist bending moments and shear forces. (Drougkas et al. 2022)

For beams and columns to resist a bending moment, steel bars are the most common option, but, for structures subjected to abrasion and superficial impact, another solution is required: the addition of fibres into the concrete mixture. Fibres increase uniformly the residual tensile strength of concrete, adding some elasticity to its fracture, and reducing its brittleness.  The improvement of these properties can also be interesting to minimize early-age cracking since they can occur due to various events that may cause changes in the concrete section exceeding its tensile strain capacity. (CIRIA 766 2018; Rahimi, Hisseine, et al. 2022).

Fibre reinforcement is used for different applications such as reducing the concrete brittleness and weight, improving its thermo-acoustic properties, increasing its elastic modulus, or also adding some post-crack residual tensile strength to the structure. (Abbass et al. 2018; Castoldi et al. 2019; Eidan et al. 2019)

There are numerous types of fibres currently available to be used on concrete reinforcement, and Figure 1.2 shows four main categories according to Zollo (1997):

a) Steel fibres

b) Synthetic fibre, including carbon and polymers fibres.

c) Glass fibres (a particular category of synthetic fibres)

d) Natural fibres

*(a) Steel fibres* (Abu-Bakr et al. 2022)

*(b) Synthetic fibre* (Rivera et al. 2021)

*(c) Glass fibres*(Tibebu et al. 2022)

*(d) Hemp fibre (*Hemp Fibers Make Concrete Stronger| Concrete Construction Magazine *[no date])*

*Figure 1.2 – Four main categories of fibres.* (R.F. Zollo 1997; Rivera et al. 2021; Abu-Bakr et al. 2022; Tibebu et al. 2022)

The criteria to be considered include aspects required by the new structure to be built, the level of moisture exposure, impact absorption, abrasion level etc. The durability of steel is also a concern related to its use, as corrosion reaction occurs when in contact with the air and humidity, and the steel not only loses its mechanical properties but also modifies its cross-section measurements bringing internal stress to the concrete leading it to a structural failure.

Each category of FRC can be divided into different groups depending on size, material, composition, diameter, and other characteristics that impact their properties. Steel fibre reinforced concrete (SFRC) and synthetic fibre reinforced concrete (SFRC), including glass fibre (GFRC), are currently commercialised and successfully accepted by the construction industry. However, concerns about all the unrenewable sources of materials being used and the environmental impact caused by the sector arise. (Alsaif et al. 2019; Liu et al. 2019)

One of the current ways to measure the environmental impact of the materials adopted in construction is through a life-cycle assessment (LCA), it evaluates the impact of a certain product or material on the environment, also quantifying the expected amount of energy consumption and $CO_2$ emission during the entire lifespan of a material. There is currently a database called "Inventory of Carbon & Energy" (ICE) containing the values for most of the building materials available in the market (Zah et al. 2007; Hammond and Jones 2008; Hammond and Jones 2011; Todkar and Patil 2019).

As discussed in the following chapters, the most common types of fibres used for concrete reinforcement require a high amount of energy and non-renewable sources of raw material to be produced. Natural fibres have been studied over the four decades for cementitious composites reinforcement and in the past two decades, they have gained pace, due to their low embodied energy and carbon emission. (Arrigoni et al. 2017; Barth and Carus 2019; Kumar et al. 2020; Shang and Tariku 2021)

In this work, a quantitative assessment of the suitability of vegetable fibres as a potential alternative to other fibres currently for concrete reinforcement was performed. A comparative study was led aiming the reduction of the environmental footprint when compared to polymer and steel fibres reinforced concrete.

Swamy (1990) appointed different inherent weaknesses related to the use of vegetable fibres for these applications. Firstly, the author mentions the low elastic modulus, which means that the material can be stretched before it breaks. However, this property can be particularly interesting to reduce the high elastic modulus of concrete if the structure built is expected to resist to impact, vibration or other conditions that could lead to a fragile rupture. (Cai et al. 2022; Wang et al. 2022)

Secondly, it is pointed out that vegetable fibres are susceptible to suffering attacks from fungus and insects and the reduction caused on their durability by the alkaline pH of concrete. In most current studies, these two features are addressed as the degradability of the fibres. Considering the long-term impact, this issue needs to be considered as it would directly affect the lifespan of the concrete structure.

Finally, the author mentioned that the same type of fibres will present significant variability in their properties. As they are natural, the composition of their structure can be affected by the weather conditions, quality of soil, position on the plant, etc. Different surface treatments have been considered as a measure to reduce the irregularity of their properties This technique is also known as "degumming", used to remove lignin and gummy compounds from the fibres resulting in a cellulose structure (Figure 1.3). (Feyisetan Adekunle 2015; Kumar Sinha et al. 2017; Ahmed et al. 2022; Rahimi et al. 2022b).

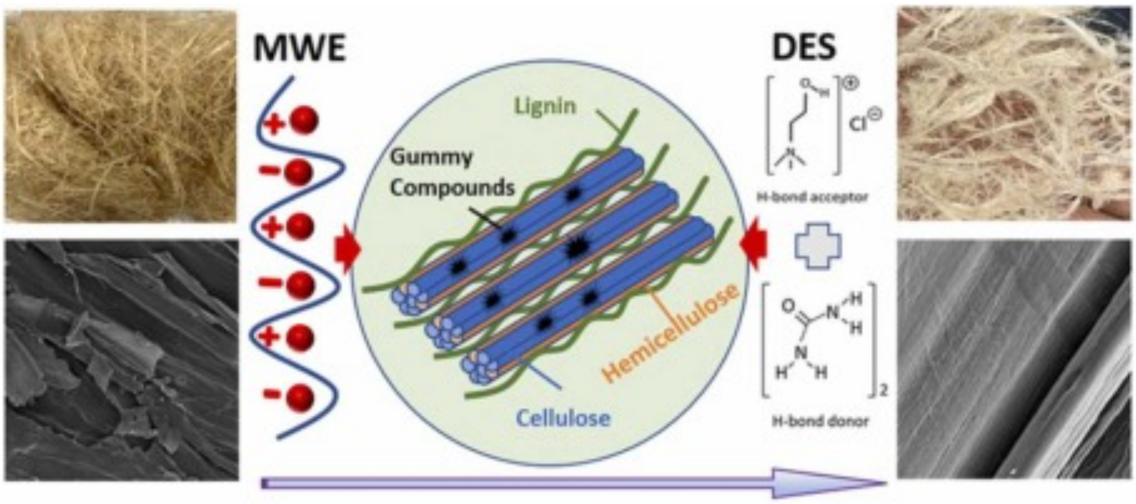



*Figure 1.3 – Chemical compounds of vegetable fibres* (Ahmed et al. 2022).

In response to the above issues, this research aims to evaluate the suitability of vegetable fibres for reinforcement of concrete, considering techniques being developed in the past years. As there is a vast range of vegetable fibres, an analysis was first conducted according to properties evaluated by authors in the literature.

For this research, the vegetable fibres selected were flax and hemp and compared to other non-natural fibres.

Having characterised the fibres selected and confirming results obtained by other authors, flax fibres presented significantly higher tensile strength when compared to hemp fibres. Focusing on the potential of flax fibres and the lack of research about their use for concrete reinforcement, the project proposal and research questions were finally defined:

## i. Project proposal and research aims

The research project aims to investigate the feasibility of using vegetable fibres as reinforcement in concrete. The primary objective is to develop a novel and sustainable type of fibre reinforced concrete (FRC) that can offer comparable mechanical and physical properties compared to traditionally commercialised FRCs. After reviewing the literature, it was found that flax fibres have been used as reinforcement in composites by different industry sectors; however, there is a gap in their application for reinforcement of concrete. Along with flax fibres, hemp fibres were also chosen due to their potentially superior tensile strength and their popularity among researchers, which offers an experimental benchmark for the results obtained for flax fibres.

The study is unique in its approach, as it compares vegetable, mineral, and non-natural fibres, and seeks a suitable surface treatment for plant-based fibres that enhances their behaviour in an alkaline matrix. The specific research aims are to:

- Identify and compare promising types of different vegetable fibres according to past research by evaluating their physical properties and tensile strength, as well as comparing them with industrially produced fibres.

- Investigate the effects on the properties of flax and hemp fibres of different surface treatments using chemical solutions to determine the most effective treatment among options suggested in the literature.

- Evaluate the degradability of treated and untreated flax and hemp fibres in an alkaline environment and compare their behaviour with hemp fibres following relevant standard.

- Design a concrete mixture and evaluate the effects of adding flax and hemp fibres into it in terms of mechanical and physical properties, compared to industrialised fibres.

## ii. Proposed solution/thesis outline

To achieve the research aims outlined earlier and considering the aforementioned parameters, this study was divided into three main sections. Firstly, a general analysis was conducted to characterize the properties of different natural fibres. Based on this analysis, untreated flax fibres were found to be superior to other fibres. A control concrete mix was prepared, and different fibre-reinforced concrete mixtures were produced by adding the studied fibres.

To improve the results obtained for flax fibres, various chemical surface treatments were conducted using solutions of Sodium Hydroxide (NaOH), Potassium

Permanganate ($KMnO_4$), EDTA ($C_{10}H_{16}N_2O_8$), and Stearic Acid ($C_{18}H_{36}O_2$) for different durations ranging from 4 to 24 hours. The fibres were subsequently neutralized, dried, and their properties re-evaluated.

While more detailed findings will be presented in the results and discussion section, it is important to note that the Stearic Acid treatment stood out as the best option for enhancing properties with minimum environmental impact among the options studied. As a result, it was selected as the preferred treatment for flax fibres.

As hemp fibres have already been extensively researched for this application, they were also subjected to the same treatments to enable a comparative analysis. Treatments using NaOH for hemp fibres are commonly reported in the literature and tin this study, the treatment using NaOH in a novel combination of time and concentration was selected for hemp fibres, confirming the efficacy of this solution outlined by researchers in the field. (Le Troedec et al. 2008; Diquélou et al. 2016; Poletanovic et al. 2021; Shang and Tariku 2021)

Next, the degradability of the treated and untreated fibres was assessed by using an adapted version of the methodology described in the "Standard Test Method for Stability of Cellulose Fibers in Alkaline Environments". (ASTM International 2019)

Lastly, the treated fibres were incorporated into new concrete mixtures, and their properties were evaluated to enable a comparison with basalt, hemp, and non-reinforced concrete.

In summary, the study evaluated the suitability of selected vegetable fibres for reinforcing concrete and the main findings are:

- Flax and hemp fibres were selected and subjected to different surface treatments to improve their properties.

- The surface treatment using stearic acid for 4 hours showed to fit better the requirements adopted for flax fibres, while the treatment using NaOH 10% for 24 hours was selected for hemp fibres.

- A mixture reinforced with 0.25% of treated flax was able to achieve the designed compressive strength values, surpassing the mix reinforced with 0.5% of treated hemp fibres.

- Although the variability on the properties of the fibres was still observed after treatment, when added into concrete results were consistent among samples.

- Both flax and hemp fibre-reinforced concrete mixes, at 28 days, presented the lowest stiffness among the mixtures studied compared to basalt FRC and the addition of fibres in the mixes enhanced the thermal conductivity, and it was observed that the concrete density was reduced according to the volume of flax fibres added.

- The mixes with 0.25% of flax fibres presented a similar level of water penetration to the control mix, indicating that at this proportion, the cement paste would be able to coat the fibres, reducing the water infiltration.

## iii. Significance

The high impact of the construction sector on the environment is being seriously addressed by governments all over the world. The topic is being studied already for decades by researchers and has now been strongly incentivised to be applied in the building industry.

In this research, an experimental study was conducted assessing the suitability of the use of vegetable fibres for reinforcement of concrete as an alternative to the currently non-natural fibres commercialized. The selected fibres for this study were flax, a fibre used in the automotive industry but with a gap in the literature on their behaviour for concrete reinforcement and hemp fibres, commonly studied by other researchers, used as a comparative parameter.

As well known by the research community, vegetable fibres present high variability and degradability, to minimize these effects, surface treatments are suggested by different authors. In this study, the effects of surface treatments using NaOH in different concentrations, $KMnO_4$, stearic acid and EDTA were evaluated for flax and hemp fibres at 4, 6, 10, 15 and 24 hours. This evaluation also addresses a gap in the literature review of surface treatments for flax fibres, in comparison with hemp fibres.

From this study, it was possible to select the treatment that increased the tensile strength of the fibres while reducing the stiffness, properties sought for the enhancement of concrete. Results achieved promising results that could lead to a safe replacement of polypropylene fibres by flax treated with stearic acid for 4h, contributing to the reduction of the negative impact of the construction industry on the environment.

## iv. List of publications

1. da Costa Santos, A.C.; Archbold, P., "Suitability of Surface-Treated Flax and Hemp Fibers for Concrete Reinforcement". *Fibers*, 2022, 10, 101. Doi: https://doi.org/10.3390/fib1011010.

2. da Costa Santos, A.C.; Archbold, P., "Experimental Investigation on the Fracture Energy and Mechanical Behaviour of Hemp and Flax Fibre FRC Compared to Polypropylene FRC," Construction Technologies and Architecture, vol. 1, pp. 1–5, 2022, doi: 10.4028/www.scientific.net/CTA.1.326.

3. da Costa Santos, A.C.; Archbold, P., "Mechanical Properties and Fracture Energy of Concrete Beams Reinforced with Basalt Fibres," Construction Technologies and Architecture, 2022, doi: 10.4028/www.scientific.net/CTA.1.316.

4. da Costa Santos, A.C.; Archbold, P., "Characterisation of Natural fibres for Composite applications," *Academic Journal of Civil Engineering*, no. 3rd International Conference on Bio-Based Building Materials, 2019, doi: 10.26168/icbbm2019.3.

# Chapter Two

# Literature Review

# Chapter 2: Literature Review

## 2.1 Concrete

Hardened concrete in its plain form is a composite that can resist compressive stress caused by loading, according to its design capacity. However, structures made of concrete, such as beams and slabs, may also experience flexural tensile stress, requiring additional reinforcement. To resist tensile and flexural tensile structures are designed to receive reinforcement, there are three main different types of reinforcements, selected according to the application. Steel reinforcement bars are the most common option for concrete pieces that will be subject to structural tensile stresses. (Alberti et al. 2018; Han et al. 2019; Drougkas et al. 2022)

Another form of strengthening concrete for structural applications could be through lateral confinement. Layers of textiles made of fibre reinforced polymer (FRP) are used, usually in hardened structures, to enhance their mechanical capacities, such as axial strain and compressive strength. Steel, glass, carbon and polypropylene fibres are commonly used for direct strengthening using FRP. (Fallah Pour et al. 2019; Isleem et al. 2020; Han et al. 2021)

However, reinforcement bars or FRP would not be satisfactory for concrete applications that require the structure to present enhanced deformation and energy absorption capabilities to minimize crack propagation at various stages, or better thermos-acoustic properties such as Industrial floorings and tunnels. For these, fibre reinforcement is recommended. (Abbass et al. 2018; Barth and Carus 2019)

## 2.2 Fibre-reinforced concrete (FRC)

Fibre-reinforced concrete (FRC) is a concrete composite that has fibres added to the fresh mixture (Figure 2.1a) and been studied for many decades. Once hardened the fibres are uniformly distributed in random orientation, becoming homogeneous in the concrete (Figure 2.1b).

With random orientation and distribution, their addition can reduce the concrete brittleness and the risk of fragile rupture of structures, as well as influence its strength, offering some post-crack tensile strength (Zia and Ali 2017; Castoldi et al. 2019; Eidan et al. 2019; Santoni et al. 2019; Tibebu et al. 2022).

*(a)*

*(b)*

*Figure 2.1 – (a) Fresh and (b) hardened mixture of hemp fibre reinforced concrete*

The reinforcement of concrete using fibres can also be employed for controlling crack-opening. Upon the incorporation of fibres into the mixture, there is a decrease in the modulus of elasticity of concrete, which permits the structure to elongate more before failure. Conversely, plain concrete exhibits no residual tensile strength once it has failed. The addition of fibres can offer a degree of tensile strength to the structure after cracking, thereby lowering the probability of structural failure. (Abbass et al. 2018; Alberti et al. 2018; Islam and Ahmed 2018; Castoldi et al. 2019)

Non-natural fibre-reinforced concrete mixes are commercially available for various applications, such as industrial flooring and tunnels, while natural fibres are typically considered more suitable for non-structural uses. However, both types of fibres can have positive and negative effects on concrete, as previously discussed in this chapter. Additionally, they can impact a range of composite properties, including compressive strength, density, heat and sound insulation, and water absorption, in addition to the tensile strength and Young's modulus of the material. (Piasta and Zarzycki 2017; Zia and Ali 2017; Degrave-Lemeurs et al. 2018; Sanjay et al. 2018; Barth and Carus 2019; Santoni et al. 2019; Tibebu et al. 2022)

While the need to minimise the environmental impact of construction has led to an increase in research on the subject in recent decades, further investigation is needed to establish a sustainable and dependable method for designing fibre-reinforced concrete (FRC) structures. (Kaur and Talwar 2017)

## 2.3 Current non-natural fibres used for concrete reinforcement

### *2.3.1 Steel fibres*

Steel fibres are commonly used for reinforcement, producing a composite called steel fibre reinforced concrete (SFRC). Their addition improves the concrete brittleness and increases its energy absorption capacity. Study using different lengths of steel fibres in the same mixture and compared them with a single length found that using only one size of fibres in a mix would present better results than combining them. (Okeh et al. 2019)

Although the authors do not discuss reason for that, reinforcing concrete with fibres of one length could be considered better than using fibres of various lengths due to the improved mechanical properties that result from the homogeneity of the composite. When using fibres of uniform length, they would distribute themselves uniformly throughout the concrete matrix, leading to enhanced tensile and flexural strengths. In contrast, the use of fibres of various lengths could lead to a more random distribution, potentially causing clustering and bridging, resulting in weakened mechanical properties.

A different study investigated the effects of varying fibre lengths in SFRC with different proportions and water/cement ratios of concrete. Steel has a known density that ranges from 7.75 to 8.05g/cm3. The results of this study indicate that the addition of steel fibres enhanced all the evaluated mechanical properties. When fibres with lengths of 40mm were added, the tensile, and flexural strengths increased proportionally to the percentage of fibres added, increasing also the compressive strength, but not proportionally. (Abbass et al. 2018)

Durability is another important factor to consider when using steel fibres in concrete. Since concrete is often exposed to water and oxygen, it creates an environment that is conducive to corrosion. Figure 2.2 illustrates the surface of a SFRC with fibres that suffered corrosion. The negative impact of this, is that the cross-section area of the steel increases due to corrosion, generating internal stress on the concrete walls, leading to cracks and ultimately structural failure. (Hwang et al. 2015)

*Figure 2.2 – SFRC with fibres corroded.*

### *2.3.2 Synthetic fibres (including glass fibres)*

A variety of synthetic fibres, including polypropylene (as shown in Figure 2.3), glass, carbon, PVC, and polyacrylonitrile, are presently available for use in concrete reinforcement (SNFRC).

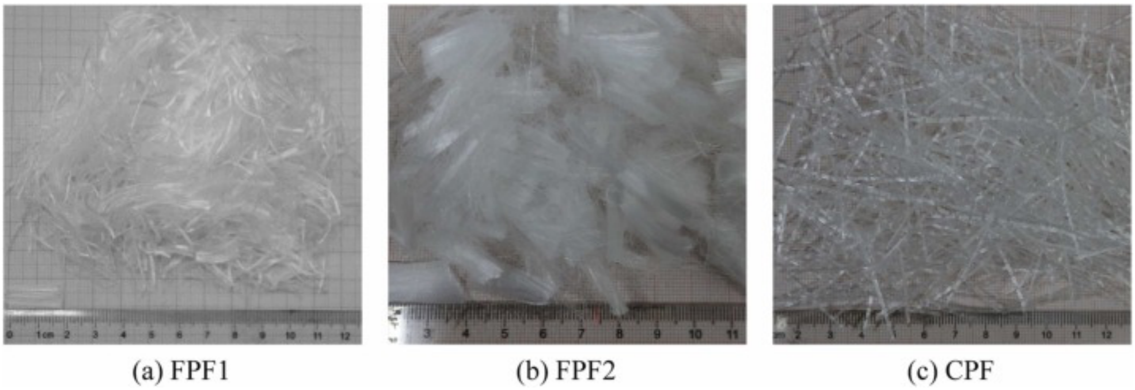



*Figure 2.3 – Types of polypropylene fibres* (Liang et al. 2022)

These fibres are produced entirely through industrial processes and necessitate high temperatures and non-renewable raw materials. Consequently, their embodied energy and carbon emissions are also high, with numerical values from the Inventory of Carbon & Energy being presented in Table 2.1. (Hammond and Jones 2008)

*Table 2.1 – Embodied Energy (EE) and embodied carbon (EC)*

| *Materials* | *EE - MJ/kg* | *EC - $kgCO_2/kg$* | *EC - $kgCO_2e/kg$* |
|---|---|---|---|
| Cement (General) | 4.5 | 0.73 | 0.74 |
| Concrete - 32/40 MPa | 0.88 | 0.123 | 0.132 |
| Concrete - 40/50 MPa | 1.00 | 0.141 | 0.151 |
| Steel - Virgin | 35.40 | 2.71 | 2.89 |
| Steel - Recycled | 9.40 | 0.44 | 0.47 |
| Polypropylene | 95.40 | 4.98 | - |
| Fibreglass (Glass wool) | 28.00 | 1.54 | - |
| Nylon (Polyamide Polymer) | 120.50 | 5.47 | 9.14 |
| Rockwool | 16.80 | 1.05 | 1.12 |
| Flax with a polyester binder (Insulation) | 39.50 | 1.70 | - |

The inventory provides two different values for embodied carbon, the first one ($kgCO_2/kg$) corresponds exclusively to the carbon dioxide ($CO_2$) emissions, and the second, ($kgCO_2/kg$) includes other emissions, such as methane and perfluorochemicals.

In 2008, vegetable fibres were not included in the inventory as a building material, except for the flax-containing polyester binder used on housing insulation. As it is possible to see, concrete by itself does not have high embodied energy or carbon emission during its lifetime.

One of the reasons can be the fact that, apart from cement, concrete is made of natural aggregates and water, thus the reinforcement material is what increases its environmental impact.

From the materials listed, recycled steel would be the option with a smaller green footprint and the figures get almost four times bigger for virgin steel. Glass fibres would present similar values and synthetic fibres would require up to 120 times more energy during their lifetime in comparison with concrete.

Incorporating synthetic fibres into concrete has been shown to enhance its durability, permeability, frost resistance, acid and alkali resistance, and reduce its overall density. Among the various synthetic fibres available, polypropylene fibres are the most widely used option, with a general density of 0.946g/cm$^3$. They effectively mitigate concrete cracking resulting from factors such as settlement or plastic shrinkage during initial stages. (Chen et al. 2018; Bolooki Poorsaheli et al. 2019)

A study examining the effects of incorporating three distinct types of synthetic fibres into a concrete mixture discovered that the addition of synthetic fibres in at least 0.33% of the volume improved the flexural strength of the structures and changed the failure mode from ductile to plastic. The fibres used were modified olefin, virgin polyolefin/polypropylene, and virgin copolymer polyolefin. The study did not report significant effects on compressive strength. Water absorption of the SNFRC mixes also increased from 0.66% to 0.99%, according to the authors. (Bolooki Poorsaheli et al. 2019)

Beyond the environmental impact and high cost, there is also a concern regarding the low interfacial shear stress between synthetic fibres and the cement matrix. Researchers have evaluated this property and concluded that the non-porous surface of synthetic fibres requires surface treatment with nano-silica to increase adhesion to concrete. The authors also emphasize the importance of considering the viscous behaviour of synthetic materials when using SNFRC. (di Maida et al. 2015; Sorzia et al. 2019)

From a sustainable perspective, the option with a smaller impact would be natural fibres potentially being an efficient alternative to the fibres currently employed in FRC production.

## 2.4 Natural fibres as potential alternative

Natural fibres are usually classified as vegetable, mineral, and animal (Figure 2.4). While the animal-based fibres comprise specific proteins such as wool, silk and hair, the mineral fibres can be produced from asbestos, wollastonite, basalt and palygorskite. Vegetable fibres can be extracted from different parts of plants since their seeds, classified  as primary or secondary, according to their application where primary plants are those cultivated for fibre extraction, while secondary plants produce fibres as a by-product. (Faruk et al. 2012; Onuaguluchi and Banthia 2016; Sanjay et al. 2018)

As for any other material, different properties can be evaluated to characterize natural fibres, giving parameters to understand and estimate their behaviour when submitted to different conditions and stresses.

A brief introduction regarding the categories just mentioned is included ahead in this section. It is worth mentioning again that animal fibres were not included in this study and although mineral fibres were tested for comparative purposes, the suitability of vegetable fibres for the reinforcement of concrete is the main target of the study.

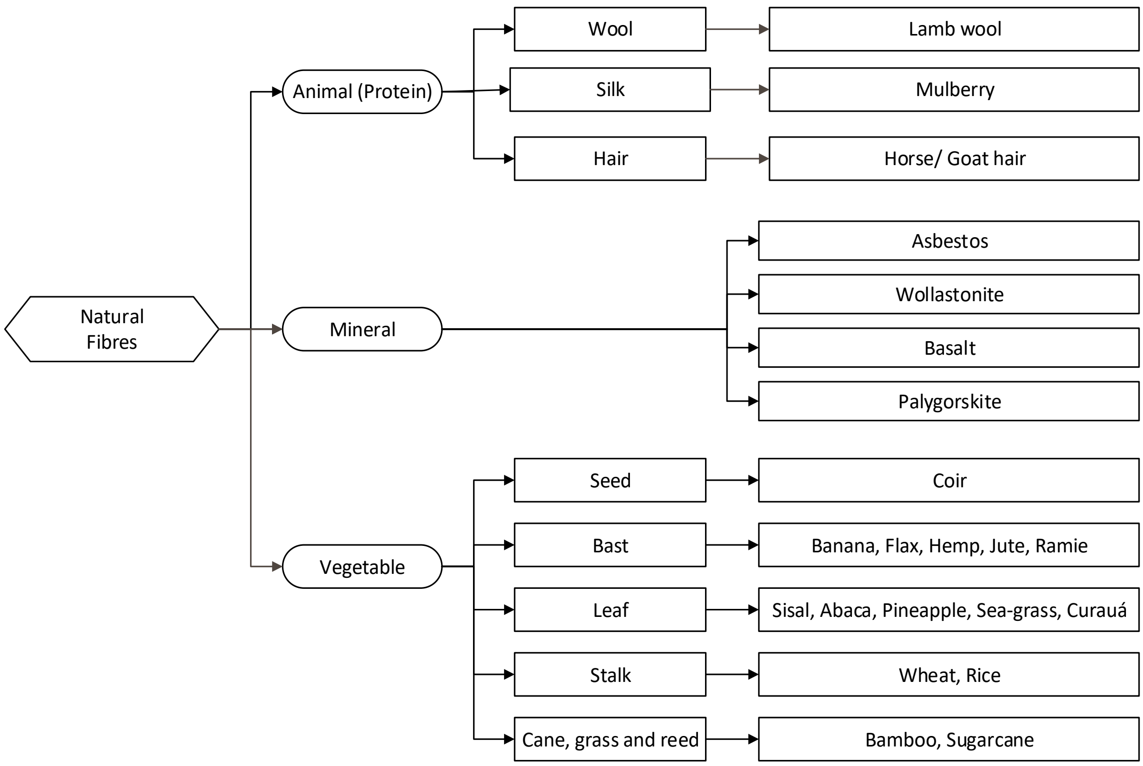



*Figure 2.4 – Classification of Natural Fibres. Source: Adapted from* (Faruk et al. 2012; Raman Bharath et al. 2015).

### *2.4.1 Mineral fibres*

Mineral fibres are natural fibres produced from mineral sources. Nevertheless, the production process is industrial, from melting stones, and sometimes industrial waste, into fibres. For this reason, the embodied energy and carbon emission associated with these fibres is much higher than the other classes of natural fibres. (Shi et al. 2020; Wang et al. 2022)

Basalt fibre is one of the most common mineral fibres available for construction applications, they are a bio-based alternative to glass fibres since both fibres require a similar manufacturing process with the difference that basalt fibres are made from stones and industrial waste. (Acar et al. 2017)

Firstly the basalt (as stones) is melted under high temperature, the basalt magma is then poured into a water tank where it quickly cools and gets a glass structure that is then extruded as basalt fibres. (Yang et al. 2022)

Research from 1998 concluded that basalt fibres effectively prevented segregation in a fresh concrete mixture and the authors suggested them as a replacement for steel fibres, especially for marine applications when the steel would not resist to the aggressive environment without oxidation or on structures exposed to high temperatures, as the fibres can resist up to 400ºC. (Jamshaid and Mishra 2016; Mahltig 2017; Guo et al. 2019)

The equivalence between steel and basalt fibres is uncertain but literature indicates that 1kg of steel would offer similar mechanical properties to 9.6kg of basalt. The authors have suggested that basalt fibres would improve the concrete compressive strength in a concentration up to 0.15% of the volume. However, another study compared mixes with different proportions of both basalt and steel fibres and the authors have not found a direct proportion. According to their results, basalt fibres were able to improve the concrete tensile strength only when added up to 1.0% by the volume, while steel fibres still increased the peak strength with higher volumes. (Jamshaid and Mishra 2016; Jabbar et al. 2021)

Given that there is no direct correlation between the dosages of steel fibres and basalt fibres, and both require industrial processes for their production, substitution with an environmentally friendly alternative would substantially mitigate the current environmental impact of FRC production.

Adopting eco-friendly alternatives to steel and basalt fibres would represent a substantial environmental improvement in FRC production. By reducing the demand for energy, water and natural resources, the manufacturing of these alternatives would significantly lower the environmental impact of FRC production. Therefore, the implementation of such measures would be crucial in promoting the sustainable development of the construction industry, whilst protecting the environment.

Table 2.2 presents values found in the literature regarding some properties of basalt fibres that will be used for discussion in comparison to values obtained in the experimental part of this research.

*Table 2.2 – Properties of basalt fibres.* (Ho et al. 2012; Sanjay et al. 2018)

| ***Type of fibre*** | ***Density g/cm³*** | ***Diameter (µm)*** | ***Tensile Strength (N/mm²)*** | ***Elastic Modulus (GPa)*** | ***Elongation at Break %*** |
|---|---|---|---|---|---|
| Basalt | 2.6 | 7-15 | 4150-4840 | >40 | 3.2 |

In this study, basalt fibres were used as a benchmark, along with polypropylene and steel fibres, performed to evaluate the suitability of vegetable fibres for concrete reinforcement.

### *2.4.2 Vegetable fibres*

Vegetable fibres are extracted from different parts of vegetal sources. They are commonly applied to the textile, sports, construction, automotive, and aircraft industries. In terms of environmental impact, authors suggest that vegetable fibres present negative carbon emission and affirm that the same size land for agriculture purposes can sequestrate 93kg of $CO_2$, while an animal farm emits 28kg of $CO_2$. (Dissanayake et al. 2009; Karlsson and Röös 2019)

Their major application in construction is for non-structural composites, however, with the environmental awareness growing in the past decades they have been researched for other applications. (Swamy 1990; Furtado et al. 2018)

The challenges impacting the safe use of plant-based fibres for structural purposes were brought to attention in 1990 and, since then, substantial research has been done addressing the matters that jeopardize their structural application.

The structural material most used in construction is concrete. As previously mentioned, it is a brittle material and requires reinforcement when submitted to non-exclusively compressive stresses. Depending on the type, a singular vegetable fibre in nature can present significant tensile strength, however, the alkaline pH of concrete is aggressive for its structure. (Gagg 2014)

A review research shows that the most studied fibres in the past decade are sisal, flax, coconut, hemp, jute, eucalyptus, and fique fibres (Figure 2.5) and the mechanical properties of those fibres, according to different authors, are presented in Table 2.3. (Laverde et al. 2022)

From the results, the high variability of the properties of vegetable fibres can, once again, can highlighted. Also, it is possible to see that flax fibres can reach higher tensile strength when in comparison to coconut, eucalyptus, fique, hemp, jute, and sisal fibres followed by hemp fibres. In terms of elastic modulus, the fibres with lower values, according to this study are sisal and fique fibres. Fique fibres also present higher elongation at break (%), while jute fibres present the lower. In terms of density, most fibres presented similar range values from 1.3 to 1.5g/cm$^3$, however, coconut fibres are denser, reaching up to 6 g/cm$^3$ and fique fibres the less dense, with values starting from 0.7g/cm$^3$.

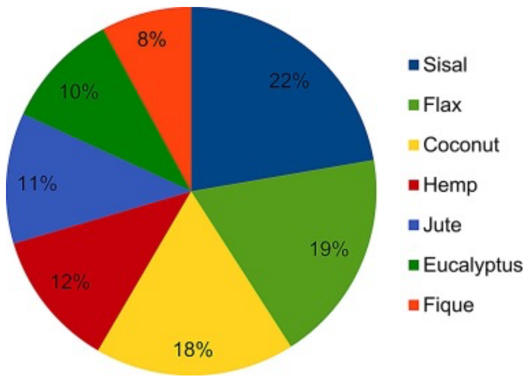



*Figure 2.5 – The percentage of experimental.* (Laverde et al. 2022)

*Table 2.3 – Properties of different vegetable fibres. Adapted from* (Sanjay et al. 2018; Laverde et al. 2022)

| *Type of fibre* | *Tensile Strength (N/mm$^2$)* | *Elastic Modulus (GPa)* | *Elongation at Break %* | *Density g/cm$^3$* |
|---|---|---|---|---|
| Coconut | 100-230 | 23.5-90 | 1.0-3.5 | 2.8-6 |
| Eucalyptus | 300-500 | 10-40 | - | 1.2-1.5 |
| Fique | 133-424 | 5.7-24 | 5.4-9.8 | 0.7-1.4 |
| Flax | 345-2000 | 27.6-70 | 1.2-3.2 | 1.4-1.5 |
| Hemp | 270-900 | 23.5-90 | 1.0-3.5 | 0.9-1.5 |
| Jute | 393-800 | 8-55 | 1.0-1.8 | 1.3-1.5 |
| Sisal | 458-720 | 9-24 | 2-4.3 | 1.3-1.5 |

In addition to their strength, the study also mentions that flax fibres possess percentage of humidity, which make them an interesting choice for use in products that come into contact with water. In comparison, they are slightly less absorbent than hemp and coconut fibres, while eucalyptus fibres would have high moisture retention.

From this list, it was possible to identify the potential of flax and hemp fibres as reinforcement of composites. While hemp fibres have been already commonly studied for this application, there is a gap in the literature for the use of flax fibres in reinforcing concrete.

#### *2.4.2.1 Flax fibres*

Flax fibres are obtained from the bast of the *Linum Usitatissimum plant*. They are usually produced in temperate regions and applications dated from ancient times can be found in the literature. Similar to other vegetable fibres, they are used to produce different materials, including luxury linen fabric in the textile and oil extraction in food industries. However, their properties have drawn the attention of scientists in different fields a few decades ago, and the plant reaches maturity after 110 days after sowing in the European region. (Melelli et al. 2022)

Figure 2.6 shows the difference between young and mature flax fibres. They are extracted from an intermediary tissue of the plan, and their presence is significantly lower on younger bast. For that reason, it is fundamental to wait until they achieve the correct age before harvesting.

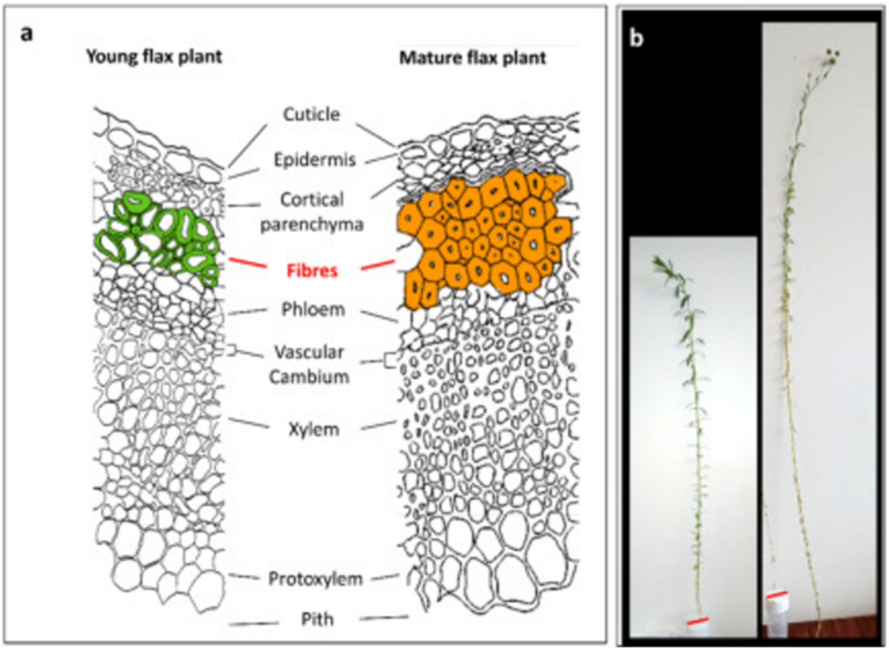



*Figure 2.6 – (a) Structure of young flax stem at young age and mature; (b) Flax plants pulled out at young (left) and mature(right) age* (Melelli et al. 2022).

In terms of mechanical properties, authors have found that flax fibres can present tensile strength significantly higher than hemp and jute fibres, in a range going from 343 to 2000MPa. The vast variability of parts of the fibres and their cross-sections is one of the reasons for the scattering in those results according to researchers. Figure 2.7 shows the structure of a flax stem. (Dittenber and GangaRao 2012; Scida et al. 2017)

Furthermore, their addition in natural form into composites can affect mechanical properties in different ways. Flax fibres were studied under compression in random and unidirectional flax fibres reinforced epoxy composites and results showed that fibres could increase the compressive strength by around 20%. (Baley et al. 2019)

Results found for flax fibre bio-composites using 3D printing indicated similar magnitude than composites containing glass fibres/ polyamide for structural applications without significantly affecting the compressive strength of the composites studied. (le Duigou et al. 2019)

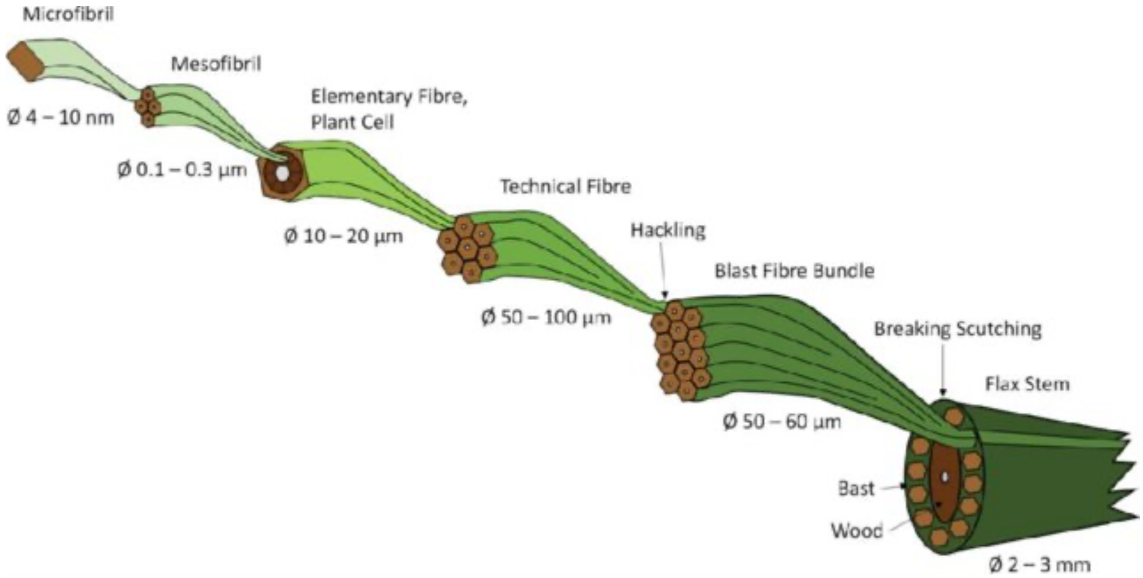



*Figure 2.7 – Structure of a flax stem.* (Güven et al. 2016)

With this research it is aimed to reduce the lack of studies evaluating the properties of flax fibres used for the reinforcement of concrete.

### *2.4.2.2 Hemp fibres*

The second type of fibre studied in this research are hemp fibres. They come from the family of *Cannabis Sativa L* and are cultivated in at least 159 countries, representing over 80% of the world. There is a variety of species in this family, and they are divided into two categories: First, the “drug-types”, or “marijuana-types” used in the pharmaceutical industry and therapeutic purposes with a significant content of hallucinogenic cannabinoids and cannabidiol. (Sander Fett et al. 2021).

Second, the “fibre-types”, or “hemp-types” grown for fibres extraction and industrial applications, including this work. Figure 2.8 shows an industrial hemp plantation and hemp fibres. As an abundant source of fibres, their extraction is made from the hemp bast and they may require surface treatment to become suitable for the application, they are intended for. (Godara 2019; Yamamuro et al. 2021)

(a) (b)

*Figure 2.8 – (a) Industrial hemp plantation (unknown author), (b) Hemp fibres.*

The main application of hemp fibres in construction is on the production of hempcrete, or hemp-lime. The composite is produced from a mixture of the hemp plant, combined with lime-based binder and water. The composite is a lightweight material with low thermal conductivity used for insulation in construction with a non-structural application. Researchers have found that the life cycle assessment of hempcrete generates a negative net carbon resulting from the carbon sequestrated on the plant growth stage and the carbonation of the lime binder. (Ip and Miller 2012; Boutin and Flamin 2013; Escadeillas et al. 2013; Arehart et al. 2020)

The addition of hemp fibres directly into concrete has been studied for years and the result is a structure with low thermal conductivity, lightweight and better acoustic insulation but with potentially lower mechanical properties. In nature, hemp fibres present properties that can vary considerably among research conducted by different authors. The range of their tensile strength goes from 270 to 900MPa as previously shown in Table 2.3. (Duval et al. 2011; Faruk et al. 2012; Ho et al. 2012; Ali et al. 2018; Laverde et al. 2022; Verma et al. 2022)

In general, vegetable fibres present interesting mechanical properties, however, as biodegradable materials, their properties require a deeper understanding of specific properties.

### *2.4.3 Current challenges related to the use of vegetable fibres*

A work pointing out challenges related to the use of vegetable fibres to reinforce cement composites was published in 1990 bringing up for the first time the potential issues related to the topic that are still relevant for current studies. (Swamy 1990)

The first concern of vegetable fibres for the application, according to the author, is their low elastic modulus, which could be problematic for certain concrete structures that require a high elastic modulus. However, for reinforcing concrete structures that are expected to withstand impact, vibration, or other conditions that could lead to brittle failure, a lower value for this property becomes advantageous in reducing the brittleness of the concrete. (Cai et al. 2022; Wang et al. 2022)

Secondly, the variability in the properties of fibres was highlighted, which can be unpredictable even if they are of the same type. As they are natural, the composition of their structure may vary depending on factors such as weather conditions, soil quality, plant position, and age during cultivation. To address this variability, different surface treatments have been proposed to reduce the irregularity of their properties. (Feyisetan Adekunle 2015; Kumar Sinha et al. 2017; Ahmed et al. 2022; Rahimi et al. 2022b)

The author also discussed the susceptibility of fibres to fungal and insect attacks, which could reduce their durability. However, it is now known that degradation of fibres in composites can be caused by various environmental factors, such as moisture, heat, and exposure to UV light, rather than biodeterioration. (Furtado et al. 2018)

Most current studies address deterioration caused by the alkaline pH of concrete as the main cause of fibre degradability. It is important to consider this parameter as it directly affects the lifespan of the concrete structure in the long term. While the short-term effects of fibres are positive, as they reduce the incidence of cracks caused by plastic shrinkage in the early days of concrete, concerns related to their long-term impact were already being addressed back in the 1990s. (Furtado et al. 2018; Laverde et al. 2022; Malalli and Ramji 2022; Miraoui et al. 2022)

Ahead in this section, the challenges above will be discussed according to relevant techniques developed by authors in the past 3 decades to overcome these challenges.

#### *2.4.3.1 Fibres behaviour under alkaline pH*

An alkaline environment will naturally change the structure of vegetable fibres. As previously said, the pH of concrete ranges from 12.5 to 13.8, which is highly alkaline and it degrades lignin, waxes, hemicellulose and pectin from the fibres, reducing the fibres hydrophily and turning their structure surface rougher. (Feyisetan Adekunle 2015; Cruz and Fangueiro 2016; Kumar Sinha et al. 2017; Furtado et al. 2018; Godara 2019; Laverde et al. 2022)

This degradation during the lifetime of the structure directly affects its durability as will be seen ahead. However, in a controlled environment before the use of the fibres, the degradation of these compounds is beneficial, as the cellulose left has properties that increase the transfer of stress and homogeneity of the matrix, for that reason, NaOH solution is one of the most common surface treatments currently studied, as discussed in this chapter. (Laverde et al. 2022)

#### *2.4.3.2 Degradation and durability*

Biodeterioration of natural fibres is the decay of the organic material of plants caused by microbes or fungi. It is favoured by the change in the moisture and temperature of the fibres and incorporated in a composite that can result from changes in the weather and UV exposure. (Hueck 2001; Furtado et al. 2018; Dungani et al. 2019; Summerscales 2021)

Although biodegradability is an advantageous property of the production of composites applied on disposable materials, concrete structures are designed to last, and the long-term effects of the fibres must be considered.

The pH of concrete is an important parameter as it can affect the durability of reinforced concrete structures. Typically, the pH of concrete ranges from 12.5 to 13.8, which is highly alkaline. This alkalinity is due to the presence of calcium hydroxide, a by-product of the hydration of cement and has to be considered when selecting the reinforcement type to be adopted. (Natkunarajah et al. 2022)

There is still a lack of experiments studying the biodeterioration of vegetable fibres into concrete, but it is now well known that the moisture content in the composite can directly affect the durability of both fibres and structures. (Tolêdo Filho et al. 1999; Tolêdo Filho et al. 2000)

The water absorption, or hygroscopicity can cause another type of degradation to concrete. In a study comparing mechanical behaviour of sisal and polypropylene fibres considering the hygroscopicity, confirmed that the change in the size of the fibres can cause microcracks in between the fibres and concrete, weakening the structural bonds of the composite as shown in Figure 2.9, where (a) shows the original size of the composite, (b) the volume increased by the water absorption, causing microcracks and (c) the degradation on the fibre-matrix interface caused by the change in the volume. (Castoldi et al. 2019)

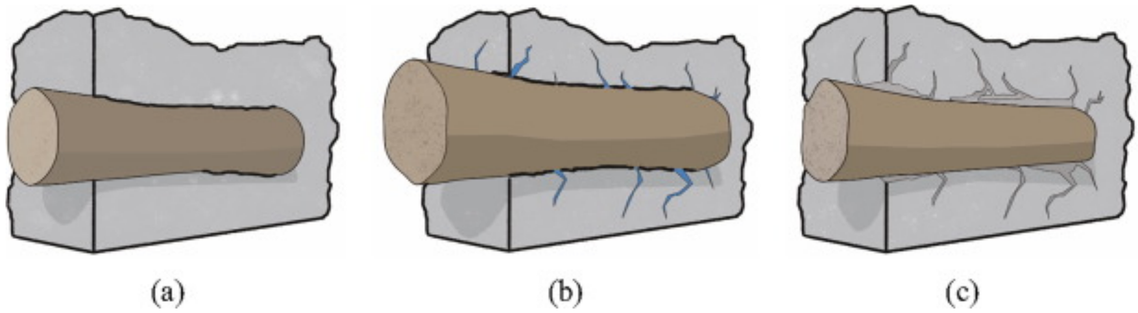



*Figure 2.9 – Effect of fibre swelling on the fibre-matrix interface.* (Castoldi et al. 2019)

Therefore, the aggressive pH of concrete damaging the fibres is not the only factor to be considered and the natural behaviour of the fibres can also damage the concrete.

### *2.4.3.3 Variability of properties*

Finally, the last challenge related to vegetable fibres was the unpredictability of their properties. Looking into mechanical, physical and chemical properties, their composition will vary according to, not only the part from the plant where they are being extracted, but also to the soil type where they were cultivated, the weather, quality of water, etc. (Saha et al. 2012; Jones et al. 2017; Furtado et al. 2018)

To estimate the performance of natural fibres and understand their properties it is ultimately important to characterize their chemical, physical and mechanical properties. This will allow an understanding of their behaviour and needs before defining potential applications, including types of composites reinforcement.

### *2.4.4 Properties of natural fibres*

In this section, some of the relevant physical, chemical and mechanical properties of vegetable fibres will be explained. As previously mentioned, the properties of fibres extracted from the same source can present a broad range of results due to the natural variability of plants.

#### *2.4.4.1 Physical properties*

Length, hygroscopicity, diameter and density are some of the physical properties of the vegetable fibres commonly studied and are characteristics measured on materials without changing their chemical composition.

##### *2.4.4.1.1 Hygroscopicity*

Hygroscopicity was defined as the ability of a material to absorb water in a reversible way (non-structured). Back in 1999, this property did not have a quantitative methodology to be measured, leading to various non-standardized procedures hindering the comparison between different authors. (L. Ford and Willson 1999)

Nowadays, different standards were developed to guide the hygroscopicity characterization of certain materials. However, there is still no specific standard to evaluate the capacity of water absorption of vegetable fibres. (Carvalho et al. 2013; Melo et al. 2021)

Moisture absorption can be considered the property with a major impact on the mechanical performance of the fibres for reinforcement of composites as the excess of humidity can generate delamination of the structure of the fibres. (Mokhothu and John 2015)

As there is still no specific method to quantify the hygroscopicity of natural literature suggests an analysis of the water absorption directly on the composite produced. The correlation between water absorption and penetration on concrete was evaluated, concluding that the lower water penetration, the lower would be the water absorption, as the difficulty of fluids to penetrate the matrix is increased. Therefore, for this work, the property evaluated will be the water penetration of concrete, to measure the level of water penetration of concrete. (Célino et al. 2014; Sekhar et al. 2018)

###### *2.4.4.1.2 Diameter*

Another physical property often studied is the diameter. Depending on the size of the fibres, their diameter can be measured by using a digital microscope as shown in Figure 2.10. The variability in the diameter of fibres for all types of plants is a topic considered in the past 40 years. (Mukherjee and Satyanarayana 1984)

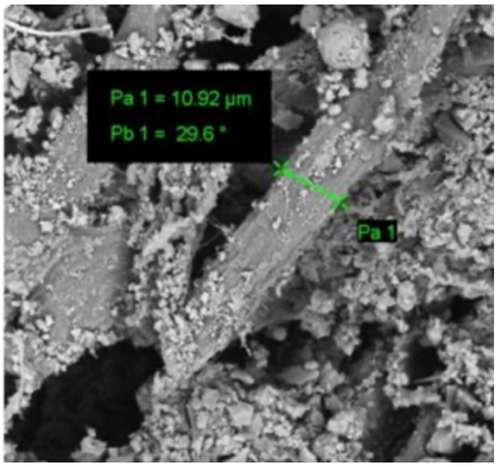



*Figure 2.10 – Diameter of Flax fibres* (Assaedi et al. 2014).

Table 2.4 presents different values obtained for the diameter of the fibres from the same sources in the past years. Confirming the concerns brought up by authors in the 80s, it is clear how the diameters can vary. Looking back to Figure 2.10, although the authors measured diameters similar, it is visible that if other regions were selected, larger and smaller values could be obtained as vegetable fibres are not uniformly shaped as non-natural fibres would be.

*Table 2.4 – Fibres and diameters according to different authors*

| ***Fibre*** | ***Diameter (μm)*** | ***Reference*** |
|---|---|---|
| Flax | 10-80 | (Dai 2006; Assaedi et al. 2014) |
| Hemp | 4-800 | (Prasad and Sain 2016; Sauvageon et al. 2018) |

Although the variability of diameters is a property important to be used to calculate mechanical properties, by itself, does not provide enough parameters to predict the behaviour of the fibres in the concrete matrix.

To evaluate a possible correlation between diameter and the mechanical performance of the fibres, studies analysed stems of flax fibres and were not able to identify a pattern relating to the diameter and the tensile strength of the fibres. While, for hemp fibres, an inverted proportion was observed, fibres with smaller diameters presented higher elastic modulus and tensile strength than larger fibres. (Shahzad 2013; Alcock et al. 2018; Sauvageon et al. 2018)

*2.4.4.1.3 Density*

Finally, the last physical property included in this review is density. Several authors include the low density of vegetable fibres as one of the main advantages of their use for reinforcement of composites. The general density of plain concrete moisture ranges from 2200-2500kg/m$^3$. (Xia et al. 2009)

As the density of a composite is calculated by a weighted average of density and volume of each compound, when materials with lower specific gravity are added into a matrix, the density of the final material will be also reduced. (Laverde et al. 2022)

As showed in Table 2.3, there is also a high variability of values obtained for densities on vegetable fibres  and it was not found in the literature an additional explanation to justify such a range apart from the natural variability already mentioned it is expected that the variability on singular vegetable fibres does not affect results obtained for the final concrete mixture reinforced.

Although physical properties are important for the characterization of the fibres, after a literature review, it was possible to understand that, apart from diameter that will be used to calculate some mechanical properties, hygroscopicity and density are properties more relevant to be studied in the final composite instead of on the fibres isolated.

### 2.4.4.2 *Mechanical properties*

Mechanical properties were defined in 1963 as "the stress-strain behaviour of materials in a macroscopic magnitude allowing the prediction of fundamental factors for engineering design". (Marin 1963)

In another words, they are the properties presented by a material when a load is applied. They can be uniaxial in compression or tension, or also result from a combination of loads in two or more directions originating from shear forces, torsion or bending moments as shown in Figure 2.11. (Gao et al. 2022; Wen et al. 2022)

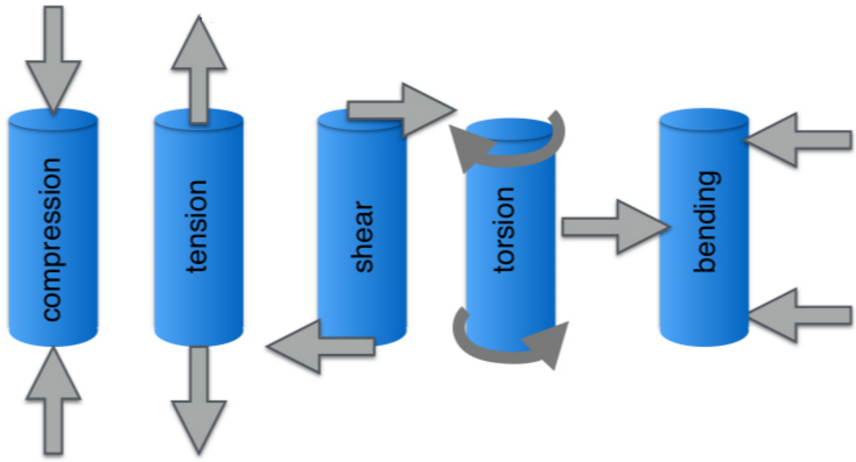



*Figure 2.11 – Types of loading (*Stress and strain: Mechanical properties of materials *[no date])*

The mechanical properties of fibres that are usually evaluated and considered for concrete reinforcement are tensile strength, elastic modulus (Young's Modulus) and percentage of elongation at break. (Shireesha and Nandipati 2019)

The three parameters can be measured from the same test. During the test, a load (F) is increased and the change in the length of the specimen ($\Delta l$) is measured until rupture, using the cross-section area ($A_0$) of the specimen it is possible to convert load into stress ($\sigma$), by using Equation 2.1.

$$\sigma = \frac{F}{A_0} \quad \text{Equation 2.1}$$

In the same way, from the change in the length, it is possible to calculate the strain (ε), as expressed in Equation 2.2 where $l_0$ is the initial measurement.

$$\varepsilon = \frac{\Delta l}{l_0} \quad \text{Equation 2.2}$$

From these parameters it is possible to plot a stress (σ) vs strain (ε) diagram. As shown in Figure 2.12, in a general diagram, the segment between the origin and A represents the elastic zone, meaning the capacity the material has to resist stress with a recoverable strain, without affecting the original format (resilient modulus). (Sun 2016; Ali Zangena 2018; Usman and Masirin 2019; Zeng et al. 2020)

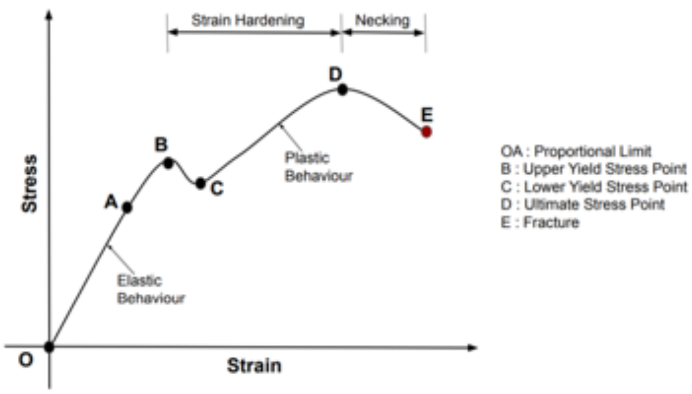



*Figure 2.12 – Stress-strain diagram. (*Stress-Strain Curve: Strength of Materials - SMLease Design *[no date])*

B represents the yield point, defined as the stress outside the recoverable capacity of the material, causing greater deformation under a small increase on the tensile load and the section between A and C represents the begin of the plastic behaviour in the material from where deformation becomes permanent. The section between C and E represents an increased capacity for resisting strain that will achieve a peak and lead to the rupture (point E). (Behera and Hari 2010; DeArmitt 2011; Abdewi 2017; Vaidya and Pathak 2018)

Point D represents the ultimate tensile strength or just tensile strength, which is one of the properties analysed in this work.

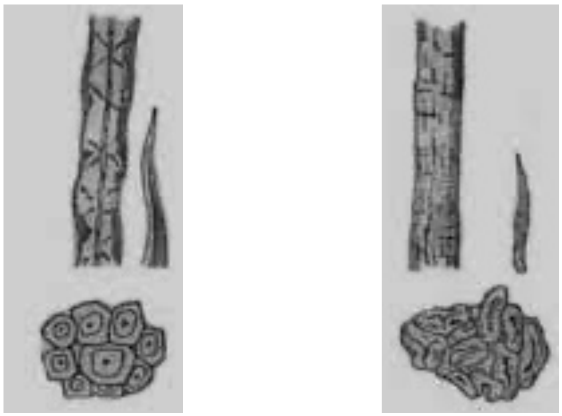
*2.4.4.2.1 Tensile strength of fibres*

The tensile strength refers to the maximum axial stress that a material can resist before failure when stretched or pulled. The load can be measured experimentally and require specific apparatus. (Pal et al. 2021)

One of the current methods available to measure it is the 'Standard Test Method for Tensile Strength and Young`s Modulus of Fibres'. The guidelines direct how to calculate the tensile strength and Young's modulus of fibres and its procedure will be further detailed in the next chapter of this work. However, as it is a standard not exclusively created to evaluate vegetable fibres, some key points are relevant to be discussed in this literature review. (ASTM International 2020)

As previously seen, the diameters of fibres can vary on a broad range, considering that, it is very unlikely that the cross-section of vegetable fibres will be a perfect circle and the phloem (vascular tissue of plants) directly affects the cross-section of the fibres. (Zhang et al. 2016)

Figure 2.13 shows the general shape of phloem for flax, hemp, and jute fibres according to the authors. Although flax fibres present a cross-section closer to a circle, hemp fibres appear to have a more undefined shape.



*(a)* *(b)*

*Figure 2.13 – Phloem shapes of (a) flax and (b) hemp. Adapted from* (Zhang et al. 2016).

The unpredictability on the cross-section is addressed by the standard as the variability along a fibre can significantly affect the results obtained for tensile strength and elastic modulus. For this reason, when performing tests on singular fibres, the procedure highlights the importance of measuring the diameter using the appropriate apparatus, measuring the diameter as close as possible to the fractured area instead of adopting an average value for all the specimens.

#### *2.4.4.2.2 Elastic Modulus/ Young's Modulus of fibres (E)*

Elastic modulus is the property that defines how stiff or rigid a material is. Graphically, it can be calculated as the slope of the region where the material presents an elastic behaviour. Looking back at Figure 2.12, it is the rate between stress ($\sigma$) and strain ($\varepsilon$) in the elastic zone of the material (O and A) and can be calculated through Equation 2.3.

$$E = \frac{\sigma}{\varepsilon}$$ Equation 2.3

Flexible materials present lower values for modulus of elasticity, while stiffer materials present higher values, which allows an evaluation of the stress possible to be applied to a material to keep it in its elastic zone and to set a range of stress where there will be plastic deformation (Donnet et al. 2003; Zhang et al. 2016; Vaidya and Pathak 2018).

*2.4.4.2.3 Elongation at break (%)*

Elongation at break is the strain at the rupture point and represents the capacity of the fibres to resist changes in their shape before failure, calculated through Equation 2.4. (Djafari Petroudy 2017)

$$Elongation\ at\ break(\%) = \frac{l_{at\ rupture} - l_0}{l_0}$$ Equation 2.4

It differs from the elastic modulus as it is calculated only based on the length of the fibres, vegetable fibres present higher elongation at break than synthetic fibres, and although it might lead to assume that they can increase the strain of reinforced composites, no evidence was found in the literature to affirm that. (Jones et al. 2017)

Results obtained by different authors could not confirm any direct relation between elongation at break, elastic modulus or tensile strength and, again, the high variability of their properties needs to be addressed as they could likely interfere with the performance of the composite reinforced. (Mohanty et al. 2000; Du et al. 2015; Santana et al. 2021)

#### 2.4.4.2.4 Variability of mechanical properties of vegetable fibres

After an overview of the mechanical properties listed in Table 2.3, it is possible now to better understand the mechanical behaviour of flax and hemp.

In terms of ultimate tensile strength, different authors have proven that flax fibres are stronger than then hemp fibres, achieving up to 2000N/mm$^2$, while coconut is the type of fibre with smaller tensile strength.

For elastic modulus, jute can present the lower values among the sources studied, however, the range of its elongation at break is also smaller, while flax and hemp fibres present similar behaviour. Although there is extensive research on the topic, it is also possible to see again the high variability in the results. For that reason, surface treatments have been studied. Before a deeper discussion on the topic, it is important to first understand the chemical composition of the fibres, and that is the topic discussed next.

### 2.4.4.3 Chemical composition

Chemical composition is evaluated at a molecular level of materials, and directly affects the performance of a composite reinforced with vegetable fibres since layers of the fibres contain different compounds with various properties. (Pettersen 1984; Laverde et al. 2022)

Cellulose, hemicellulose, lignin, waxes, and pectin are the main compounds present in the chemical structure of vegetable fibres and Table 2.5 summarizes the composition of flax, hemp, and jute fibres by weight percentages according to values found in the literature.

*Table 2.5 – Chemical Composition of flax, hemp and jute fibres.*

| ***Fibre*** | ***Cellulose (wt%)*** | ***Hemicellulose (wt%)*** | ***Lignin (wt%)*** | ***Waxes (wt%)*** | ***Pectin (wt%)*** | ***References*** |
|---|---|---|---|---|---|---|
| Flax | 62-80 | 12-20 | 2-5 | 1.5-1.7 | 1.4-6.7 | (Faruk et al. 2012; Wang et al. 2015; Laverde et al. 2022) |
| Hemp | 68-74 | 15-22 | 4-10 | 0.8 | 0.8-2.5 | (Ali et al. 2018; Benin et al. 2020; Laverde et al. 2022) |

There are various methods to quantify the chemical composition, and the extraction is commonly adopted in studies interested specifically in the quantitative amount of certain compounds from plants, for example, an analysis led to evaluate the extracts present to be used in the pharmaceutical industry. More simply, for composites application, a spectroscopic analysis allows a general identification of the presence of compounds. (Speakman 1950; Wang et al. 2003; Sasidharan et al. 2011; Dhali et al. 2021; Sun et al. 2022)

As will be further explained in the next chapter, Fourier-transform infrared spectroscopy (FTIR) is the methodology selected in this work to evaluate the presence of each compound on the fibres studied, values for the main infrared transition of hemp fibres as shown in Table 2.6.

With a better understanding of their quantities and how each compound can be identified, some of their properties and functions will be detailed ahead, allowing a deeper comprehension of their potential beneficial or prejudicial effects on the concrete matrix.

*Table 2.6 – Main infrared transition for vegetable fibres* (Dai and Fan 2010).

| ***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, 1362 | 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, 1019, 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 |

2.4.4.3.1 *Cellulose*

Cellulose is considered as the most abundant biopolymer on Earth and described in 1838 as a fibrous material present on plant tissues capable of resisting extraction using organic and aqueous solvents and identified its molecular formula as $C_6H_{10}O_5$. (McNamara et al. 2015)

Over 6 decades later, it was discovered that cellulose was a material consisting of glucose units interatomically linked with bonds extremely stable, which led to what is currently known about cellulose being the structure that keeps the plant steady. Figure 2.14 shows a schematic representation of the chemical structure of cellulose. (Wolfenden et al. 1998; Brigham 2018)

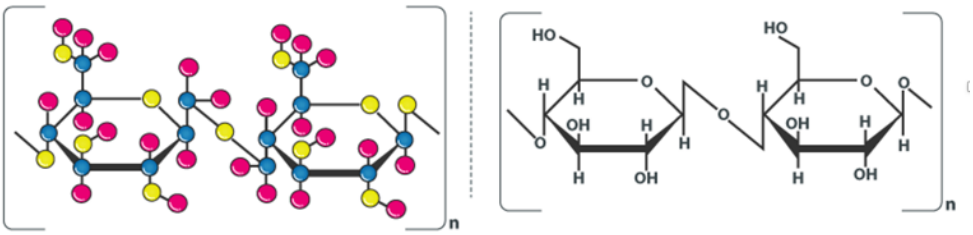


*Figure 2.14 – Structure of cellulose (Unknown author).*

Although it is not the scope of this work, several types of bacteria can synthesize cellulose, producing the bacterial cellulose. The chemical formula is the same, however, the configuration of its structure is different as shown in Figure 2.15. Different from vegetable cellulose, this is a permeable material often used for biomedical applications. (Manoukian et al. 2019; Lai 2022)

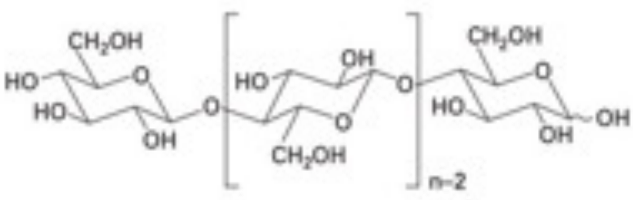


*Figure 2.15 – Chemical structure of bacterial cellulose.* (Ciechańska et al. 2012)

Cellulose is the most abundant natural polymer, and while it can be found in its pure form, it is more common to be found mixed with other substances in the vegetable structure, such as hemicellulose and lignin as showed in Figure 2.16. (Michelin et al. 2014; Naidjonoka et al. 2020)

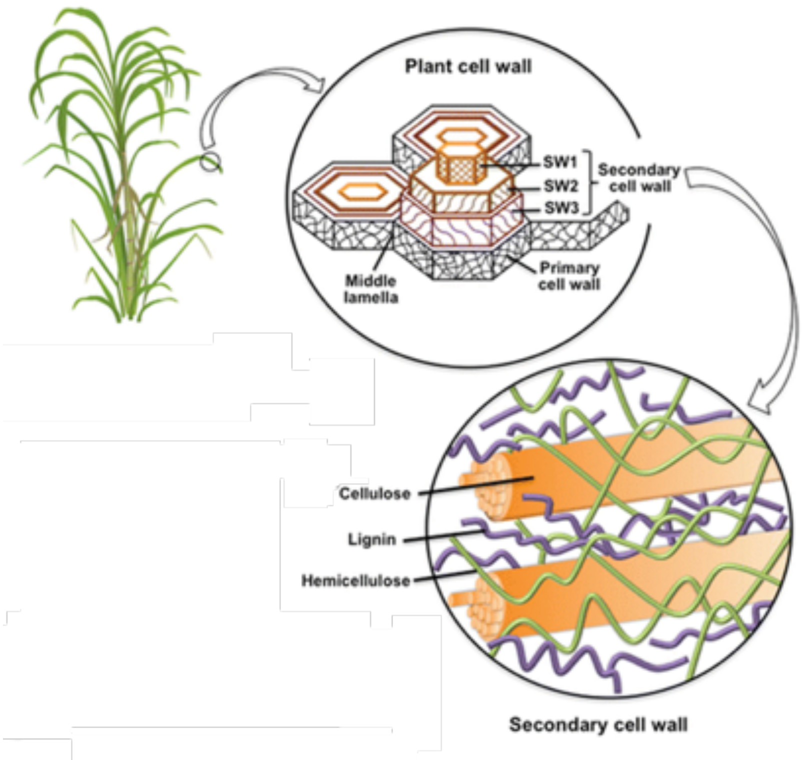



*Figure 2.16 – Cellulose involved by lignin and hemicellulose. Adapted from* (Michelin et al. 2014).

### *2.4.4.3.2 Hemicellulose*

Hemicellulose is the second most predominant component of the plant cell. Defined as polysaccharides that are insoluble in water but can be extractable from the walls of the cells with alkaline solutions. Combined with lignin, they bond the plant structure together and the chemical structure of hemicellulose is non-crystalline and more open, as shown in Figure 2.17, which leads to an increased hygroscopicity. (Whistler 1993; Holtzapple 2003; Huffman 2003; Benaimeche et al. 2020; Naidjonoka et al. 2020)

As a highly biodegradable polysaccharide, it is currently used on hemicellulose-based hydrogels and other decomposable materials. Therefore, as a compound soluble in alkaline solutions, biodegradable and with high hygroscopicity, when considering the use of vegetable fibres in the reinforcement of concrete, hemicellulose does not provide properties compatible with the application: an alkaline environment, designed to last.

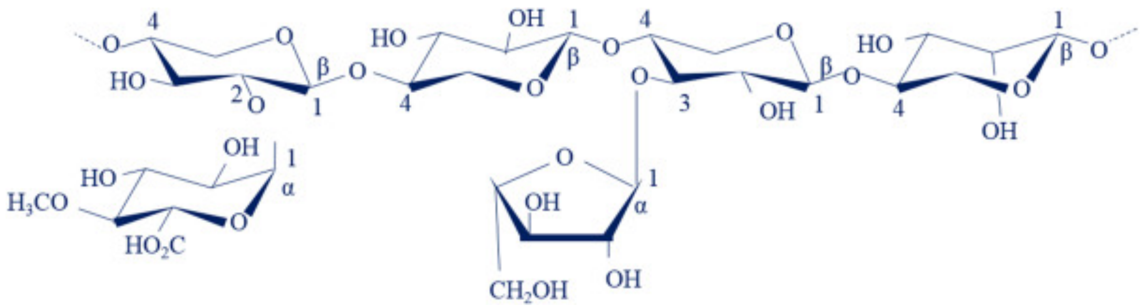



*Figure 2.17 – Structure of hemicellulose.* (Benaimeche et al. 2020)

For that reason, different surface treatment methods are being developed aiming at the removal of substances naturally present on the fibres that can become harmful to the concrete life-span and different surface treatments currently studied will be detailed in this chapter.

#### *2.4.4.3.3 Lignin*

Lignin was first described in 1838 as an incrust of cellulose, being named lignin only a few decades later. Lignin provides permeability and thermal stability to the cell wall, giving stiffness and strength to the plant structure, being essential for the vertical growth of the plants. (Santos et al. 2014; Haghdan et al. 2016).

With limited solubility and reducing the access of enzymes in the plant tissue, it also protects the plant against fungal and microbial attacks. (Verma and Dwivedi 2014)

Considered for years a low-value waste, from pulp and paper production, lignin was often used as fuel for energy production. In the past decade, it started to be used in polymers, biomedical and antioxidant activities. Figure 2.18 shows different structures of lignin, although lignin has interesting properties, its characteristic odour limits its applications. (Haghdan et al. 2016; Cassoni et al. 2022)

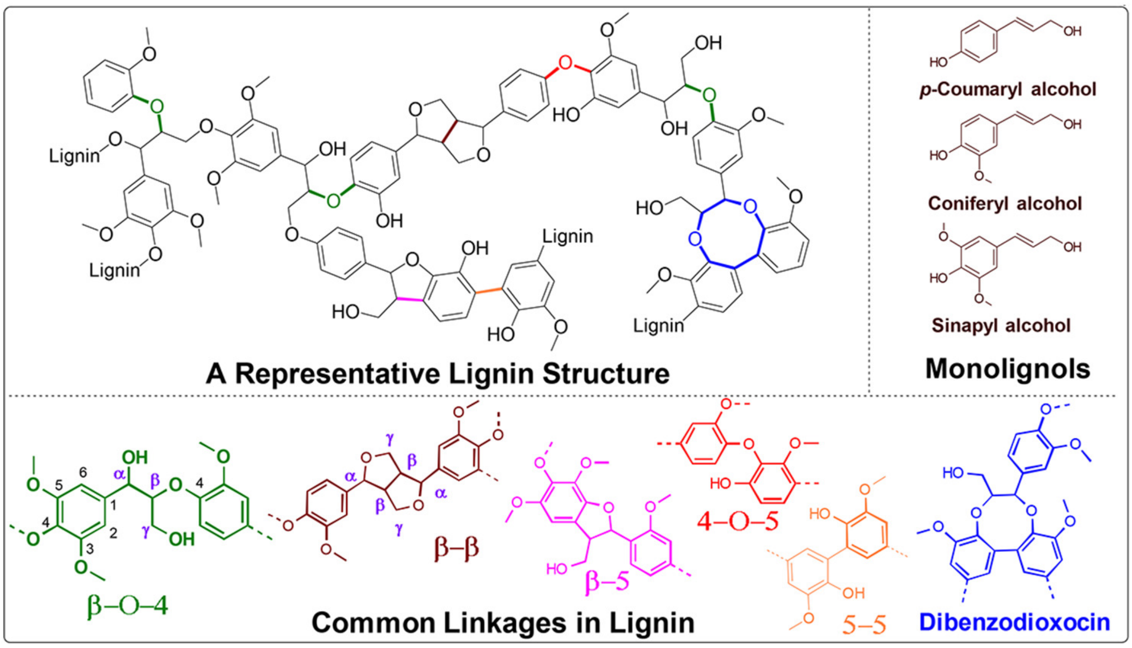



*Figure 2.18 – Lignin structure (representative and common linkages)* (Ullah et al. 2022).

### *2.4.4.3.4 Waxes*

Plant waxes are hydrophobic and lipophilic components found on external layers of plants, insects, animal skins, etc. In vegetable sources, it can be found as a continuous, coarse blade or rod-shaped projections along the cuticle part of aerial. (Tinto et al. 2017; Lan 2019)

Depending on the source, waxes present different properties, and their quality may also vary according to weather and harvesting conditions and they can be soluble in acid, acetic and alkaline solvents. (Ahmad et al. 2021)

It was not found in the literature their direct effect on cementitious matrices, however, one of the aims of surface treatments in vegetable fibres is the removal of waxes, due to their deterioration under a high pH environment. (Rahimi et al. 2022b)

### *2.4.4.3.5 Pectin*

Pectin is another polysaccharide and binds materials on the cell walls of vegetable tissues, a natural moisture retentive dressing (hydrocolloid) that can be used for various applications requiring a viscosity enhancer in the food and biomedical industries. (Pawar et al. 2008; Nasrollahzadeh et al. 2021b)

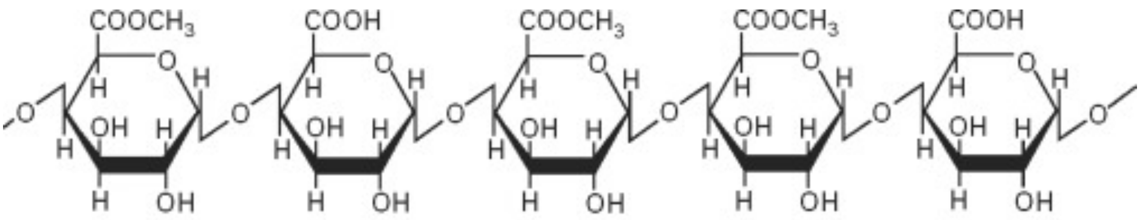



*Figure 2.19 – Molecular structure of pectin* (Pawar et al. 2008).

Pectin stabilizes and emulsifies oil and water mixtures, as explained in Figure 2.20, molecules of pectin absorb droplets of oil acting as a shield avoiding that those adjacent droplets get separated, causing a short to long-term stability in the emulsion. (Nasrollahzadeh et al. 2021a)

On cementitious composites, research showed that the presence of pectin can accelerate the setting time of ordinary Portland cement paste. Regarding the compressive strength, the addition of pectin reduced the mortar strength at an early age and slightly increased it at 28 days in quantities up to 0.5%. (Kavas et al. 2007)

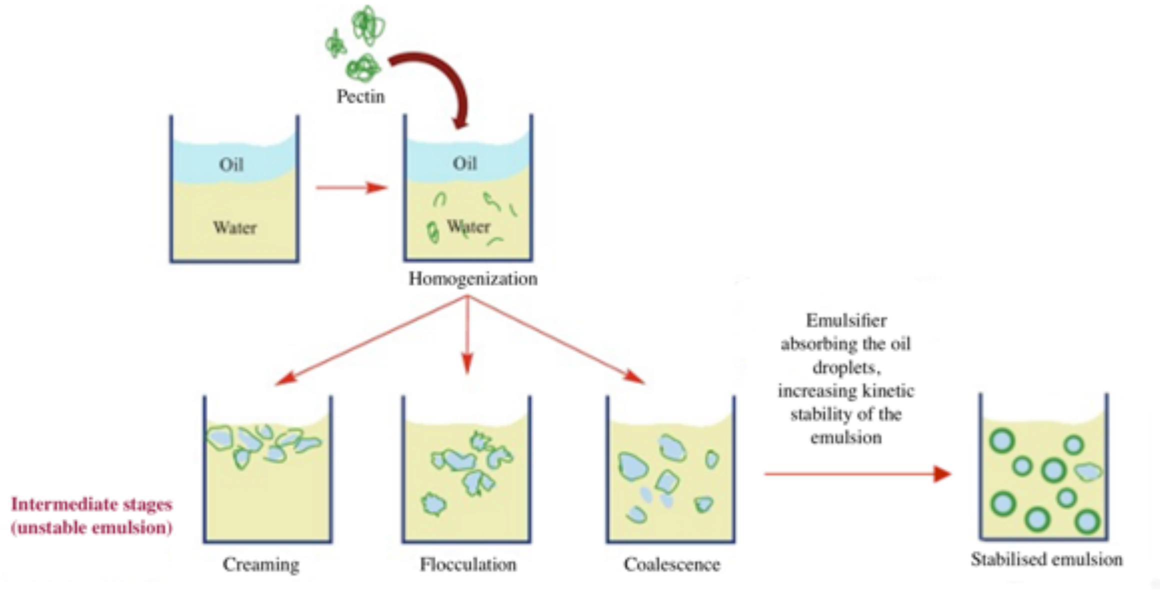



*Figure 2.20 – Use of pectin as emulgent. Adapted from* (Nasrollahzadeh et al. 2021a)

Their hydrophilic behaviour could reduce the interfacial bonds between the fibres and matrix, therefore similar to waxes, and lignin, pectin is also aimed to be removed from the vegetable fibres structure with surface treatment. (Cristaldi et al. 2010)

### *2.4.5 Surface treatment*

As introduced before, the broad variability of the vegetable fibre properties combined with the chemical behaviour of some of their compounds are factors that must be considered when evaluating their suitability for concrete reinforcement.

Aiming to minimize the presence of the degradable compounds in an alkali environment, to increase the tensile strength and elongation at break, and to reduce the elastic modulus, authors have suggested the performance of surface treatments on the fibres before their application. (Le Troedec et al. 2008; Rahimi et al. 2022b)

Different chemical, physical and biological procedures can be found in the literature. However, considering the viability and reproducibility, in this work only chemical treatments will be evaluated, seen that using plasma or other biological substances could make the reproducibility unfeasible for potential commercialization (Cruz and Fangueiro 2016; Vellaichamy and Gaonkar 2017).

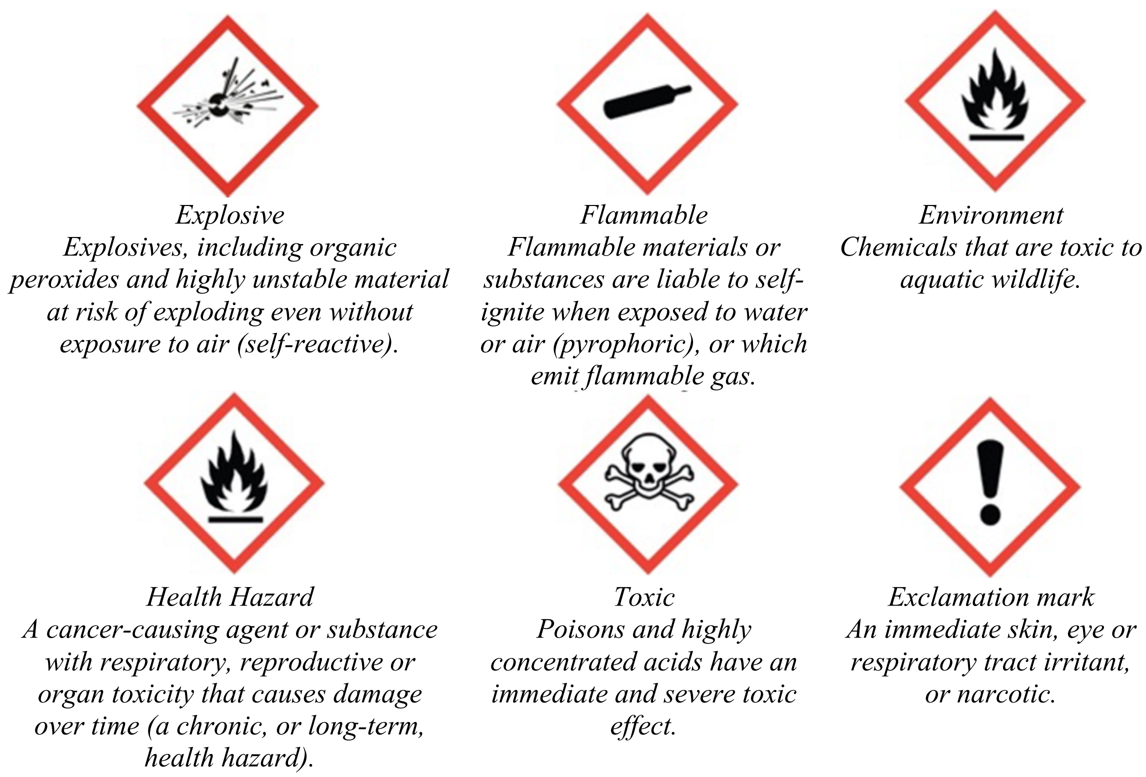
Chemical treatments provide better dimensional stability to the fibres, reducing their hygroscopicity and giving resistance to fungal deterioration, different methods of chemical surface treatments that could be used on vegetable fibres are summarized in Table 2.7. (Sood and Dwivedi 2018)

*Table 2.7 – Chemical Treatments for Vegetable fibres. Adapted from* (Sood and Dwivedi 2018).

| ***Treatment*** | ***Chemical(s) used*** | ***Chemical Structure*** | ***Concentration*** |
|---|---|---|---|
| Alkali | Sodium Hydroxide | $NaOH$ | 5%, 10%, 15% |
| Cyclohexane modification | Cyclohexane | $C_6H_{12}/C_2H_6O$ | 1:1 vol solution in ethyl alcohol |
| Fluorocarbon | Perigurard UFC | | 50 g/L |
| Modification with maleic anhydride | Maleic anhydride | $C_4H_2O_3$ | 20wt% solution in acetone |
| Permanganate | Potassium Permanganate | $KMnO_4$ | 0.005% in acetone |
| Peroxide | Benzoyl peroxide or Dicumyle peroxide | $C_{14}H_{10}O_4$, $C_{18}H_{22}O_2$ | 6% solution in acetone |
| Sulfuric Acid | Sulfuric Acid | $H_2SO_4$ | 1.0% (wt/vol) |

| | | | |
|---|---|---|---|
| EDTA | Ethylenediamine-tetraacetic acid | $C_{10}H_{16}N_2O_8$ | 5g/L |
| Stearic acid | Stearic acid | $C_{18}H_{36}O_2$ | 1% solution in ethyl alcohol |

Before discussing each option of treatment, from a health and safety perspective, it is relevant to first perform a risk assessment of these chemicals. Early in the 90s, started the implementation of the ‘‘Globally Harmonized System of Classification and Labelling of Chemicals’’ (GHS). The system consistently standardizes the labelling for chemicals providing information allowing an easy assessment of their hazards, seeking the protection of human health and the environment through pictograms representing the hazard classification as shown in Figure 2.21. (Winder et al. 2005; Hill 2010; Clark et al. 2013)



*Oxidizers*
*Chemicals that facilitate burning or make fires burn hotter and longer.*

*Corrosion*
*Materials causing skin corrosion/burns or eye damage on contact, or that are corrosive to metals.*

*Gas Cylinder*
*Gases stored under pressure, such as ammonia or liquid nitrogen.*

*Figure 2.21 – GHS pictograms for hazard classification. Adapted from* (Princeton University 2016).

Following the GHS classification, hazards associated with the chemical treatments listed above are presented in Table 2.8.

*Table 2.8 – Hazard classification of chemicals considered for surface treatments.*

| ***Chemical*** | ***Hazard(s)*** | ***Reference*** |
|---|---|---|
| Sodium Hydroxide |  | (Sigma-Aldrich 2021) |
| Cyclohexane |  | (New Jersey Department of Health 2016b) |
| Maleic anhydride |  | (INEOS Joliet [no date]) |
| Potassium Permanganate |  | (Fisher science Education 2015) |
| Benzoyl peroxide |  | (New Jersey Department of Health 2016a; Parchem 2016a) |
| Dicumyle peroxide |  | (Parchem 2016b; Thermo Fisher Sientific 2022) |
| Sulfuric Acid |  | (Teck Metals Ltd 2015; New Jersey Department of Health 2016c) |

| | | |
|---|---|---|
| Ethylenediaminetetraacetic acid (EDTA) |  | (New Jersey Department of Health 2008; ThermoFisher Scientific 2022) |
| Stearic acid | *None* | (Fisher Science Education 2014; ThermoFisher Scientific 2021) |
| Ethanol/ Acetone |  | (Merck 2014; T R 2015) |

As it is possible to see, except for stearic acid, all the treatments suggested by Sood et al. (2018) present some hazardousness. Some of the chemicals present fewer hazards, like EDTA, however, the majority are extremely dangerous for the environment and human health, including sodium hydroxide, the treatment found more commonly in the literature.

Both solvents included on the list are flammable and can cause an immediate skin, eye or respiratory tract irritation, or have narcotic effects.

Research evaluating the effects of NaOH solution for surface treatment of abaca fibres, found that alkali treatment removes wax and non-cellulosic materials from the surface of the studied fibres, increasing their surface roughness and, therefore, improving the interfacial bonding between fibre and matrix in a composite, confirming also, that the capacity of water absorption was also reduced. (Kumar Sinha et al. 2017)

In Figure 2.22, a comparison between abaca fibres after treatments using alkali solution in different concentrations is presented in a scanning electron microscope

(SEM) Image. It is possible to see that solutions in higher concentrations removes compounds from the fibres, leaving a more uniformly rough surface that could improve the adhesion between fibres and matrix.

In addition, there are certain factors to be considered with regard to the utilization of sodium hydroxide in the treatment of fibres intended for incorporation into concrete mixes. Given its aggressive nature, this substance has the potential to compromise the strength of the fibres, and the alkaline pH of the concrete may exacerbate their degradation over time. (Moraes et al. 2012).

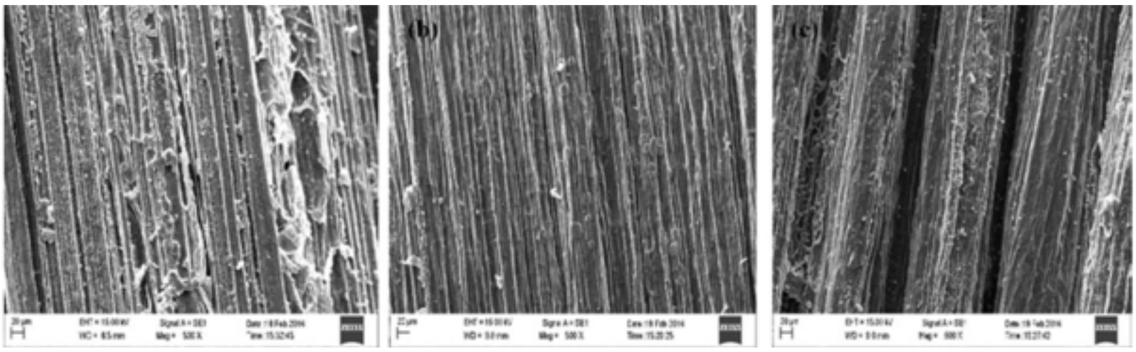


*Figure 2.22 – SEM micrographs (1000x) of abaca fibres. Untreated (left), 1% NaOH (centre) and 3% NaOH (right)* (Kumar Sinha et al. 2017).

Although it is a low-cost product, one of the main reasons to consider the adoption of vegetable fibres over other fibres is the sustainability they offer. As shown in Table 2.8, sodium hydroxide is a hazardous chemical, and their LCA will be negatively affected if a highly toxic chemical is used. (Thannimalay 2013)

A study using cyclohexane treatment, combined with NaOH indicated that it could improve the adhesion between matrix and fibres, however, the mechanical properties were reduced. (Razera et al. 2014)

Regarding maleic anhydride, despite its relatively lower risk profile compared to other treatments, it is a corrosive chemical. A comparative study evaluating the effects of sodium hydroxide, maleic anhydride, acetic anhydride, and silane treatments on hemp fibres revealed that sodium hydroxide can augment the mechanical properties of these fibres, while maleic anhydride, acetic anhydride, and silane treatments could potentially reduce their tensile strength. (Sawpan et al. 2011).

Oppositely to maleic anhydride, different studies suggest that treatments using potassium permanganate (KMnO4) can improve the properties of different vegetable fibres, reducing their hydrophilic nature and removing pectin, hemicellulose and waxes. (Li et al. 2016; Imoisili and Jen 2020; Ezeamaku et al. 2022)

Similarly to NaOH, solutions containing benzoyl peroxide, dicumyle peroxide and sulfuric acid are severely hazardous, studies suggest they can also be effective to remove unwanted compounds from the plant fibres, improving their adhesion on a composite matrix. (Sari et al. 2019; Kamath and Bennehalli 2021)

Although not common, research suggests that EDTA helps to separate the fibres with potential to increase the fibre-matrix bond in a composite and a study using fibre flour wood treated with EDTA to reinforce mortars, indicates that the final composite presented enhanced mechanical properties in comparison to untreated fibres (Le Troedec et al. 2008; Fadhel and Sabrine 2018).

Regarding stearic acid, different authors affirm that stearic acid can enhance the properties of fibres treated with stearic acid solution. As it is a chemical with no hazards involved and considering that the scope of this research is to evaluate the suitability of vegetable fibres for FRC production, focusing on the environmental impact, treatments using stearic acid becomes an interesting option in comparison to the other treatments listed. (Nayak et al. 2021; Pickering and Sunny 2021; Santiagoo et al. 2021)

Stearic acid is a common fatty acid, present in plant and animal fats, that is not toxic and is biocompatible with the human body and environment. The chemical is insoluble in water and soluble in ethanol and is commonly used in the production of cosmetics, soaps and detergent products. Research indicates that stearic acid treatment on flax fibres also removes non-crystalline compounds from vegetable fibres. (Zhen et al. 2015; Chand and Fahim 2021)

As it will be detailed in the next section, seeing all the advantages of stearic acid, this study will compare stearic acid treatments in different concentrations and for different periods with NaOH, the most common treatment used by other authors, $KMnO_4$, another treatment with potential according to the literature and EDTA, a less hazardous chemical but with not many studies about.

Recently, a study investigated the effects of linseed oil treatment of flax fibres applied on the reinforcement of mortars. According to the authors, linseed oil treatment reduces the hygroscopicity of the fibres, improving the workability of a fresh mixture. (Page et al. 2021)

Concerning mechanical properties, the authors found that both treated and untreated flax fibres would decrease the compressive strength of the studied mortar but treated fibres would present better behaviour than the untreated. They also found that the addition of fibres improves the tensile strength and slightly enhances the thermal insulation of the composite. The study was published after the practical work of this research was concluded, and for that reason, linseed oil was not included in the options of treatments, however, as a fatty acid, linseed oil contains stearic acid in its composition and results will be compared in the discussion section of this study. (Mailer 2004; CLARKE 2008)

After reviewing concepts regarding vegetable fibres and treatment options, it is possible now to go back and study properties related to the composite that is the target product of this research, the FRC.

The subsequent sections of this chapter will cover the characteristics of FRC, along with the fibres that are currently employed for this purpose, as well as the alternatives that are being investigated by the academic community.

## 2.5 Properties of fibre-reinforced concrete (FRC)

As previously introduced, FRC is produced from the addition of fibres during the concrete mix stage, aiming to enhance properties that plain concrete would not provide by itself. . (Pöhler et al. 2021)

In this section, the main properties influenced by the fibre reinforcement of concrete will be detailed, according to current standardized methods and past research.

### 2.5.1 *Consistency*

Consistency is a rheological property evaluated on a fresh concrete mixture and is directly related to the plasticity and workability of the mixture. The test is well known in civil engineering and is conducted just before concrete is moulded and specified by the Irish Standard 'Testing fresh concrete – Part 2: Slump test'. (Choi et al. 2016; National Standards Authority of Ireland 2019a)

The slump test measures the consistency and workability of concrete by determining the change in height of the concrete after it is placed in a cylindrical mould and then removed. The test is crucial to ensure that the concrete has the desired properties for its intended use.

The workability of concrete is influenced by its consistency, and as explained earlier in this chapter, the high hygroscopicity of vegetable fibres must be considered to prevent any adverse effects on the moulding or casting process of the concrete.

### 2.5.2 *Relative density*

Relative density, also known as bulk specific gravity, is the ratio of the weight of a particular material to its volume, which includes both impermeable and permeable voids. In a composite material, the final density can be determined using the Archimedes principle by summing the densities of each component multiplied by its respective volume. (Shadheer Ahamed et al. 2021)

As it has been confirmed by different authors, vegetable fibres are lightweight in comparison to concrete and their addition can decrease the density of fibre-reinforced concrete (FRC). Typically, the density of concrete is considered to be around 2400 kg/m$^3$, and depending on the application, reducing its density can be beneficial as it can enhance the insulation capability of the structure, extend moisture-curing, and increase durability. (Bremner 2008).

Lightweight concrete has a density ranging from 300 kg to 2000 kg/m3, and it is usually produced by modifying the aggregates in the mixture or by creating an air-entrained mixture. (Newman and Owens 2003; Bremner 2008)

Despite confirming that the addition of fibres can reduce the density of concrete, current literature lacks evidence to suggest that adding fibres alone can lower the density enough to classify a mixture with natural aggregates as lightweight concrete. However, a combination of lightweight aggregates and vegetable fibres can result in even lower relative densities. (Charai et al. 2022)

### *2.5.3 Water penetration*

The high hygroscopicity of vegetable fibres can directly affect the mechanical capacity of brittle composites such as concrete. This is due to the change in volume suffered by the fibres, creating internal stresses in the bond fibre-matrix and triggering the start of microcracks that could reduce the lifespan of the structure as previously shown in Figure 2.9. (Tolêdo Filho et al. 1999; Tolêdo Filho et al. 2000; Boran et al. 2016; Ferreira et al. 2017; Castoldi et al. 2019)

Considering that when fibres get in contact with water they will absorb water, the evaluation of the level of water penetration in a VFRC allows an understanding of the possible damages the structure will be susceptible to if it gets in direct contact with water. One of the methods to evaluate this parameter is given by the standard "Testing hardened concrete -Part 8: Depth of penetration of water under pressure". (Irish Standard EN 12390-8 2009)

Research on the influence of the length of palmyra fibres, into the water penetrability of fibre reinforced concrete found that FRC mixes containing shorter fibres (25mm) had a lesser depth of penetration compared to those with longer fibres (50mm). However, literature studies assessing this property for flax or hemp FRC have not been found in the literature. (Rai P. et al. 2022)

Unlike other available methods that measure water absorption by submerging the specimen in water, this method is particularly interesting as it provides an intensified procedure that can simulate scenarios where the structure is exposed to direct water, such as rainfall or possible leaks commonly seen in construction. More information on this procedure will be provided in the next chapter, Experimental Details.

### *2.5.4 Thermal insulation*

Considering the suitability of VRFC for different applications, the evaluation of thermal insulation is also relevant, especially for countries such as Ireland that are currently working on the increase of the energy efficiency in buildings to reduce the consumption of non-renewable heating sources and $CO_2$ emissions (Walsh 2021).

An efficient method to assess the thermal insulation of a composite is detailed by ISO 8301(1991) "Thermal insulation – Determination of steady-state thermal resistance and related properties – Heat flow meter apparatus" (The International Standards Organisation 1991).

The procedure for measuring heat flow on the FRC specimen will be detailed in the next section. In summary, the test involves placing the specimen between a hot and a cold chamber in a controlled and steady environment. The heat flow on the material is then measured, as illustrated in Figure 2.23.

Despite FRC being a homogenous composite, it is made up of different materials that may present different phases. Consequently, the thermal insulation of FRC is likely to vary according to factors such as the type of fibre reinforcement used, the water content, and the presence of air voids after hardening. (Taoukil et al. 2013; Bessenouci et al. 2014; Asadi et al. 2018)

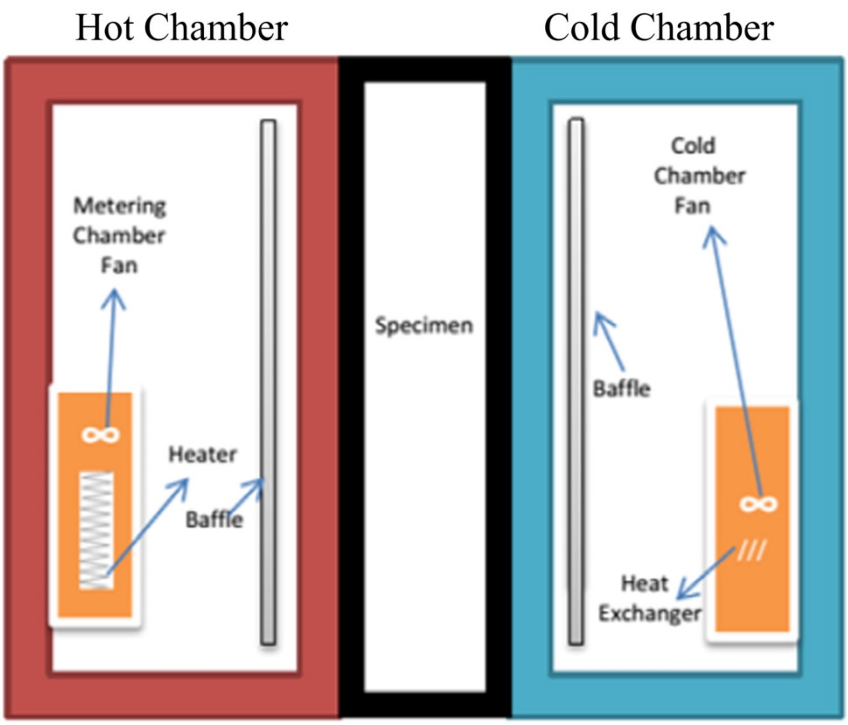



*Figure 2.23 – Steady-state of box method.* (Asadi et al. 2018)

### *2.5.5 Compressive strength*

As previously highlighted, the capacity of concrete to withstand compressive stresses is a fundamental property. Various studies have demonstrated that the addition of fibres can significantly affect the compressive strength of concrete, in comparison to plain concrete. (Siddique et al. 2012; Gupta and Kumar 2019; Alaskar et al. 2021)

The compressive strength of concrete can be influenced by various factors, including the source, length, and fibre volume fraction. Previous research has indicated that the addition of steel fibres in dosages up to 1.5% of the weight of concrete can enhance its compressive strength, irrespective of the fibre length, as illustrated in Figure 2.24. (Han et al. 2019)

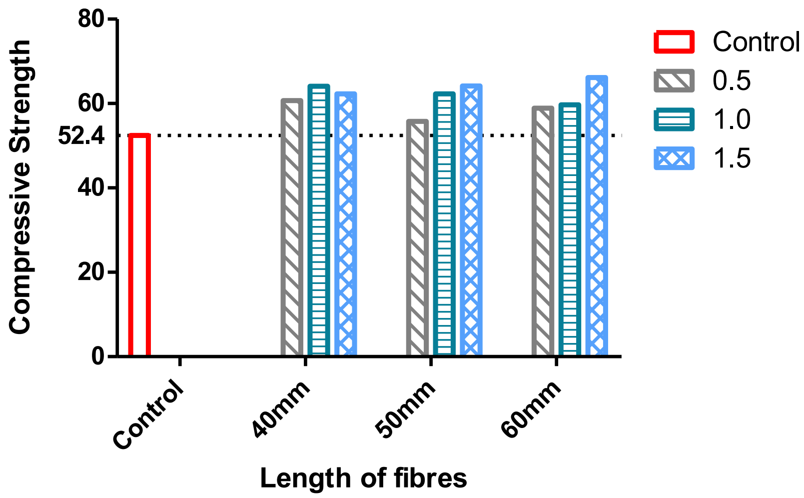



*Figure 2.24 – Compressive strength of SFRC vs length of fibres.* (Han et al. 2019)

Research suggests that the compressive strength of cement-based composites reinforced with vegetable fibres can be influenced by various factors, including the maximum aggregate size and fibre length. For instance, in the case of mortars, the addition of fibres ranging in length from 10mm to 15mm at concentrations of up to 0.75% has been shown to improve compressive strength. (Zakaria et al. 2015; Zakaria et al. 2017)

When it comes to natural fibre reinforced concrete (NFRC), the impact of fibres on the compressive strength is not well established. While some studies suggest that the inclusion of fibres up to 50mm in length can enhance all mechanical properties, others suggest that compressive strength may decrease while flexural tensile strength and energy absorption improve. (de Gutiérrez et al. 2005; Kim et al. 2012; Sawsen et al. 2015; Page et al. 2017; Page et al. 2019; Ghaffar et al. 2020)

The evaluation of compressive strength is commonly performed using standardized tests, such as 'Testing hardened concrete, Part 3: Compressive strength of test specimens.' This standard outlines the dimensions of cubic and cylindrical specimens based on the maximum aggregate size utilized in the mixture. The procedure defines three types of acceptable failures for cube specimens, as illustrated in Figure 2.25. (National Standards Authority of Ireland 2019c)

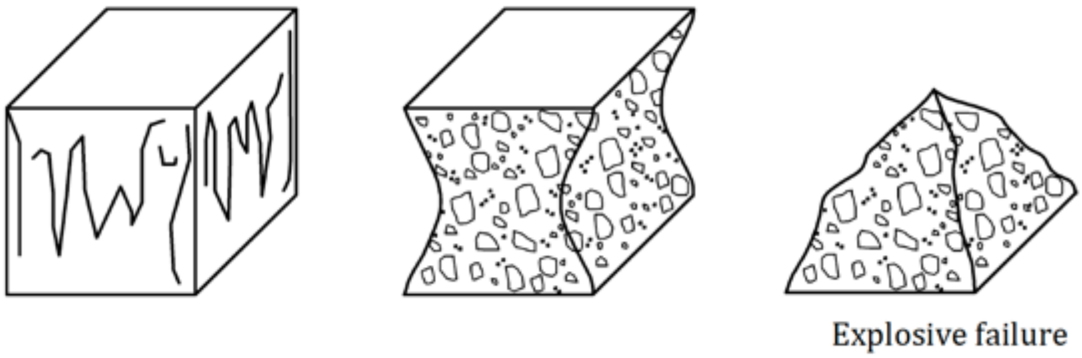



*Figure 2.25 – Types of failure satisfactory for cube specimens.* (National Standards Authority of Ireland 2019c)

The optimal failure modes for cube specimens are characterized by uniform cracking across all lateral faces and minimal or no damage to the two faces situated between the loading plates.

The standard also provides examples of unsatisfactory failures that may occur in cubic specimens, as illustrated in (Figure 2.26). In the case of an unsatisfactory failure, the cracks occur non-uniformly, and a tensile crack (T) may appear on isolated faces, indicating that the load distribution over the specimen was non-uniform. The determination of the type of failure must be carefully conducted, and results obtained only from specimens with satisfactory failures should be considered. (National Standards Authority of Ireland 2019c)

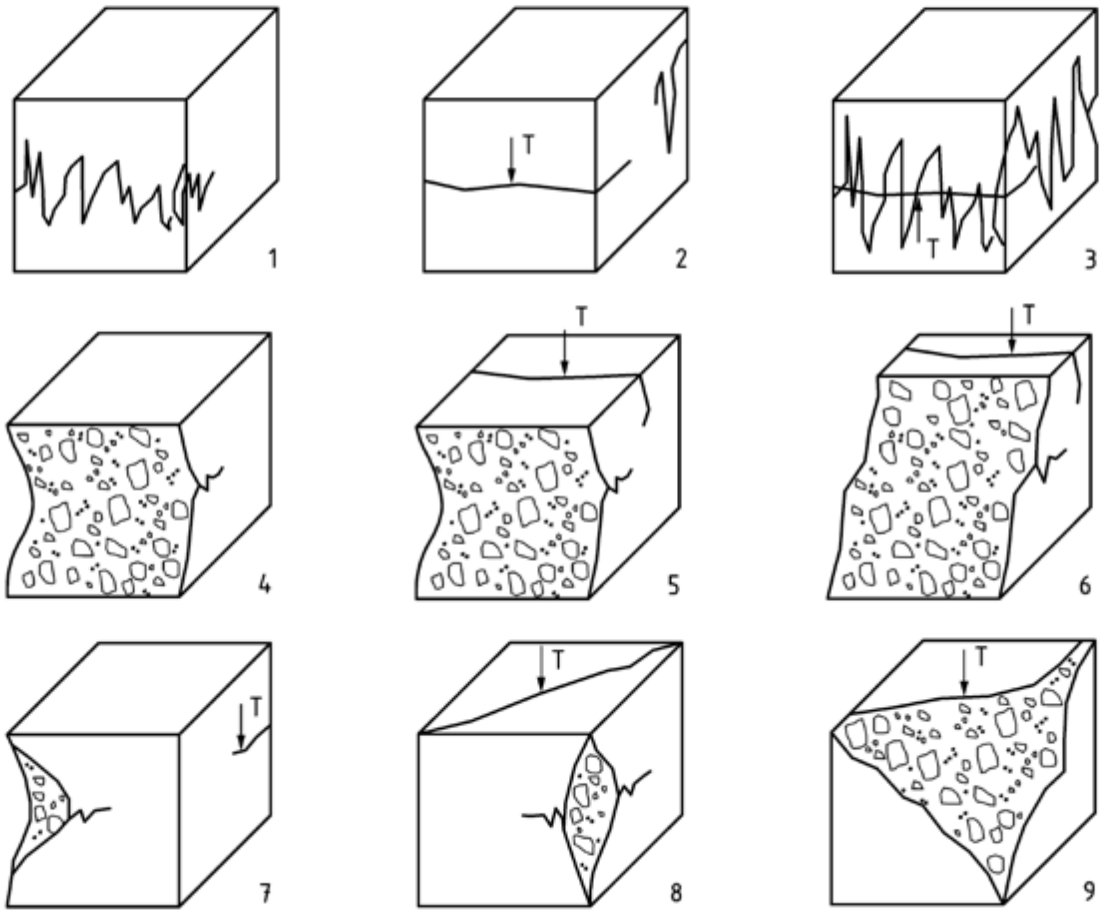



*Figure 2.26 – Unsatisfactory failure types, where T means a tensile crack.* (National Standards Authority of Ireland 2019c)

### 2.5.6 *Flexural tensile strength (at failure and residual)*

As previously discussed, the flexural tensile strength of plain concrete mixtures is typically low, and as a result, natural movements that occur during the lifetime of a structure can lead to the opening of cracks, including during the early age curing period. (Benaimeche et al. 2020)

Section 7.3 of the Eurocode 2 and the CIRIA C766 provide essential guidance on the control of crack openings in concrete structures. According to these guidelines, crack openings are expected to occur, especially during the early stages of concrete curing, as the heat generated by cement hydration reactions and the subsequent shrinkage of concrete can induce plastic shrinkage. Therefore, it is crucial to maintain the structure's saturation in water throughout the cement curing process to prevent or minimize the occurrence of cracks. (British Standards Institution 2004; Bamforth 2018)

Subsequently, when the structure is exposed to tensile, shear, bending, or torsion stresses resulting from direct load or due to restraints and other deformations, crack-opening is likely to occur during its lifespan. Although cracks with widths smaller than the estimated value might not pose a significant problem, their long-term existence can potentially affect the overall performance of the structure due to external factors.

As detailed in the upcoming chapter, the assessment of the flexural tensile strength (at failure and residual), fracture energy, and elastic modulus of FRC can be carried out following the standard 'Test Methods for Fibres in Concrete - Part 2: Effect on

Concrete.' This standard recommends applying the procedure outlined in "Test Method for Metallic Fibre Concrete - Measuring the Flexural Tensile Strength (Limit of Proportionality (LOP), Residual)" not only for mixtures reinforced with metallic fibres but also for other types of fibres. (National Standards Authority of Ireland 2007; NF EN 14651 2012)

The test is performed on beams specimens cast with 150 x150 x 550m, notched in the centre as shown in Figure 2.27.

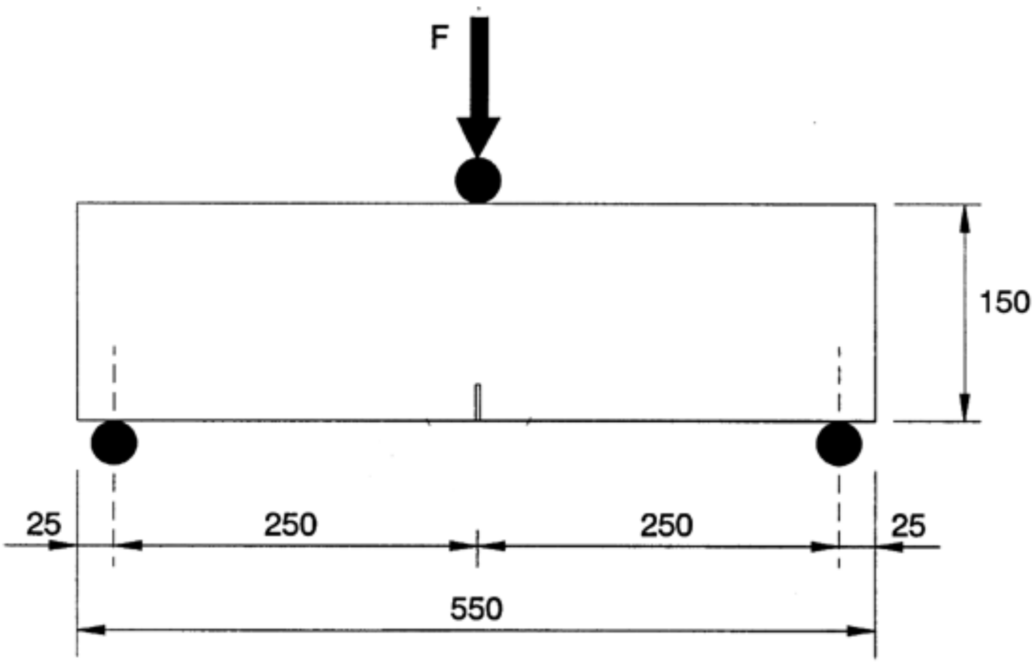



*Figure 2.27 – Beam arrangement according to EN 14651* (NF EN 14651 2012),

The standard guides the measurement of the stress in relation to the deflection (δ) or the crack mouth opening displacement (CMOD), establishing the relationship between CMOD and deflection as presented in Table 2.9. With this correlation, it is possible to compare diagrams with both defection and CMOD values.

*Table 2.9 – Correlation between CMOD and deflection* (NF EN 14651 2012).

| *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 |

From this, it is possible to measure the load capacity of the structure in different parts of the diagram obtained from the test as represented in Figure 2.28. Although the standard uses CMOD on the set-up parameters for the experiment, it allows a conversion to deflection depending on the equipment used.

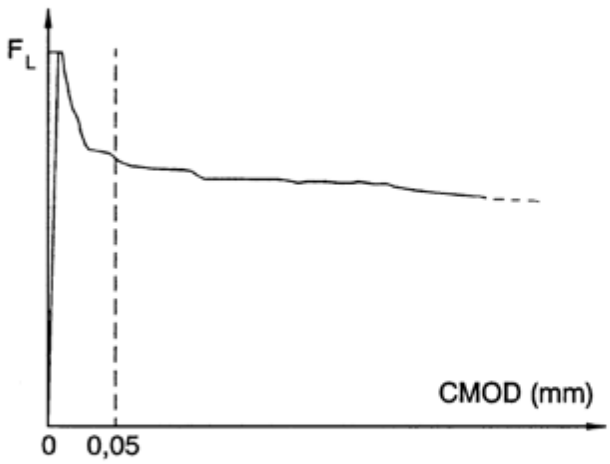



*Figure 2.28 – Example of Load-CMOD diagrams* (NF EN 14651 2012).

A study conducted on the crack control of canal-lining structures using FRC. The authors aimed to explore sustainable and environmentally friendly options for canal-lining structures. They evaluated the performance of jute fibre reinforced concrete (JFRC) in comparison with plain concrete (PC) and nylon fibre reinforced concrete (NFRC) and polypropylene fibre reinforced concrete by 5%. The authors examined the differences in their linear shrinkage, water absorption, and flexural strength. (Zia and Ali 2017)

According to the study, polypropylene fibre reinforced concrete (PPFRC) showed superior flexural load and increased deflection at rupture when compared to JFRC and NFRC, as shown in Figure 2.29. However, the residual tensile strength was found to be the same for all FRC after the deflection reached 1.7mm.

From this and other studies, it is possible to understand that although the fibres might not affect the peak flexural load, they have the potential to offer some post-crack strength in a residual analysis (Shi et al. 2020; Ikumi et al. 2021).

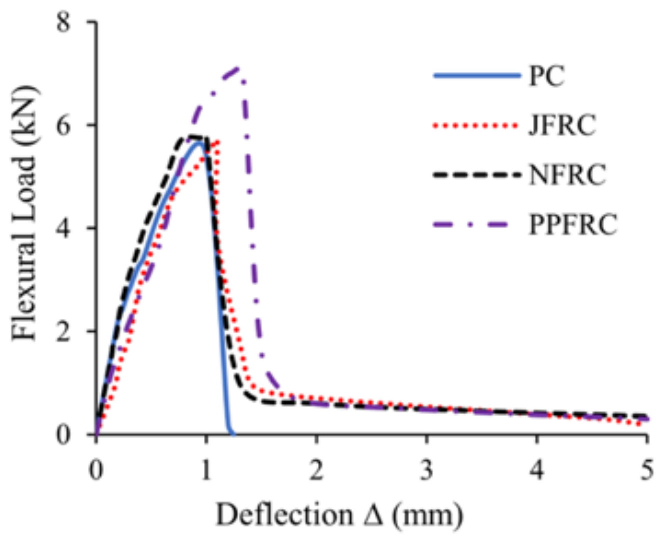



*Figure 2.29 – Load deflection curves from flexure strength tests* (Zia and Ali 2017)

### 2.5.7 *Fracture energy (Gf) and elastic modulus (E)*

After performing the tensile strength, it is possible to also obtain the fracture energy and the elastic modulus of concrete. The fracture energy (Gf) is the property used to measure the amount of energy required for the fracture of a material. To better understand how these properties are calculated, the measurements adopted on each equation are detailed in Figure 2.30 and the fracture energy (Gf) can be calculated through Equation 2.5 and the elastic modulus (E) through Equation 2.7.

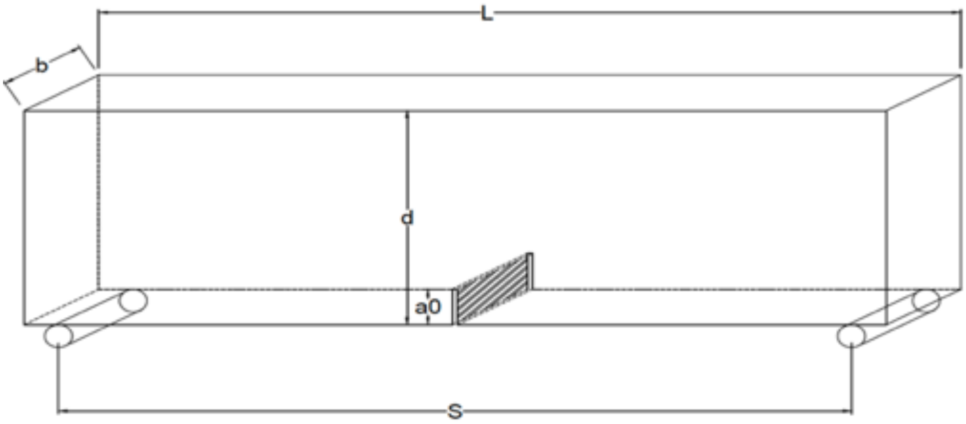



*Figure 2.30 – Details of notched beam specimen.*

$$G_f = \frac{W_0 + mg\delta}{A}$$ Equation 2.5

Where $W_0$ is the area below the curve on the Load [N] x 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 2.6.

$$A = b(d - a_0)$$ Equation 2.6

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

$$E = \frac{6Sa_0V_1(\alpha)}{Cib(d^2)}$$ Equation 2.7

Where $V_1$ and α can be obtained from Equation 2.8 and Equation 2.9

$$V_1(\alpha) = 0.76 - 2.38\alpha + 3.87\alpha^2 - 2.04\alpha^3 + \frac{0.66}{(1-\alpha)^2}$$ Equation 2.8

$$\alpha = \frac{a_0}{d}$$ Equation 2.9

### *2.5.8 Impact of the fibre length on the FRC behaviour*

Fibre length is not an inherent characteristic of natural fibres, as it can be modified to meet user specifications by adjusting the fibre length from the original plan size. For this reason, it was not included in the section on the properties of natural fibres. Nevertheless, fibre length can significantly impact the mechanical properties of concrete.

Regarding industrialised fibres, a broad range of lengths is currently available in the market. Although the relationship between fibre length and concrete reinforcement is not fully understood, a significant number of studies have suggested that short fibres (up to 25mm) may have a greater positive effect on compressive strength compared to long fibres (25mm to 60mm). Short fibres can either slightly improve or have a lesser negative effect on compressive strength, in contrast to a plain mixture or longer fibres.

Furthermore, research has shown that longer fibres may enhance tensile strength and elastic modulus. Longer fibres can also provide additional structural strength, thereby reducing crack opening due to early age plastic shrinkage. Moreover, longer fibres may offer residual tensile strength after failure and increased fracture energy. (Singh 2009; Caggiano et al. 2012; Yoo et al. 2016; Mastali et al. 2018a; Islam et al. 2022; MD and Unnikrishnan 2022)

Another property that can be influenced by fibre length is workability. Studies have suggested that shorter synthetic fibres may increase concrete slump. However, longer steel fibres tend to exhibit the same behaviour. (Caggiano et al. 2012; Han et al. 2019; Al-Baghdadi et al. 2021)

Therefore, the ideal length of fibres varies depending on the type of fibre, mix design and applicability for the concrete mixture and for additional residual tensile and fracture energy longer fibres are recommended.

### *2.5.9 Other properties*

Other properties that can also be relevant to be evaluated are sound insulation and abrasion, however, they will not be included in the experimental part of this research due to the insufficient availability of apparatus.

Similar to heat insulation, sound insulation is a property that affects users of a building during the lifetime of the structure. Studies have assessed the sound absorption coefficient reproducing the procedure from the standard “Acoustics — Determination of sound absorption coefficient and impedance in impedance tubes — Part 2: Transfer-function method”, the study indicates that the sound insulation presents a direct correlation with the density, as composites with lower densities have increased presence of air voids, which corroborates to what was already in the density section of this chapter. (ISO 10534-2 1998; Mastali et al. 2018b)

Whilst Fibre-Reinforced Concrete (FRC) may exhibit lower compressive strength than plain concrete, it can also demonstrate a reduced susceptibility to abrasion and impact damage on its surface. This response is expected since the inclusion of fibres in the concrete decreases its elastic modulus. (Siddique et al. 2012; Gupta and Kumar 2019; Alaskar et al. 2021)

The authors conducted a test to measure the roughness of the surface of various Fibre-Reinforced Concrete (FRC) mixes containing up to 1.25% polypropylene fibres under both dry and wet conditions. As depicted in Figure 2.31, the authors observed that the greater the quantity of fibres, the rougher the surface became, indicating that the inclusion of fibres enhanced the skid resistance of the concrete surface. (Alaskar et al. 2021)

The improved abrasion, roughness and reduced impact damage can be particularly interesting if the concrete will be used for pavement and floor applications. (Castoldi et al. 2019)

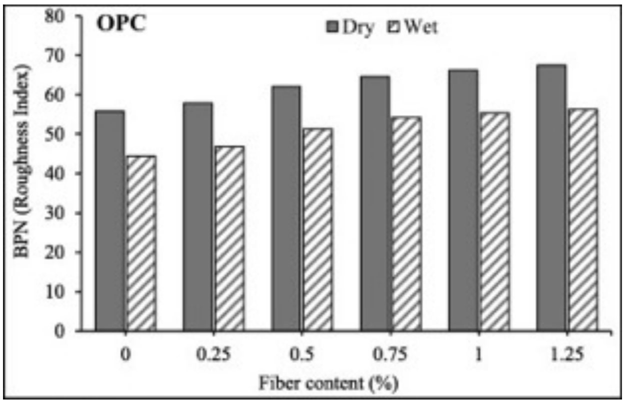


*Figure 2.31 – Correlation between roughness and fibre content.* (Alaskar et al. 2021)

The presence of both fibres in any dosage provided ductility to concrete, resulting in a deflection-softening behaviour. Similar flexural performances under monotonic and cyclic loads were presented for the composites tested, where the increase of fibres volume ratio led to a gain in concrete toughness. They evaluated how sisal fibres compare to polypropylene fibres and, as an outcome, observed that the level of residual strength provided by sisal fibres could the same as polypropylene fibres under equivalent dosages. However, it was also observed that sisal fibres reduced the flexural tensile strength of FRC, and the authors attribute this, to the fact that sisal fibres absorb higher amounts of water.

However, given the environmental drawbacks associated with commonly employed fibres, research has focused on identifying natural fibre sources as potential alternatives to conventional materials. (Castoldi et al. 2019; Shi et al. 2020)

## 2.6 Final Considerations

Although the topic has been studied for a few decades, there is still the need to develop a novel treatment to turn vegetable fibres into a suitable and safe alternative to replace fibres currently produced from non-renewable materials.

Studies using NaOH solution as surface treatment showed positive results in removing soluble and waxy compounds from the fibres wall that could reduce the fibre-matrix adhesion and the durability of the fibres on a composite. However, recently, research developing treatments using less hazardous chemicals for the environment and the human health started to increase.

To conclude this literature review, with a better understanding of the state-of-the-art of research related to vegetable fibres reinforced concrete, it was possible to plan the experimental part of this study, as detailed in the next chapter, aiming to contribute with the current gap on the literature of studies including the characterization of flax fibres previously and post surface treatments in comparison with hemp and synthetic fibres.

# Chapter Three

# Experimental Details

# Chapter 3: Experimental Details

To answer the research questions of this study, a schematic the flow of the strategy followed is presented in Figure 3.1 summarizing the methodology detailed in this chapter.

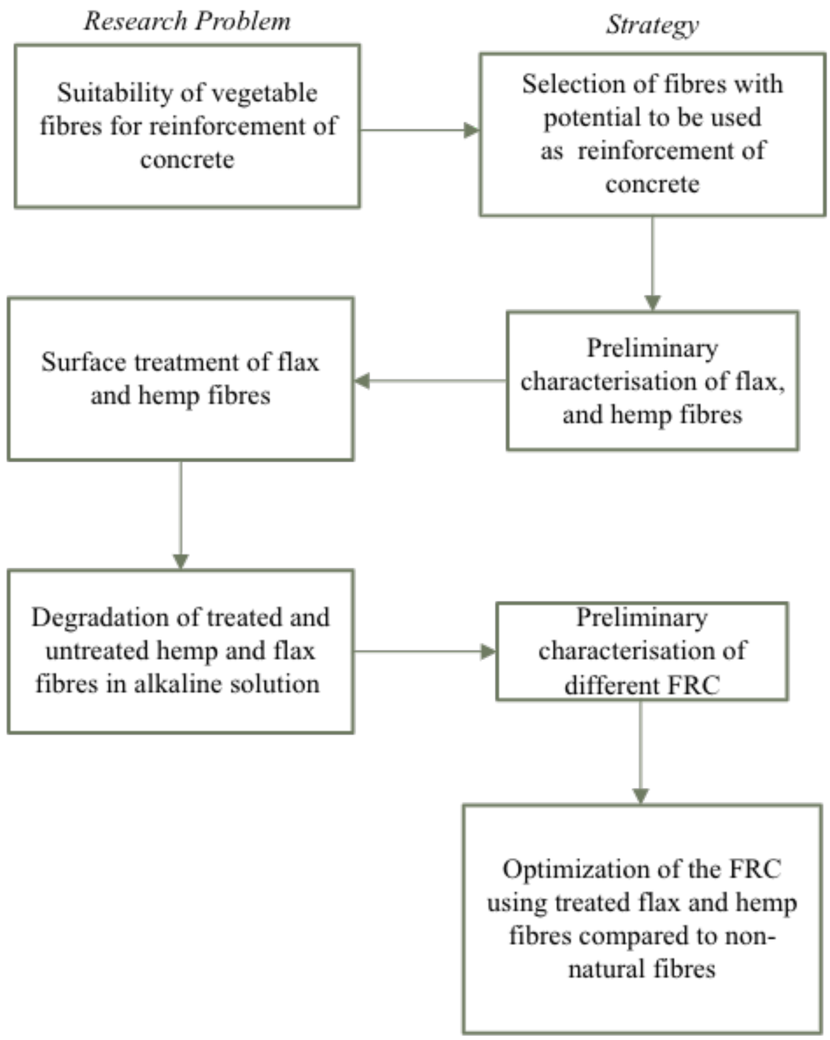



*Figure 3.1 - Schematic chart of results and discussion.*

Although vegetable fibres are natural and extracted from unlimited sources, they still require further research to become safely adopted for concrete reinforcement, as studied in the last chapter. Different from fibres produced through industrial processes, the variability of their properties and their short lifespan in an alkaline environment are currently the biggest impasses highlighted by past studies on the topic.

One of the approaches currently being pursued by researchers is the surface treatment of vegetable fibres to eliminate or coat compounds that are soluble or that could affect the adhesion between the fibres and the matrix (such as lignin, hemicellulose, and waxes). Given the need to reduce the negative impact of the building industry on the environment, this research aims to compare the effects of various chemical treatments and their hazards on the properties of fibres.

As a broad topic, this research has been divided into different stages, which are detailed in this chapter. First, a preliminary analysis was conducted on the properties of flax and hemp fibres. Then, different surface treatments were performed on the fibres, and their impact on the mechanical properties was evaluated. Thirdly, an assessment was made of the effects of an alkaline environment on the mechanical properties of the fibres.

After selecting the most suitable surface treatment for this study, a first batch of FRC was prepared and analysed using untreated flax and hemp, steel, polypropylene, and basalt fibres. Based on the results obtained the preliminary mix was optimized, aiming to achieve a mix suitable for flooring applications, precast panels, or 3D printers, as well as the percentage of flax fibres added to assess the suitability of the studied vegetable fibres for the reinforcement of concrete. In the next sections of this chapter, the experimental detail of this research will be explained complementing the experiments already seen in the literature review of this work.

## 3.1 Preliminary analysis of the mechanical properties of flax and hemp fibres

This section outlines the methodology employed to characterize the mechanical properties of flax and hemp fibres. Flax and hemp fibres have recently garnered significant attention as potential substitutes for conventional synthetic fibres in several industries such as textiles, composites, and construction. Hence, comprehending their mechanical properties is critical for the development of novel applications and reliable products.

Flax fibres were acquired in retted straw while hemp, as natural rope as showed in Figure 3.2. Both fibres were manually extracted and had their properties tested.

*(a)Flax*

*(b)Hemp*

*Figure 3.2 – Flax and hemp fibres as purchased.*

### *3.1.1 Density of fibres*

The initial examination carried out involved the determination of the densities of the fibres. Prior to the test, a specimen of each type of fibre was dried in an oven at 105ºC for 24 hours to remove any surface moisture content, as depicted in Figure 3.3a. The digital density balance rolbatch model RBDT-01, illustrated in Figure 3.3b, was used to measure the density of each sample. The readings were conducted five times for each type of fibre under investigation, and the obtained values were recorded. The mean value was adopted, and the outcomes are presented in the subsequent chapter. Benchmark samples of basalt and steel fibres were also evaluated.

*(a)*

*(b)*

*Figure 3.3 – (a) Sample of natural fibres after drying (b) Digital density balance.*

### *3.1.2 Tensile strength of singular fibres*

This procedure This study outlines the methods employed to determine the tensile strength properties of vegetable fibres, including the maximum force (Fmax), force at break (Fbreak), elongation at maximum force (dL at Fmax), elongation at break (dL at break), and diameter. The "Standard Test Method for Tensile Strength and Young`s Modulus of Fibre" was followed during the procedure, which is designed for testing individual fibres and can be utilized for fibres with diameters exceeding 250x$10^{-6}$ m. The mechanical properties were determined through the analysis of load vs. extension values and measured diameters, employing the algorithm outlined in Appendix A. (ASTM International 2020)

### 3.1.2.1 *Fibres extraction and preparation*

Prior to testing, the vegetable fibres were extracted and prepared in singular form, as per the standard procedure. Flax fibres were manually extracted from the straw, while hemp fibres were separated and both were manually separated into singular fibres, as per the recommended method.

Since there was no clear indication in the literature regarding the ideal measurement of vegetable fibres, a length of 40 ± 5mm was selected, based on common commercial lengths for long, non-natural fibres that are utilized to reinforce concrete mixtures for crack reduction, elastic modulus enhancement and residual tensile strength. Due to manual chopping of the fibres, a standard deviation of 5mm was observed in the sample analysis after preparation.

*(a) Flax* *(b) Hemp*

*Figure 3.4 Samples of (a)flax and (b)hemp fibres after extraction.*

### 3.1.2.2 *Tensile Strength and Young`s Modulus of Fibre*

A sample of 40 specimens was produced for both flax and hemp fibres. Tabs designed for the test were created as depicted in Figure 3.5a, printed on sulphite paper, and singular fibres were attached to leave the required length between the two fixed points using superglue. The central circle was manually cut, and each specimen was labelled with its corresponding number and fibre type.

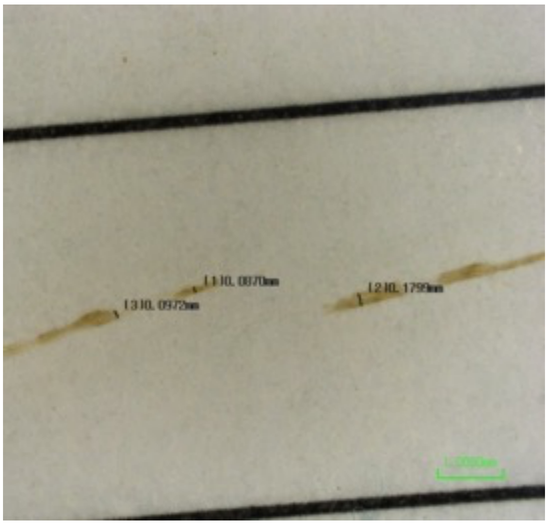
The tensile strength test was conducted using the Zwick/Roell Z010 equipment with grips, as shown in Figure 3.5b. The software was set to reflect the cross-sectional shape of the specimen as yarn, a pre-load of 0.1N, a pre-load speed of 50mm/min, and a test speed of 0.6mm/min with position control. The force shutdown threshold was set at 80% of Fmax. Each specimen of basalt, hemp, flax, and jute fibres was placed, and the paper was cut in the horizontal centreline of the tab, leaving only the singular fibre between the grips, as per the recommended standard. Any faulty specimens during test were not included in the study. Following the standard, after rupture, the diameter (d) of each fibre was measured. (ASTM International 2020)


*(a)* *(b)*

*Figure 3.5 – (a) Specimen tab and (b) Equipment used.*

### 3.1.2.3 Diameter

Following the ASTM C1557 guidelines (2014), each specimen of singular fibres had its diameter measured in at least two areas close to the fracture section using digital microscopy, as shown in Figure 3.6a and b. After individual measurements were recorded, the mean value was calculated to determine the cross-section using Equation 2.1. It should be noted that although the cross-sections of natural fibres are not perfectly circular, as is commonly assumed in the research community, circular cross-sections were considered for the purposes of calculation. The standard followed advises to take multiple measurements close to the fracture area to reduce the error in the results obtained.



*(a)*

*(b)*

*Figure 3.6 – Digital microscopy.*

### *3.1.3 Summary*

Based on the literature review, flax fibres were identified as the most promising vegetable fibres among the available options. As a novel addition to the existing research, hemp fibres were also included in the study as a comparative parameter. This allowed for a comparison of results between the two types of fibres. After this, the study moved on to the next section, which focused on analysing the effects of chemical surface treatment on the vegetable fibres.

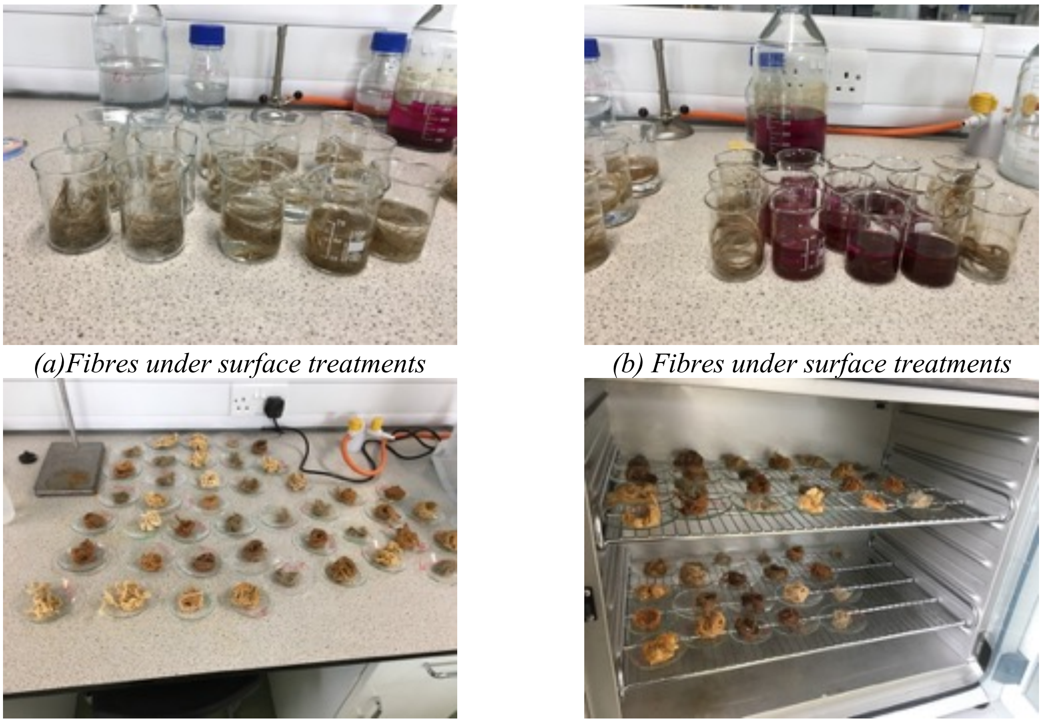
## 3.2 Surface treatment of flax and hemp fibres

From a list of surface treatments suggested by researchers for vegetable fibres, different types of solutes and solvents were selected to gain a broad view of their effects. Among all the options mentioned, the following solutions were chosen: an acid saline (EDTA), an acid oxide ($KMnO_4$), a fatty acid (Stearic Acid), and an alkali solution (NaOH) in different concentrations. Each solution was produced as presented in Table 3.1. To avoid damage to the internal layers of the fibre structure as pointed out in the literature, the samples of hemp and flax fibres were treated for 4 hours, 6 hours, 10 hours, 15 hours, and 24 hours. (Sood and Dwivedi 2018; Nayak et al. 2021)

*Table 3.1 – Treatment solutions suggested by M. Sood and G. Dwivedi (2018).*

| ***Treatment/Solute*** | ***Solvent*** | ***Concentration*** |
|---|---|---|
| Alkali (NaOH) | Water | 5%, 10%, 15% |
| Potassium Permanganate ($KMnO_4$) | Acetone | 0.05% |
| Stearic Acid ($C_{18}H_{36}O_2$) | Ethanol | 1.0% |
| EDTA ($C_{10}H_{16}N_2O_8$) | Water | 5g/L |

After preparing all the solutions, individual samples of the fibres were fully submerged into separate beakers (as shown in Figure 3.7a and Figure 3.7b). Once the specified time for each treatment was reached, the solutions were neutralized, washed (as shown in Figure 3.7c) and left to dry in an oven at 60°C (as shown in Figure 3.7d) for 24 hours. After drying, the fibres were separated, and new samples were made, containing 20 specimens of single fibres of 40mm length. These samples had their tensile strength and elastic modulus evaluated following the same procedure described in the previous item, based on the relevant standard. (ASTM International 2020)



(a)Fibres under surface treatments

(b) Fibres under surface treatments

(c) Fibres neutralized and ready to dry

(d) Fibres in the oven

*Figure 3.7- Surface treatments conducted on the fibres*

The results were analysed with a focus on treatments that could decrease the elastic modulus while increasing the tensile strength, as well as considering the associated chemical hazards. As discussed in the literature review, stearic acid, a natural fatty acid, was chosen for not having chemical hazards, while EDTA was selected for being less hazardous. NaOH and $KMnO_4$ were also included, as they are commonly studied by other researchers and allow for comparative analysis.

Based on the outcomes of all the treatments, the most effective surface treatments for the fibres to be used as concrete reinforcement were selected.

### *3.2.1 Chemical composition of the vegetable fibres*

Spectroscopy analysis is a common method for identifying the chemical composition of materials, and there are several methods available. According to researchers, X-Ray diffraction is one such test that can be used for vegetable fibres, as it measures the chemical bonds and functional groups present in the fibres. (Jones et al. 2017)

Initially, this experiment was carried out by an external laboratory. However, contrary to expectations, the X-Ray diffraction was inconclusive in characterising the chemical composition of the fibres. The position and size of each peak of the graphs could not be used to classify any chemical property, and therefore, X-Ray diffraction was not a suitable experiment for this purpose.

Alternatively, FTIR was successfully performed as outlined in the literature review. The test was carried out to evaluate the chemical composition of the fibres in their original form and to estimate the effects of each treatment on their composition. The FTIR test was conducted using a PerkinElmer Spectrum One FTIR Spectrometer, as shown in Figure 3.8. The obtained spectra were analysed according to Table 2.6 to evaluate the presence of different compounds based on the changes in the peaks in the obtained spectra. Results are also presented and discussed in the following chapter.

*Figure 3.8- FTIR Spectrometer*

### *3.2.2 Summary*

he experiments carried out in this section led to the selection of an innovative and optimised treatment for flax fibres using stearic acid, which improved the mechanical properties of the fibres and provided sustainability benefits. As hemp fibres were used for comparative purposes based on analyses conducted by other researchers, NaOH was chosen as the treatment.

After this analysis and before evaluating the effects of surface treatments on flax and hemp fibres in an FRC matrix, their degradation in an alkaline solution was evaluated.

## 3.3 Fibres behaviour under alkaline pH

In order to gain a better understanding of the behaviour of treated fibres within a concrete mixture, an experiment was conducted to evaluate the effects of an alkali environment on their tensile strength and elastic modulus. As no specific standard procedure for this test was found, the methodology described by the 'Standard Test

Method for Stability of Cellulose Fibers in Alkaline Environments' was adapted. Since the focus was on whole fibres rather than their pulp, 10g of whole fibres were used, to which 23.3g of 1N NaOH was added to obtain a consistency of 30%, totalling 33.3g.(ASTM International 2019)

A 1N NaOH solution is a water-based solution of sodium hydroxide with a concentration of 1 mole per litre, making it highly alkaline with a pH of 14. This solution is frequently employed in various industries to neutralise acidic solutions, clean equipment, and regulate the pH of solutions. The standard procedure stipulates the use of this concentration.

As the pH of concrete typically ranges from 12.5 to 13.8, this method was considered potentially applicable to assess the impact of alkaline pH on the properties of the fibres.

Samples containing untreated and treated flax and hemp fibres were prepared to enable a comparison of results between both forms. The samples were subjected to an alkaline environment for 3, 7, 14, 21, and 28 days to test their tensile strength. SEM images were produced to evaluate the physical appearance of the fibres after 28 days in the alkaline environment.

Fibres were submitted to the same procedure described in *3.1.2* to evaluate the impact on their tensile strength and elastic modulus.

### *3.3.1 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 the next chapter.

### *3.3.2 Summary*

The adaptation of the methodology described in this section provided insight into the behaviours of both treated and untreated flax and hemp fibres in an alkaline environment, such as concrete. These results can be used to estimate changes in fibre properties over time in fibre reinforced concrete.

## 3.4 Fibre-reinforced concrete

### *3.4.1 Preliminary mix design of the fibre reinforced concrete mixture*

The concrete mixture was designed following the ACI method with a slump range of 10-30mm, C30 at 7 days with 5% defective specimens. Following the standard method for a new mixture, a standard deviation of 8 was included, increasing the target mean strength to 43.12 N/mm2. Cement type II, 42 MPa at 28 days, fine aggregate grading of 52% uncrushed, and relative density of 2.6g/cm3 for crushed coarse aggregate were used.

It is well-established in the research community that adding long fibres in quantities greater than 1% per volume to a concrete mix reduces its mechanical properties. (Yoo et al. 2016; Al-Baghdadi et al. 2021; MD and Unnikrishnan 2022)

Therefore, mixtures containing 1.0% and 0.5% of volume of fibres were used in this stage. To equalize the proportion between steel fibres and the other types of fibres, the percentage of steel fibres was reduced by 10, following the ratio presented by Jamshaid and Mishra (2016). The mix design is detailed in Table 3.2. High hygroscopicity was observed in the mixture containing 0.5% of flax fibres due to an insufficient amount of cement paste to uniformly coat all fibres and aggregates. Therefore, a mixture containing 1.0% of flax fibres was not produced.

*Table 3.2 – Concrete mix designed for preliminary analysis.*

| ***Material*** | ***Quantities in kg per m³*** |
|---|---|
| Cement | 385 |
| Water | 170 |
| Fine aggregate | 585 |
| Coarse aggregate (10mm) | 415 |
| Coarse aggregate (25mm) | 830 |
| Basalt 0.5% | 13 |
| Basalt 1.0% | 26 |
| Flax 0.5% | 5.57 |
| Hemp 0.5% | 5.55 |
| Hemp 1.0% | 11.09 |
| Polypropylene 0.5% | 4.73 |
| Polypropylene 1.0% | 9.46 |
| Steel 0.05% | 3.95 |
| Steel 0.1% | 7.9 |
| Steel 0.15% | 11.85 |

| | |
|---|---|
| Steel 0.2% | 15.8 |

All mixtures were prepared using an electric concrete mixer, and their workability was measured immediately after preparation.

### *3.4.2 Concrete workability*

The slump test was carried out in accordance with the EN 12350-2 standard procedure for consistency measurement, and the results were recorded as shown in Figure 3.9. The test was performed by moistening the slump cone and base plate, and then removing any excess water with a damp cloth. Next, the apparatus was placed on a horizontal surface, and the concrete was poured in three layers, compacted with 25 strokes using a compacting rod to ensure uniform distribution over the surface. Once complete and levelled, the cone was lifted within 2 to 5 seconds and immediately turned upside down to measure the slump. The slump value was recorded for each mix and followed by specimen moulding. (National Standards Authority of Ireland 2019a)

*Figure 3.9 – Slump measurement.*

As will be discussed in the next chapter, the addition of vegetable fibres caused the slump to decrease to zero, and therefore, it was decided to incorporate plasticizer into the optimized mixture design produced in the final part of this experimental study.

### *3.4.3 Specimens moulding and curing*

For this preliminary analysis, only compressive and flexural tensile strength tests were conducted. As the nominal maximum size of the aggregate was 25mm, three cubes measuring 150x150x150mm and three beams measuring 150x150x550mm were cast, following the relevant standard procedures. (European Committee for Standardization 2005; National Standards Authority of Ireland 2012; National Standards Authority of Ireland 2019b)

After 24 hours, the specimens were unmoulded and cured in a water tank at a temperature of 21°C with a variability of ±1°C. One day prior to testing, notches were made at the centre of the beams, as previously shown in Figure 2.27.

*Figure 3.10 Concrete curing tank set at (21± 1)ºC.*

### 3.4.4 Relative density

Each cube had its sides measured using a Vernier calliper, and its relative density was obtained using a specific gravity balance (Figure 3.11) prior to the compressive strength test. Following the equipment manual, the test was initiated by zeroing the scale with the hanging basket, inserting the specimen onto it, and measuring its dry weight. Subsequently, the container with water was carefully lifted until the water level completely covered the concrete cube, and the wet weight was recorded. The specific gravity was then determined and recorded as the density value.

*Figure 3.11 – Specific gravity scale.*

### 3.4.5 Compressive strength

After evaluation of the specific gravity, each cube was tested for compressive strength at 7 days following the parameters given by the British Standard EN 12390-3:2009.

The ELE International ADR 2000kN BS/EN Compression machine was used (Figure 3.12).

*Figure 3.12 Equipment used to determine the compressive strength of specimens..*

Following the concrete failure, broken fragments of the cube were manually extracted, and a post-cracking analysis was performed to assess whether the failure was satisfactory, according to Figure 2.25. The analysis involved verifying that the centre of the cubes had maintained its strength and that there was good adhesion between the cement paste and the aggregates.

### 3.4.6 Three-point bending test

As previously mentioned, a 3-point-bending test was carried out on each sample following NF EN 14651:2005. After 5 days of being cured, each beam had a 2.5cm notch swan and at the age of 7 days according to the sketch previously presented in Figure 2.30, the tests were led using a tensile strength machine INSTRON Model 300DX K8043, as pictured in Figure 3.13.

*Figure 3.13 – Three-point-bending test.*

From the 3-point bending test, it was possible to calculate the flexural tensile strength at peak load, the residual flexural tensile strength, fracture energy and elastic modulus.

### 3.4.6.1 Flexural tensile strength ($F_{R,j}$), ($F_n$) [MPa]

Following EN14651, the limit of proportionality (LOP) was calculated from Equation 3.1. 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 corresponding load [N] at specific deflection $\delta_j$(N) points are used to calculate the residual flexural tensile strength ($F_{R,j}$) following Equation 3.2.

$$F_n = LOP = \frac{3F_L S}{2b(d-a_0)^2} \qquad \text{Equation 3.1}$$

$$F_{R,j} = \frac{3F_j S}{2b(d-a_0)^2} \qquad \text{Equation 3.2}$$

Where S is the span, b is the with and measurements from Figure 2.31. For the peak strength (Fn), FL is the value of the peak load [N], and for the residual strength (FR,j) at net deflections, Fj is the corresponding load [N] at specific j deflection points.

Values of FL and Fj were extracted from the load vs deflection points obtained from the 3-point bending tests, where a0, b, and d were measured using a Vernier calliper for each specimen. The flexural strength at the peak load and the residual strength were calculated for net deflections j, from Table 2.9.

The residual strength was calculated in accordance with NF EN 14651, and the load vs deflection diagrams were used, instead of CMOD, as per the parameters established in the standard and the procedure detailed in the literature review.

#### *3.4.6.2 Fracture energy and elastic modulus*

As detailed in the last chapter, fracture energy is the property used to measure the amount of energy required for the fracture of a material. Mathematically it is obtained from the area below the curve of the load-deflection diagram and can be calculated by Equation 2.5.

The elastic modulus is the capacity of a material to modify its shape in a non-permanent form obtained through Equation 2.7. Both parameters were calculated using an algorithm developed in MATLAB presented in Appendix B and C and the results are presented in the next chapter.

After conducting the preliminary mix design test, several areas for improvement were identified. Firstly, the characteristic strength of C32 at 7 days was found to be higher than required for any of the intended applications. As such, it was deemed more appropriate to use a C32 mix at 28 days. Secondly, the initial slump of 10-30mm was deemed insufficient to provide adequate workability for the fibre reinforced concrete. To address this, it was decided to increase the slump to 30-60mm and incorporate a plasticizer additive. As noted in the literature review, reducing the maximum aggregate size can also improve the workability of concrete mixtures. Furthermore, it was discovered that using a maximum aggregate size of 25mm would limit the thickness of the produced structures, rendering them unsuitable for certain applications. To address this, a new mix was developed, limiting the coarse aggregate size to 10mm.

As the amount of flax fibres proved to higher than the ideal for the proposed purpose, the percentage of flax fibres should also be reduced during the optimization. Additionally, it was found that it was not possible to compare the results of steel fibre reinforced mixes to those of other fibres due to their differing effects on the concrete. Consequently, steel fibres were excluded from the optimized concrete mixture designed, as detailed in the following chapter.

### *3.4.7 Optimized FRC mix design*

The mixture studied in this research represents an improvement on the previously adopted mixture, and its composition is detailed in Table 3.2. The primary goals of the optimization were to enhance workability by increasing the water/cement (w/c) ratio, reducing the maximum aggregate size, and optimizing the percentage of flax fibres to be included. The optimization was accomplished using the ACI method, and the target mean compressive strength remained at 32N/mm² at 28 days, with a maximum coarse aggregate size of 10mm. By selecting a smaller size for the coarse aggregate, the mixture could have different applications based on its behaviour, including the production of masonry blocks, thinner precast structures, and 3D printed structures. Quantities are presented in Table 3.3 and the water/cement ratio was decreased from 0.8 to 0.5 to avoid shrinkage and cracking, as noted by other researchers. (Piasta and Zarzycki 2017)

*Table 3.3 – Concrete mix designed for preliminary analysis.*

| ***Material*** | ***Quantities in kg per m³*** |
|---|---|
| Cement | 460 |
| Water | 230 |
| Fine aggregate | 760 |
| Coarse aggregate (10mm) | 930 |
| Coarse aggregate (25mm) | - |
| Basalt 0.5% | 13 |
| Flax 0.5% | 5.57 |
| Flax 0.35% | 3.89 |
| Flax 0.25% | 2.785 |
| Hemp 0.5% | 5.55 |
| Polypropylene 0.5% | 4.73 |
| Polypropylene 0.25% | 2.37 |

The slump of the designed mixture was increased to 30-60mm to account for the expected moisture absorption by the fibres, and plasticizer additive was added following the manufacturer's instructions. After discovering that the optimized mixture was still overdosed with 0.5% flax fibres, the percentage was reduced until the compressive strength values were comparable to control mixtures without fibres, with a focus on obtaining a homogeneous, workable, and mouldable mixture with less impact on compressive strength. (Netinger Grubeša et al. 2018)

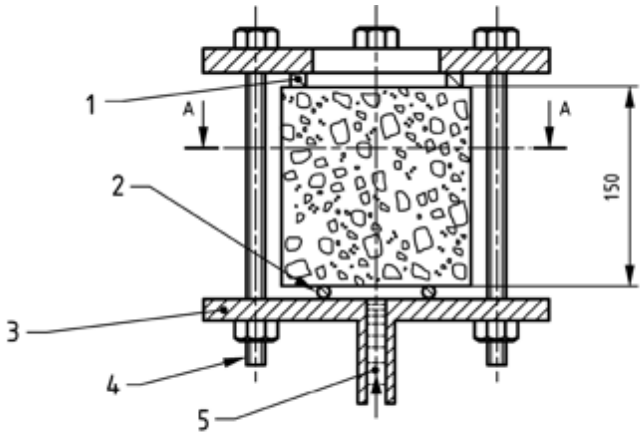
Trial samples were made with 0.5%, 0.35%, and 0.25% flax fibre content, and the mix with 0.25% presented a satisfactory slump. This mix was repeated to verify its compressive strength at 7 and 28 days. Mixes containing polypropylene and basalt fibres were included as benchmarks, but the analysis of the flexural tensile strength of the polypropylene fibre reinforced concrete could not be evaluated due to the unavailability of the same fibres from the provider and, only results from the preliminary study were considered in the analysis.

### *3.4.8 Mechanical properties of FRC*

The mechanical properties of the optimized FRC mixtures were tested using the same procedure as described earlier in this chapter. However, since the maximum aggregate size was reduced, cubes measuring 10x10x10cm were used, following the I.S. EN 12390-2. The samples were tested at 7 and 28 days to enable a comparison with the previous mixes tested and to evaluate the efficiency of the optimized mix. The results will be presented and discussed in the next chapter.

### 3.4.9 Depth of penetration of water under pressure

To measure water penetration depth, 3 cubes measuring 150x150x150mm were moulded and cured for 28 days according to the relevant standard procedure, the depth of penetration of water under pressure was tested after 28 days, following the standard I. S. E. 12390-8:2009 - Testing hardened concrete - Part 8: Depth of penetration of water under pressure. For the experiment, as shown in Figure 3.14 water pressurised in 500±5 kPa for 72 ± 2 hours is applied by the equipment in the bottom of the cube. Where (1) refers to the packing piece, (2) to the sealing ring, (3) and (6) screwed on plates, (4) screw-threaded rod, and (5) water under pressure. (National Standards Authority of Ireland 2019b; National Standards Authority of Ireland 2019d)



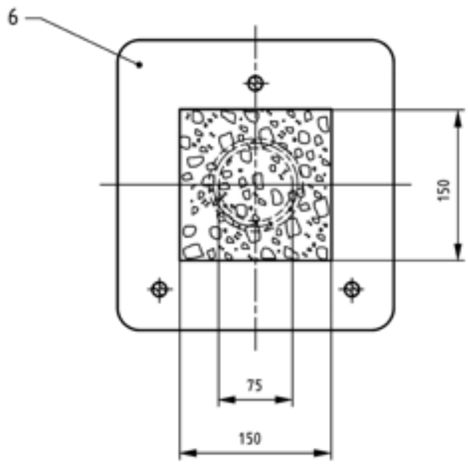
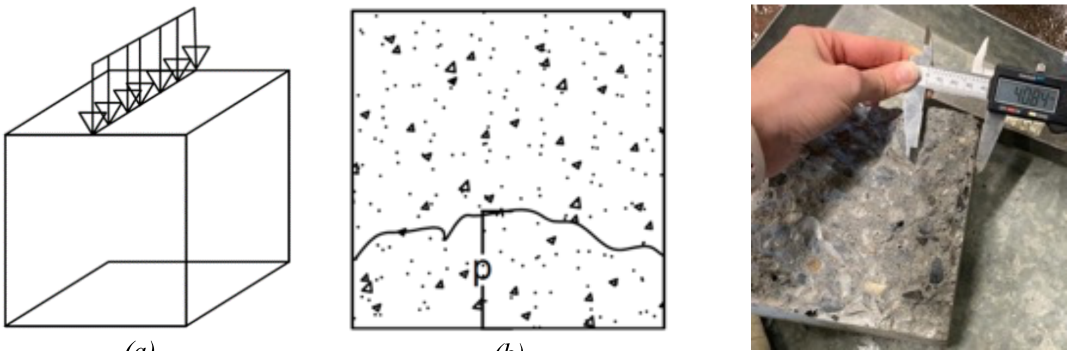




*Figure 3.14 – Test arrangement according to EN 12390-8: 2019.* (National Standards Authority of Ireland 2019d)

After 72 hours, the cubes removed from the equipment (Figure 3.15) and were split in two, using a compressive one-point load applied on the centre of each cube as shown on the sketch from Figure 3.16a. After having them divided, the water penetration depth (p) was measured using a vernier calliper Figure 3.16b and c.

*Figure 3.15 – Test apparatus.*




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

*Figure 3.16 – (a)Load distribution model used to split the cubes, (b) and (c)Highest line of water penetration*

### *3.4.10 Thermal conductivity*

In order to evaluate and compare the effect of fibre reinforcement on heat transfer, slabs measuring 50x300x300mm were analysed for each mixture. The ISO 8301 (1991) and the "Thermal Conductivity of Building Materials" manual were used to guide the procedure. The steady box method was employed to test each slab for a minimum of 3 hours. The equipment recorded readings every minute, and the thermal resistance was determined based on the reference standard. (The International Standards Organisation 1991; P.A. Hilton Ltd 2008)

The equipment used for the steady box method as presented in Figure 3.17. The box consists of a hot plate on top and a cold plate on the bottom, whose temperatures are regulated by an electric heater and water, respectively. The thermal conductivity of each specimen, which represents the heat transfer between its surfaces, was measured by placing it between the plates and increasing the temperature on one side. The temperature difference was then recorded every minute by the equipment on the opposite plate. Using the thickness ($l_s$)[ [m] of each specimen, the thermal conductivity (λ) [W/m² K] was calculated using Equation 3.3. (Bala and Gupta 2021)

*Figure 3.17 –Equipment used to measure thermal conductivity.*

$$\lambda = \text{fe}\frac{l_s}{\Delta T} \qquad \text{Equation 3.3}$$

Where f was the equipment calibration factor and e the heat flow meter output [mV/m²]. Results are presented in the next chapter.

### 3.4.11 SEM Images

To visually analyse the changes on the adhesion between the fibre-concrete matrix, SEM images were produced on concrete samples at 28 days and after over 90 days. Images and discussion are provided in the next chapter.

### 3.4.12 Summary

An experimental study was conducted to assess the water penetration, thermal conductivity, and mechanical properties of an innovative flax fibre reinforced concrete treated with stearic acid solution. The optimized mix was designed from a range of concrete mixtures and compared to hemp fibres treated with NaOH, which are commonly found in the literature. Results were also compared to those of basalt and polypropylene fibres, thereby completing the experimental study.

### 3.4.13 Final Considerations

Considering the unsatisfactory performance of the first designed concrete mixture with vegetable fibre reinforcement, an optimization was carried out to increase workability and reduce the maximum nominal size of the coarse aggregate. This modification offers an additional advantage of enabling the use of the concrete in various applications, such as 3D printing or casting into thinner products such as blocks, panels, and levelling layers for flooring.

The mechanical properties of both plain mixtures achieved similar compressive strength, as discussed in the next chapter, while the vegetable fibre reinforced concrete demonstrated improved workability as intended. The evaluation of thermal conductivity and water absorption facilitated a comprehensive analysis of mixture properties for building applications. Unfortunately, measuring sound insulation and abrasion of samples was not feasible. However, as demonstrated in the previous chapter, sound and heat insulation are proportionate properties of materials. Hence, with increased thermal insulation, the acoustic properties are also expected to be enhanced.

The following chapter presents and discusses the results obtained from the tests outlined herein.

# Chapter Four

# Results and Discussion

# Chapter 4: Results and Discussion

Vegetable fibres have been widely studied to be used as an ecological replacement for non-natural fibres on composites reinforcement. Their applications englobe, among many, the automotive, polymer and construction industries and can be classified according to their source of extraction, usually, as animal, mineral and vegetable. (Amir et al. 2017; Degrave-Lemeurs et al. 2018; Barth and Carus 2019)

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 to low cost of production and higher biodegradability. (Zah et al. 2007; Huang and Young 2019)

On the other hand, their biodegradability can also be considered an issue when it comes to the application of vegetable fibres in composites produced to offer longer lifespan such as concrete. (Saha et al. 2012; Manna et al. 2013)

As discussed in the Literature Review, flax fibres were selected for this study based on their superior mechanical properties evaluated by past research and the lack of studies on their use in concrete reinforcement. For comparison, hemp fibres were also included as they have been extensively studied for similar applications and potentially offer higher values of tensile strength, compared to other fibres studied for composite reinforcement. As benchmarks, basalt, polypropylene and steel fibres were also studied.

As seen, the main issues reported by other authors in relation to the use of vegetable fibres are the variability on their properties and the degradation of their mechanical properties when under an alkaline ph.

Aiming the production of a novel fibre reinforced concrete using flax fibres and a contribution to the scientific community to overcome the listed challenges, the results obtained in experiments described in the previous chapter are now presented and discussed.

## 4.1 Preliminary analysis of the properties of flax and hemp fibres

For a comparative analysis, Table 4.1 summarises values previously presented in Table 2.2, Table 2.3 and Table 2.4.

*Table 4.1 – Physical and mechanical properties of fibres according to the literature review.*

| ***Type of fibre*** | ***Density g/cm³*** | ***Diameter (µm)*** | ***Tensile Strength (N/mm²)*** | ***Young`s Modulus (GPa)*** | ***Elongation at Break %*** |
|---|---|---|---|---|---|
| Basalt | 2.6 | 7-15 | 4150-4840 | >40 | 3.2 |
| Flax | 1.28-1460 | 10-80 | 345-2000 | 27.6-80 | 1.2-4.0 |
| Hemp | 0.86-1.48 | 4-800 | 270-900 | 23-90 | 1.0-4.0 |

The average of diameters and densities measured is presented below in Table 4.2 and, as expected, it was observed high variability in the diameters and mechanical properties. As a benchmark, the density of polypropylene and steel were also measured under the same methodology.

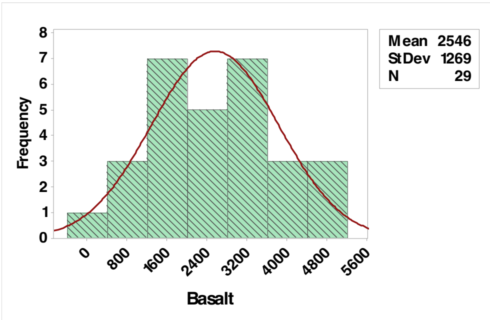
In comparing Table 4.1 and Table 4.2, the experimental values obtained are consistent with the ranges reported by other researchers, with the exception of the densities of basalt fibres. The densities obtained in this study were found to be 16.4% lower than those reported in the literature. This difference may be due to variations in the types of fibres used in this study and those considered by previous researchers.

*Table 4.2 – Physical properties of fibres.*

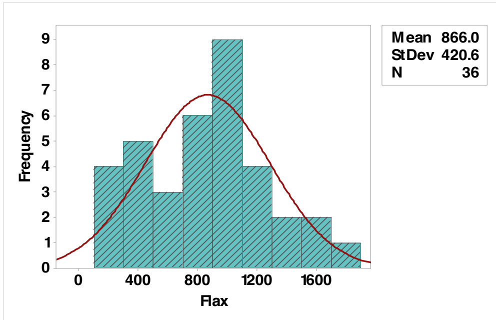
| ***Material*** | ***Density [g/cm³]*** | ***Diameter [µm]*** | ***Tensile strength [MPa]*** | ***Young`s Modulus (GPa)*** | ***Elongation at Break %*** |
|---|---|---|---|---|---|
| Flax | 1.114 | 82.4 | 865.96 | 40.78 | 2.09% |
| Hemp | 1.109 | 73.0 | 262.68 | 22.44 | 1.47% |
| Basalt | 2.172 | 14.0 | 2546 | 136.18 | 1.67% |
| Polypropylene | 0.953 | - | - | - | - |
| Steel | 7.922 | - | - | - | - |

As there were no recommendations in the standard procedures for determining the initial sample size (n) for natural fibres, it was found in the literature studies adopting samples containing 4 to 8 specimens. To obtain values with lower standard deviation a sample size of n=40 was initially defined for this study. This sample size was selected in order to obtain at least 30 valid specimens for each sample, considering the fragility of the specimens and the potential for damage during the testing procedure and it was reduced later after a descriptive analysis. (Bandyopadhyay and Cherry 2011)

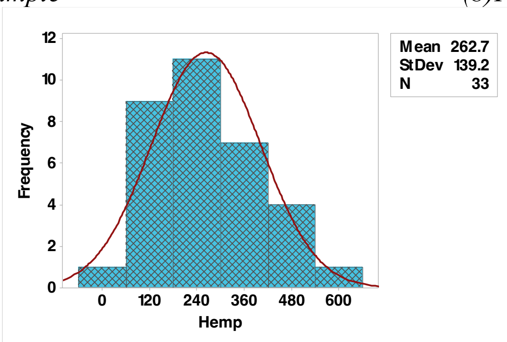
The descriptive analysis is a statistical study conducted to evaluate the tensile strength of each sample and determine the confidence interval of the distribution. This analysis was carried out because the tensile strength property incorporates the load and diameter variability. Figure 4.1 shows the Gaussian distribution obtained for each sample. It was observed that the confidence interval for basalt was 69%, 67% for flax, 64% for hemp, and 70% for jute. This indicates that the data falls within one standard deviation in a normal distribution, which is 68%. These results confirm the ranges reported by other authors for similar materials.  (Bandyopadhyay and Cherry 2011)




*(a)Basalt sample*




*(b)Flax sample*




*(c)Hemp sample*

*Figure 4.1 – Histogram and normal distribution of samples tested.*

Appendix D presents a complete descriptive analysis performed on each sample to better understand the variability of the properties shown in  Figure 4.1. Confirming the unpredictability of fibre properties pointed out in the Literature Review, all the samples presented distributions with high values of standard deviation and sample variance, indicating that the values are strongly spread out from the mean. (Swamy 1990)

Among the vegetable fibres studied, flax fibres achieved, on average, a significantly higher tensile strength of 866 MPa compared to hemp fibres, which reached 262.7 MPa, confirming their potential for composite reinforcement.

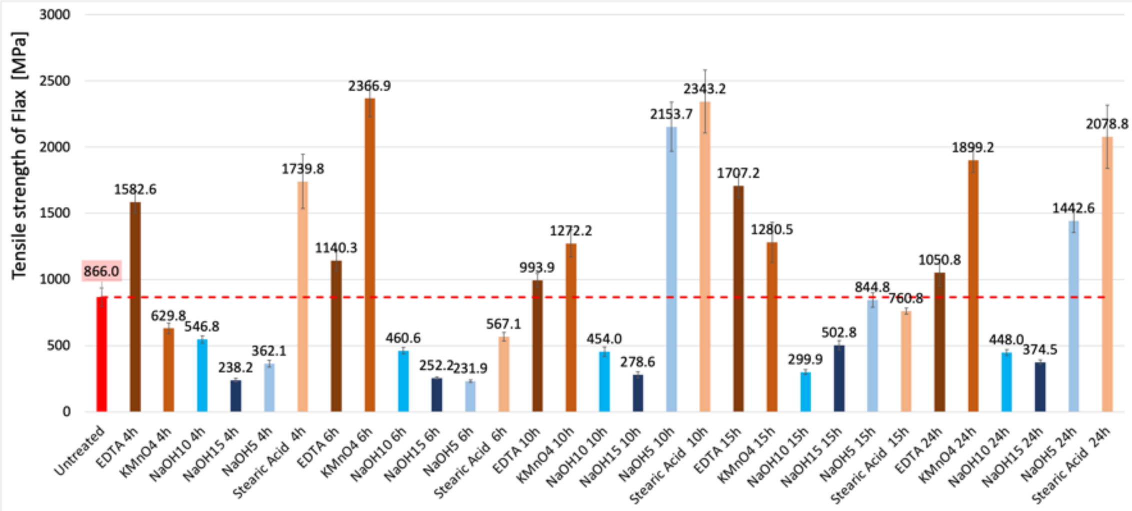
Although authors usually adopt samples containing 4 to 8 specimens for this purpose, the possibility of reducing the sample size from 30 to 10 specimens was evaluated through another descriptive analysis done for the first 10 specimens of samples of flax and hemp fibres. Values are also included in Appendix D. As presented in Table D.4 and Table D.5, the mean values still fit into the range obtained by other authors; however, there was a smaller standard deviation for all the parameters except for the elastic modulus and elongation at break of flax fibres, which showed a 19% and 22% increase, respectively.

Considering these values, it was decided to reduce the sample size to a minimum of 10 specimens during the analysis of the effects of surface treatment on the properties of singular fibres.

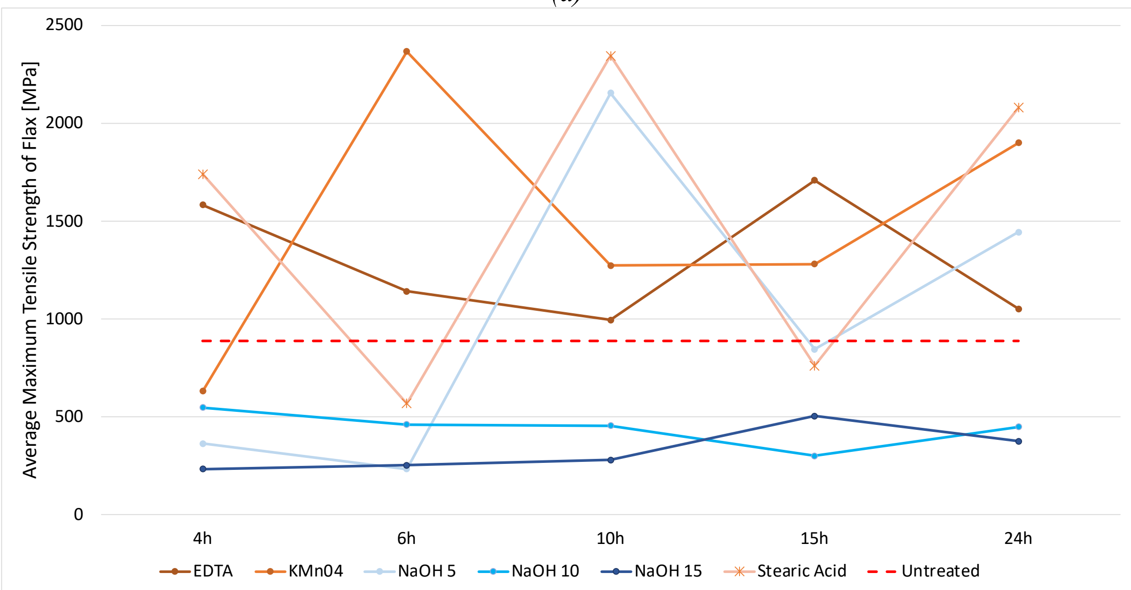
## 4.2 Surface treatment of flax and hemp fibres

As detailed in the previous chapter, the surface treatments conducted on flax and hemp fibres were selected according to the literature review. The mechanical properties of the treated samples were evaluated using the same methodology described in item. 3.1.2 (Sood and Dwivedi 2018)

Ahead, results obtained for treatments carried out on the flax fibres are summarised in Figure 4.2 and Figure 4.3 and on hemp fibres in Figure 4.4 and Figure 4.5. In each figure, the bar graph (a) includes error bars and numerical values for a global overview, while the line graph (b) shows the changes in properties for each treatment over time. In both graphs, the dashed line parallel to the x-axis represents the values obtained for untreated fibres from Table 4.2.



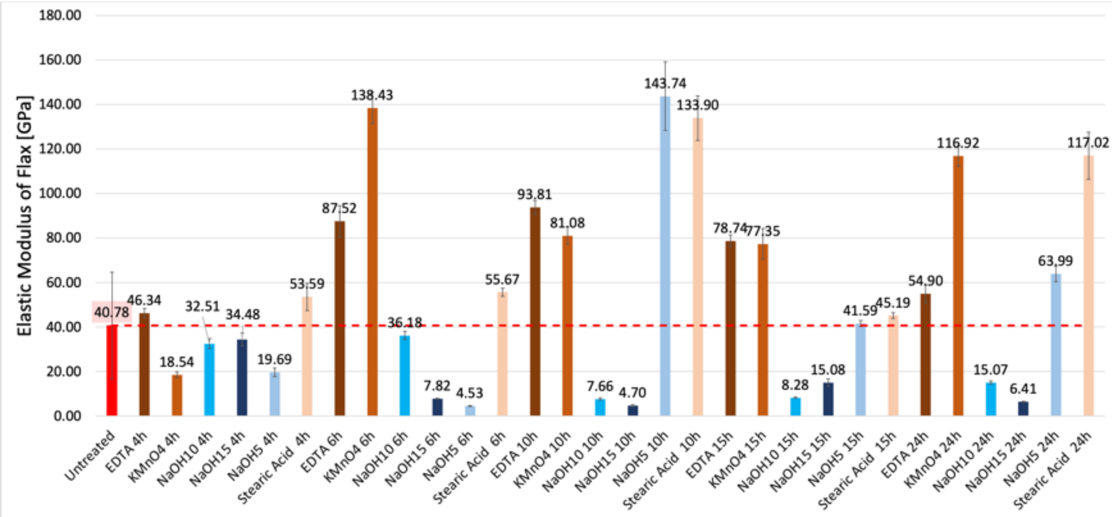

*(a)*



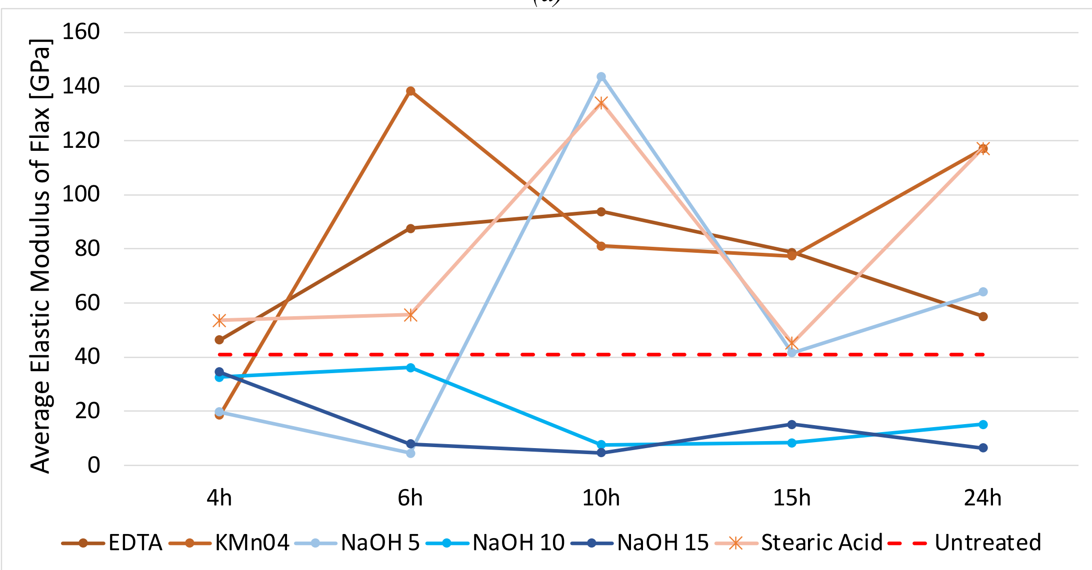

*(b)*

*Figure 4.2 –Tensile strength obtained for each treatment on flax fibres.*

Starting this analysis with flax fibres, graphs (a) and (b) in Figure 4.2 show the tensile strength and graphs in Figure 4.3, the elastic modulus obtained for each sample tested. Treatments with EDTA (4h), stearic acid (4h), EDTA (6h), KMnO4 (6h), EDTA (10h), KMnO4 (10h), NaOH 5% (10h), EDTA (15h), KMnO4 (15h), EDTA (24h), KMnO4 (24h), NaOH 5% (24h), and stearic acid (24h) increased both tensile strength and elastic modulus of the fibres.




*(a)*




*(b)*

*Figure 4.3 – Elastic modulus obtained for each treatment on flax fibres.*

Also, looking at graphs b, results indicate that treatments using NaOH in concentrations of 10% and 15% for the entire timeframe studied reduced the two properties for flax fibres, while a solution of 5% of NaOH increased them after 10 and 24 hours. Among the other chemicals, EDTA for the entire timeframe evaluated increased this property. After 4 hours, $KMnO_4$ also improved the tensile strength, and results for stearic acid alternated between increasing and reducing the property.

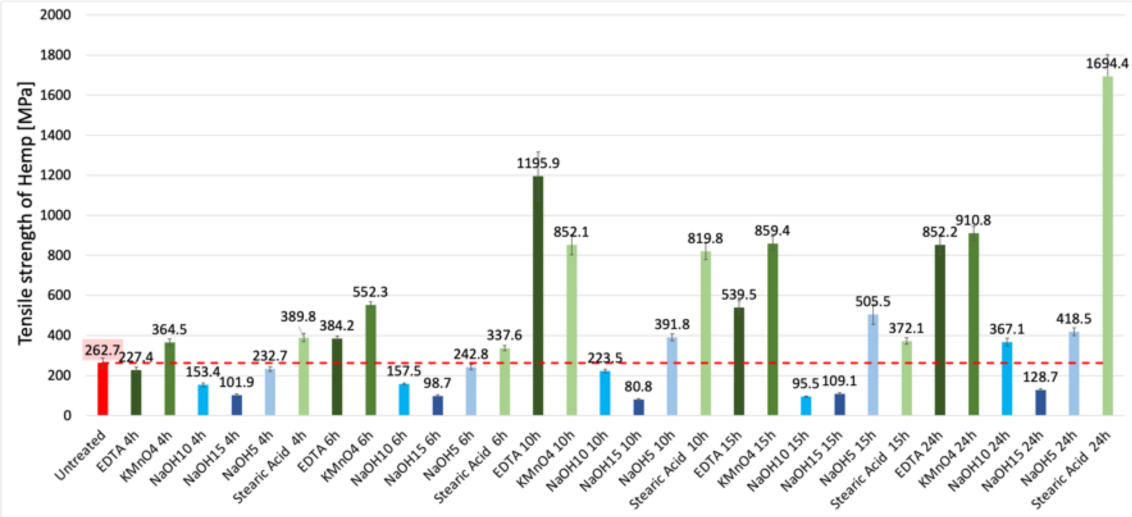
As the ideal treatment should present higher tensile strength and reduced elastic modulus, when comparing the two properties, none of the treatments achieved the desired outcome of reducing the elastic modulus while increasing the tensile strength. Still, the two treatments that enhanced tensile strength with a lower increase in the elastic modulus were the treatments with EDTA (4h) and stearic acid (4h).

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

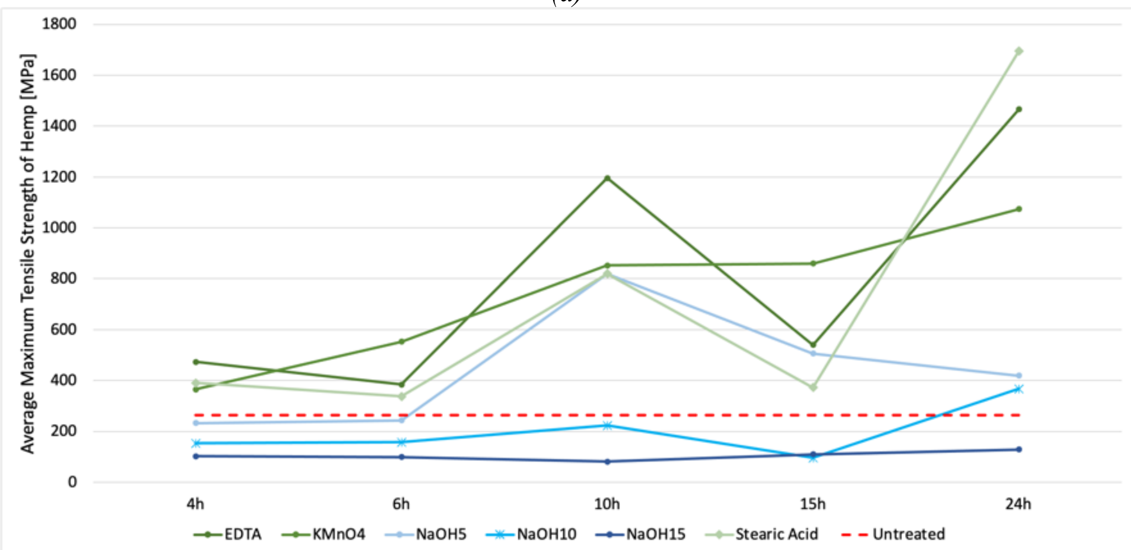
Regarding their hazardousness, going back to Table 2.8, EDTA presents an exclamation mark and health hazard causing possible respiratory irritation, while stearic acid presents none, however the second requires ethanol as a solvent flammable substance with, also, an exclamation mark like acetone. In terms of variability, both treatments presented reduced standard error, in comparison to the sample of untreated fibres.

A research investigation conducted using linseed oil demonstrated that fatty treatment provided a coating that shielded the fibres. Additionally, stearic acid (4 hours), as a type of fatty-acid chemical, was also considered for its potential positive impact on the intended application. (Page et al. 2021)

In a similar analysis performed on hemp fibres, all surface treatments, with the exception of NaOH 10% for 24 hours, resulted in an increase in both tensile strength and elastic modulus, as indicated by Figure 4.4 and Figure 4.5. The treatment using NaOH 10% for 24 hours, while showing only minor changes, caused an increase in tensile strength and a reduction in elastic modulus of the sample.



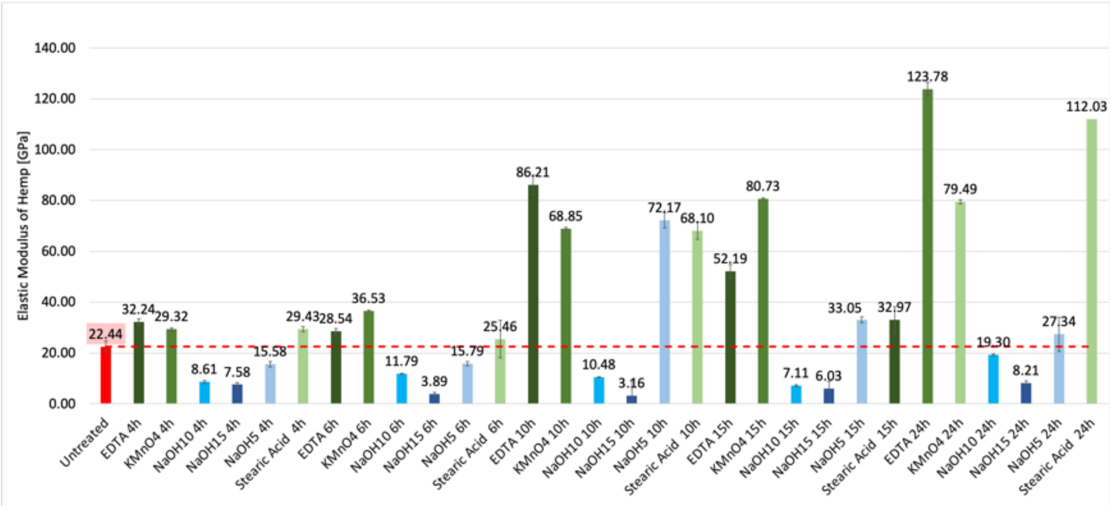

*(a)*



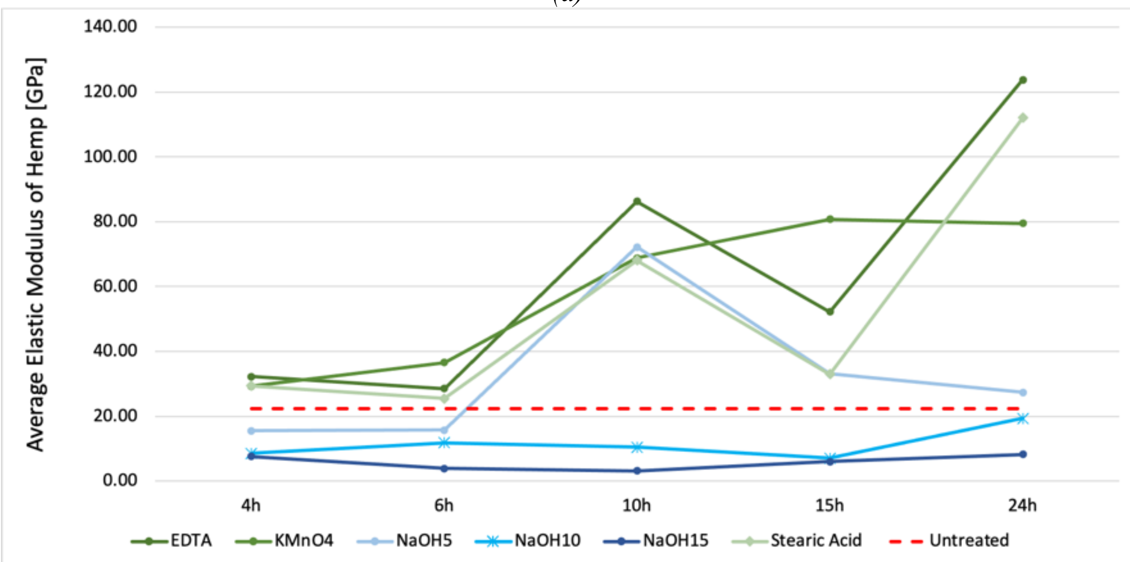

*(b)*

*Figure 4.4 – Maximum tensile strength obtained for each treatment on hemp fibres*

Therefore, the treatment using NaOH at a concentration of 10% for a duration of 24 hours was selected as it resulted in a 37% increase in the original tensile strength while only affecting the elastic modulus by 17.9% compared to the untreated option. Similarly to the findings for flax fibres, the treatment using EDTA for 4 hours also exhibited similar effects for hemp fibres. However, stearic acid treatment for 4 hours stood out in this analysis, as it resulted in a 101% increase in tensile strength, with a simultaneous increase in the elastic modulus by 31%. It is worth mentioning that the use of the stearic acid solution is a more sustainable option, being a natural saturated fatty acid. (Lodha and Netravali 2005).




*(a)*




*(b)*

*Figure 4.5 – Elastic modulus obtained for each treatment on hemp fibres.*

Confirming previous research findings, it has been demonstrated that specific surface treatments can lead to a significant increase in the tensile strength of vegetable fibres by removing organic compounds and coating the layers of cellulose. Although the results obtained in this study show an improvement in tensile strength, they remain lower compared to results found in the literature, which reported an increase in tensile strength values for treated singular flax fibres from 5705 MPa to 14103 MPa. (Sood and Dwivedi 2018; Nayak et al. 2021)

For the purpose of material characterization and future reference, Appendices E and F present the average results of elongation at break and diameter obtained for each examined sample. From these values, no relationship between changes in diameters and treatments could be identified. However, the measured values for the densities of the treated fibres did not present enough reproducibility to be included in this analysis and further investigation is recommended for future studies.

### *4.2.1 Spectroscopic analysis*

Spectroscopic analysis is suggested by authors to estimate the chemical composition of vegetable fibres. As mentioned in the previous chapter, X-Ray Diffraction was the test initially selected for this analysis, however as explained in 3.2.1 XRD showed to be unsatisfactory for the identification of chemical structures of the fibres, thus 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 on their transmittance (%T) at certain Wavenumber. (Zafeiropoulos et al. 2003; Netinger Grubeša et al. 2018; Sood and Dwivedi 2018)

Figure 4.6 brings the spectrums obtained for flax and hemp fibres before and after the selected surface treatments.

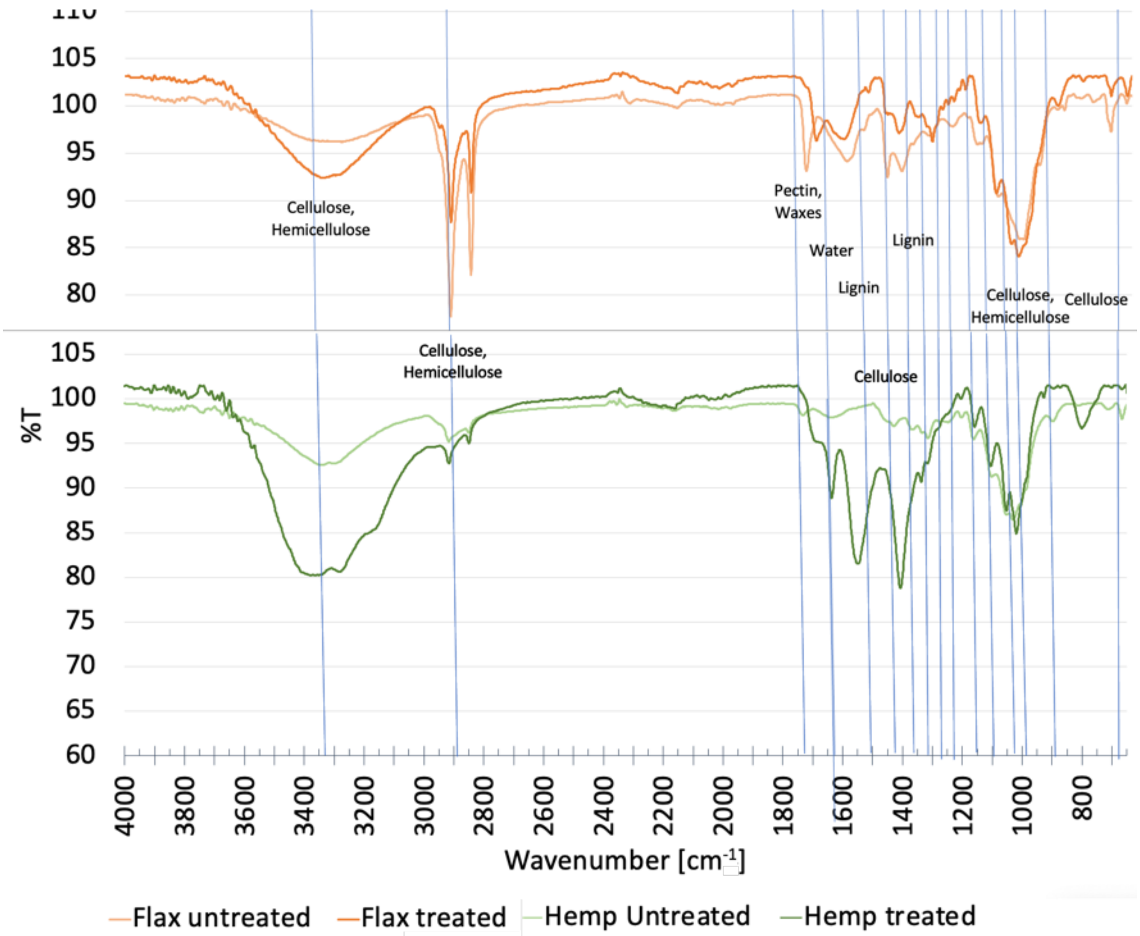



*Figure 4.6 – FTIR spectrums obtained for untreated and treated flax and hemp fibres.*

Analysing the effects of the two selected treatments (stearic acid 4h for flax and NaOH10% 24h for hemp) on the chemical structure of the fibres, it was possible to observe an increase on the presence of cellulose and hemicellulose on the wavenumber 3336cm$^{-1}$, representing an OH stretching according to Table 2.6. For flax, a reduction in pectin, waxes, water and lignin while for hemp all the compounds investigated were reduced, except for pectin and waxes. Confirming what was suggested by the literature. (Sood and Dwivedi 2018; Nayak et al. 2021)

## 4.3 Fibres behaviour under alkaline pH

The impact of an alkaline environment in the mechanical properties was estimated for samples containing 15 specimens of treated and untreated fibres. The results are presented in Figure 4.7 and Figure 4.8.

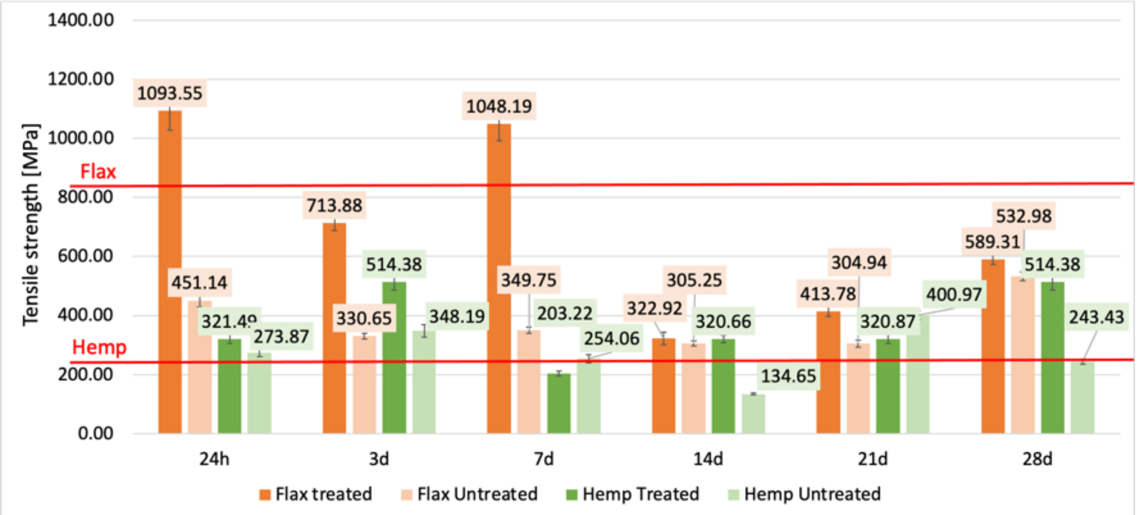



*Figure 4.7 – Effects of the degradation at alkaline solution on the tensile strength.*

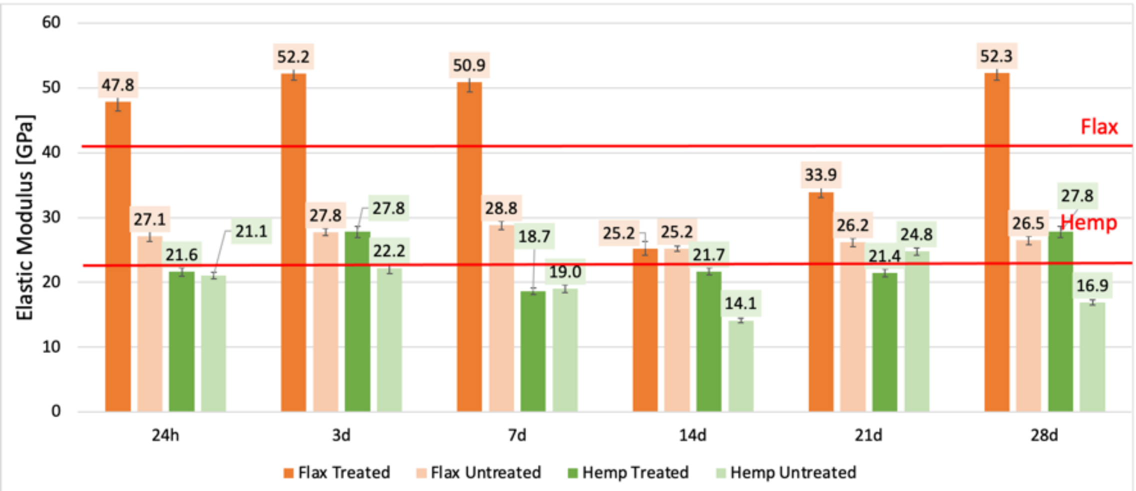



*Figure 4.8 – Effects of the degradation at an alkaline solution on the elastic modulus.*

It was observed that treated flax fibres had their maximum tensile strength increased during the first 24h, reduced after 3 days, increased again after 7 days. It dropped by 70% after 14 days and gradually increased again for the following two weeks.

However, when compared to the natural untreated fibres from Table 4.2 (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 on the tensile strength during the whole period considered.

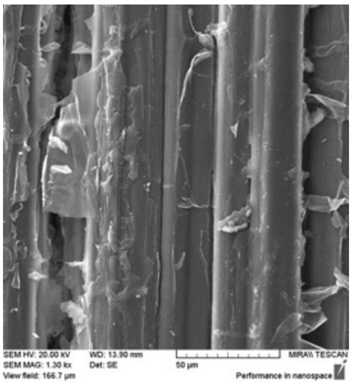
For hemp fibres, no pattern could be found for enhancement of the tensile strength through time (non-linear behaviour) but compared to the strength of natural untreated hemp fibres, after 28 days of degradation the treated fibres were 95% stronger than the natural untreated fibres from Table 4.2. Untreated fibres reduced their tensile strength by 8.4%.

In terms of the elastic modulus, the untreated flax fibres did not show significant changes during the experiment, with E reduced from 40.78 GPa (Table 4.2) to values from 25.2 to 28.8GPa. However, the treated and degraded flax fibres had their elastic modulus increased in the first 24 hours, gradually decreased during the first two weeks, and increased again during the final 14 days of the experiment. This trend could indicate an increase in the stiffness of the fibres over time.

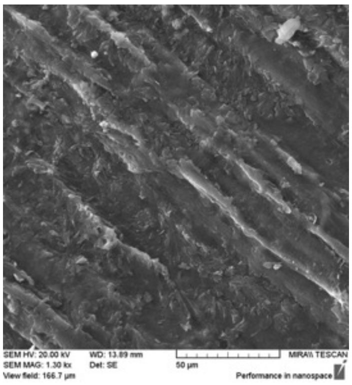
Regarding treated hemp fibres, the variability of the elastic modulus was smaller compared to flax. Results indicate that treated fibres presented increased elastic modulus after 3 and 28 days of the experiment, while degraded untreated fibres presented lower elastic modulus throughout the test except for the samples tested at 21 days. These findings suggest that treatment using NaOH 10% for 24 hours reduced the variability of property changes of fibres when exposed to an alkaline environment. Additionally, the treatment with stearic acid could have coated the fibres, allowing chemical changes in an alkaline matrix.. While the treatment with stearic acid could have coated the fibres allowing chemical changes in an alkaline matrix.

### 4.3.1 SEM images

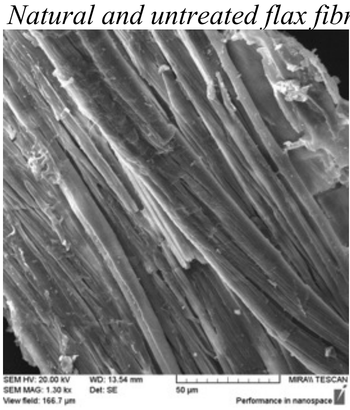
Using the scanning electron microscopy (SEM) it was possible to visualise the changes on the fibres surface on both natural and treated states before and after the degradation test, images are shown in Figure 4.9.



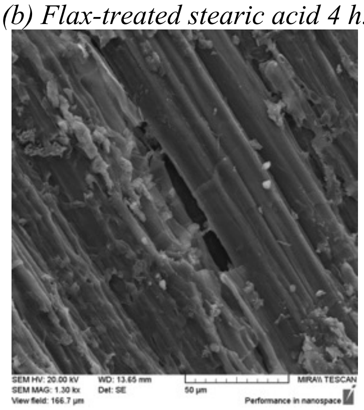
*(a) Natural and untreated flax fibres.*



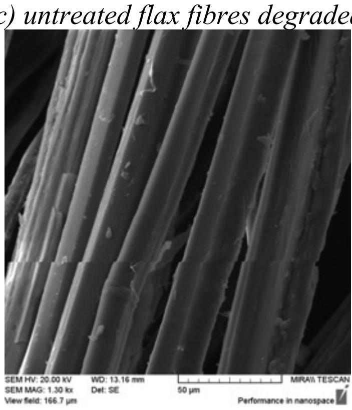
*(b) Flax-treated stearic acid 4 h.*



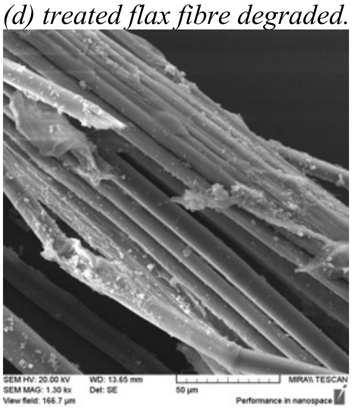
*(c) untreated flax fibres degraded.*



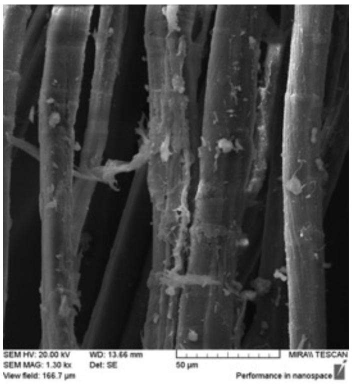
*(d) treated flax fibre degraded.*



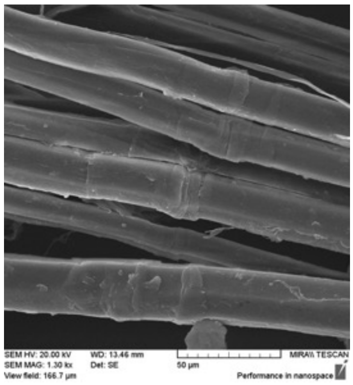
*(e) Natural and untreated hemp fibres.*



*(f) Hemp fibres treated NaOH10% 24 h.*



(g) untreated hemp fibres degraded.



(h)treated hemp fibre degraded.

*Figure 4.9 – SEM images of untreated and treated flax and hemp fibres before and after degradation test.*

Both flax and hemp fibres exhibited the presence of a coating after being treated (as shown in Figure 4.9b and f) , which indicates the effectiveness of the treatments. Upon exposure to an alkaline environment, the untreated fibres suffered damage and their internal layers were exposed (as seen in Figure 4.9c and Figure 4.9g) , whereas the treated samples exhibited structures similar to those of the natural and untreated samples, confirming that the adopted treatments could enhance the durability of the fibres in concrete (Figure 4.9d and Figure 4.9h, similar to Figure 4.9a and Figure 4.9e).

### 4.3.2 Summary

30 different combinations of treatment were assessed in this section for samples of fax and hemp fibres. Aiming an increase on the tensile strength and a reduction on the elastic modulus, the treatment selected for flax fibres was with stearic acid during for hours for increasing the tensile strength, in comparison to untreated fibres, and presented lower increase on the elastic modulus, in comparison to the other treatments

studied. For hemp fibres, the only treatment that presented increase on the tensile and reduction on the Young's modulus was with NaOH 10% during 24h.

By exposing the fibres to an alkaline solution, it was possible to observe that treated and untreated flax fibres had their tensile strength reduced overtime, however, the elastic modulus of treated fibres increased, while the elastic modulus for untreated fibres decreased with smaller deviation.

On the other hand, both untreated and treated hemp fibres did not present high variability in the elastic modulus through the degradation test, indicating a possible stabilisation of the stiffness with treatment. However, analysing the SEM images, for treated and untreated fibres, both hemp and flax fibres treated presented more uniform surface after the degradation test in comparison to the untreated fibres in the same test.

Although the selected treatment for flax fibres did not allow a predictability on its behaviour in concrete through time, their practical effects were still studied in the next section of this research for an optimised fibre-reinforced concrete.

## 4.4 Fibre-reinforced concrete

As detailed in the previous chapter, a preliminary concrete mixture was formulated and multiple mixes were produced, comprising a control mix with no added fibres, as well as FRC using untreated flax and hemp fibres, polypropylene fibres, basalt and steel fibres. Triplicate tests were conducted at 7 days and based on the results obtained, an optimised control mixture was formulated. This section presents and discusses the preliminary results obtained, as well as the results for the optimised mixture in the latter part of this section, including additional relevant tests conducted as part of the study.

### *4.4.1 Preliminary fibre reinforced concrete mixture*

#### *4.4.1.1 Concrete workability*

The slump of each mixture was measured at its fresh state, and the results are plotted in Figure 4.10. The slump designed was 10 to 30mm and comparing the values obtained for the control mixture with the FRC, the addition of all types of natural fibres reduced the workability of the mixtures. Polypropylene also reduced the workability, however, the results still fit the workability designed.

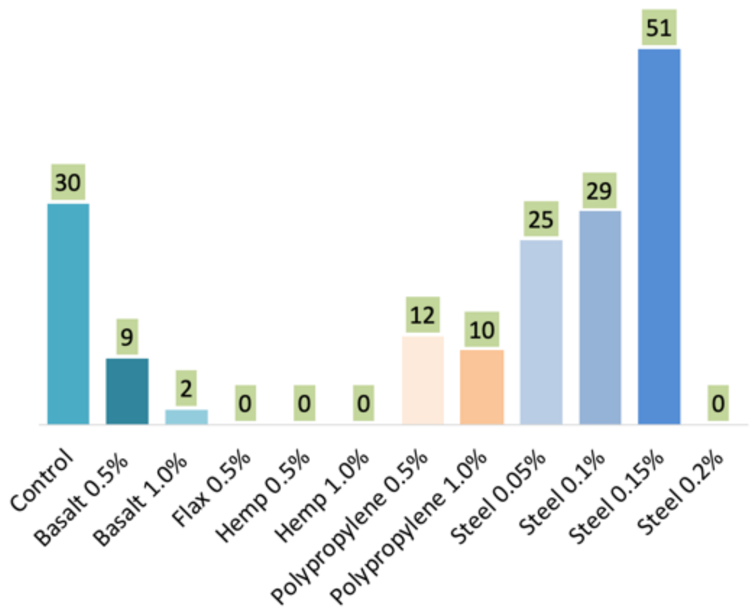



*Figure 4.10 – Slump of fresh preliminary mixtures.*

Confirming the high hygroscopicity of flax and hemp fibres, the slump obtained for the vegetable FRC containing these fibres was reduced to zero. For steel FRC mixes, the results indicate that their addition over 0.1% increases the slump of concrete. This behaviour can be justified by the non-porous surface of steel, which in small quantities, would reduce the friction between the concrete contents, increasing its flowability. However, for the mix containing 0.2% of steel fibres, a slump of zero was observed. In a study on the effects of the workability of steel fibres reinforced concrete, it was observed that workability reduced with an increase in fibre content, which could corroborate the result obtained for the mix of 0.2%. (de Figueiredo and Ceccato 2015)

As shown in Figure 4.10, the control mix had a slump of 30 mm, while the mixes containing 0.5% and 1.0% of basalt fibres had slumps of 8 mm and 2 mm, respectively. The flax and hemp FRC mixes at 0.5% had a slump of zero. The FRC mixes reinforced with 0.5% and 1.0% of polypropylene fibres had slumps of 12 mm and 10 mm, respectively. For the steel FRC mixes, the slump increased with the increase in fibre content up to 0.15%, which had a slump of 51 mm. However, for the mix containing 0.2% steel fibres, the slump was reduced to zero.

The obtained results suggest that the addition of flax and hemp fibres can significantly affect the workability of concrete mixes due to the hygroscopic behaviour of vegetable fibres. Basalt and polypropylene fibres had a smaller effect on the slump values, while steel fibres showed an increase in the slump up to a certain threshold, after which it suddenly decreased. These results show the differences between behaviours of steel fibres and the other considered types indicating incompatibility for comparison, and also highlight the importance of carefully selecting and optimizing the type and amount of fibres added to concrete mixes to achieve the desired properties.

#### 4.4.1.2 *Compressive Strength ($f_{cu}$) and relative density*

As described in section 3.4.1 the control mixture was designed as C30 with 5% defectives at 7 days, and included a standard deviation of 8 to increase the strength to 43.12N/mm². After 7 days, the cubes were tested in their hardened state, and the resulting compressive strength values are presented in Figure 4.11. The figure also includes the relative density values, allowing for a visual comparison of both properties.

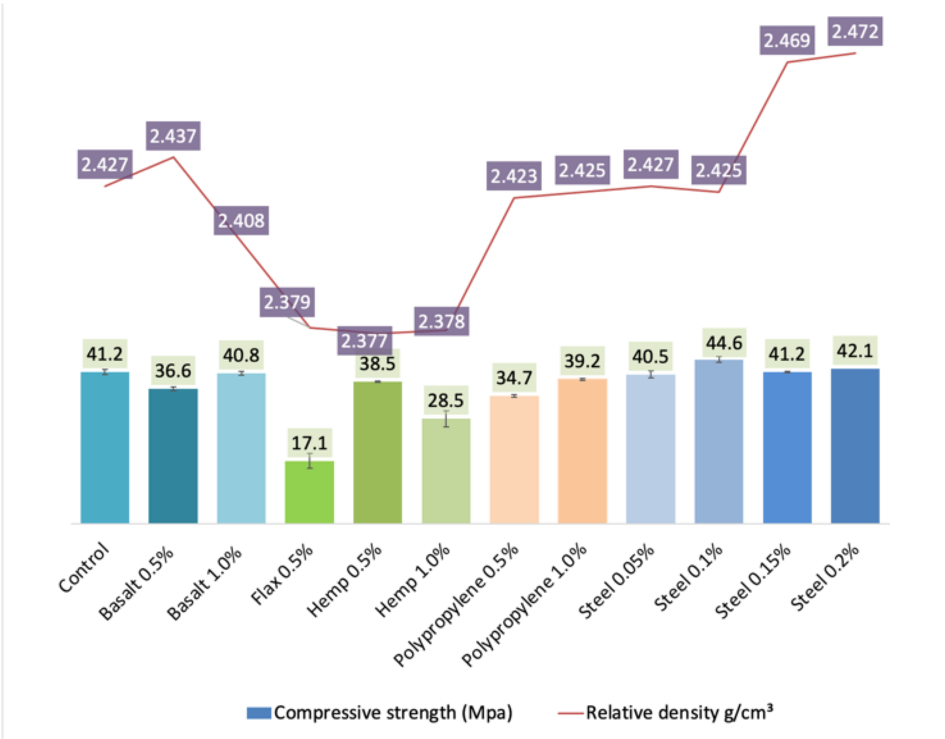



*Figure 4.11 – Compressive strength and relative density at hardened state.*

As shown, the control mix achieved, on average, 95% of the target compressive strength. Among the mixtures studied, the one containing 0.1% steel fibres presented the highest compressive strength. Basalt and polypropylene fibres showed similar behaviour, with values slightly higher for BFRC. However, the mixes containing 0.5%

of basalt and polypropylene, and 1.0% of hemp fibres showed a reduction of up to 30% when compared to the control mix.

The results obtained for vegetable FRC indicated a reduction in compressive strength for all mixtures tested. An increase in the content of hemp fibres significantly reduced the strength, and a higher standard error between specimens was also observed. The mix containing 0.5% flax fibres also showed low workability, as measured by the slump test.

The specific gravity values revealed a drop in density for all vegetable FRC mixes, with values ranging from 2.2 to 2.5g/cm$^3$, which is compatible with values reported by other authors in the literature review.

Also, the graph shows that the compressive strength values vary greatly between the different types of fibres used. Hemp fibres resulted in a significant reduction in compressive strength as the percentage of fibres increased. The specific gravity values were generally consistent across the different types of fibres tested, with a notable decrease in density observed for all vegetable FRC mixes. In contrast, the compressive strength of concrete mixes increased as the percentage of steel fibres added increased, indicating once again that it would not be adequate to perform a comparison between them and vegetable fibres. The findings confirm that the relationship between fibre content and compressive strength is complex, and that adding vegetable fibres reduces the density of the concrete.

#### 4.4.1.3 Post-cracking analysis

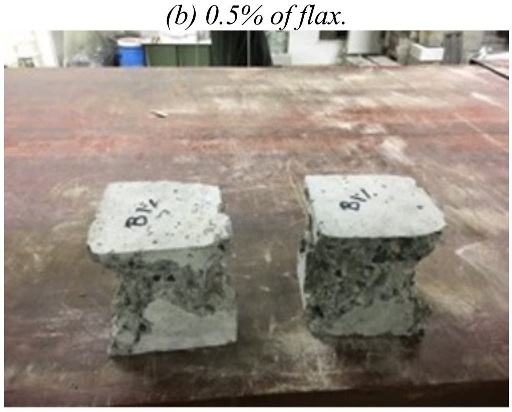
To validate the results obtained, as recommended by the standard, an evaluation of the type of fracture of the cubes was performed to define if they were or not satisfactory according to the types of failures shown in Figure 2.25 and Figure 2.26.

Figure 4.12 (a) shows the apple shaped fracture of the plain control mixture. It could be observed from this fracture a satisfactory failure on both aggregate and mortar, as recommended by the followed standard.

*(a) Control mixture.*

*(b) 0.5% of flax.*

*(c) 0.5% of basalt.*



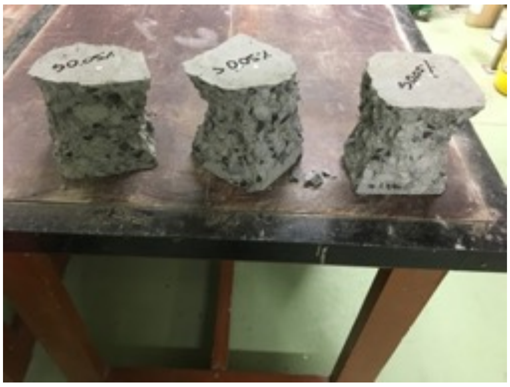
*(d) 1.0% of basalt.*

(e) 0.5% of hemp.

(f) 1.0% of hemp.

(g) 0.5% of polypropylene.

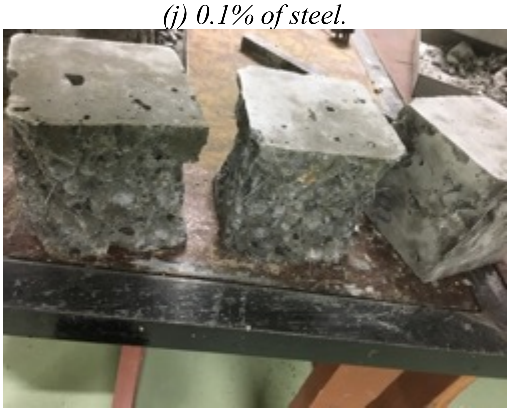
(h) 1.0% of polypropylene.



(i) 0.05% of steel.

(j) 0.1% of steel.

(k) 0.15% of steel.



(l) 0.2% of steel.

*Figure 4.12 – Failure analysis of samples tested.*

Mixtures containing basalt (c and d), steel (k to l), 0.5% of hemp and polypropylene (e and g) showed satisfactory performance, as evidenced by the apple or pyramid-shaped specimens that confirm the well-centred strength of the cubes. Additionally, all specimens exhibited both aggregate and mortar failures, which is indicative of good adhesion of the mixtures.

Cubes containing 1.0% of hemp and polypropylene (f and h) could not have their post-cracking behaviour analysed since it was not possible to separate the fractured sections from the rest due to the strong bond between fibres and concrete. On the other hand, the mix containing 0.5% of flax fibres (b) showed a bond failure between the aggregate and mortar, which suggests that the fibre content was too high, affecting the fibre-matrix bond.

As detailed in the previous chapters, from the three-point bending test it was possible to evaluate the fracture energy (Gf), elastic modulus (E), the tensile strength at peak load ($F_{R,j}$) and the residual tensile strength at deflection *n* ($F_n$). The results obtained for the preliminary part of this study are presented ahead.

### *4.4.1.4 Fracture energy (Gf) and Elastic modulus (E)*

Table 4.3 shows the fracture energy and elastic modulus results obtained from the three-point bending test for various types of fibre-reinforced concrete mixes compared to the control mix. The addition of fibres resulted in an increase in fracture energy and a reduction in the elastic modulus compared to the control mix, indicating a decrease in concrete brittleness. This is consistent with the findings from the literature. (Liu et al. 2019)

*Table 4.3 – 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% |

Among the vegetable fibres, the mix with 0.5% flax fibres had the highest fracture energy increase of 88% compared to the control mix, which was greater than the increases obtained for mixes reinforced with basalt (0.5% and 1.0%) and polypropylene (0.5% and 1.0%) fibres. Hemp fibres resulted in the highest increase in fracture energy among the 0.5% fibre mixes, surpassing even the mix reinforced with hemp at 1.0%.

For elastic modulus, the mix with 0.5% flax fibres showed a 49% reduction in elastic modulus compared to the control mix, which increased the concrete ductility and reduced its brittleness. This was the third-smallest reduction in elastic modulus among the mixes, only higher than the mixes with hemp at 1.0% and polypropylene at 0.5%. The addition of basalt fibres at 0.5% and 1.0% resulted in 62% and 60% of the control mix's elastic modulus value, respectively. The mixes with steel fibres showed only a slight reduction in elastic modulus. There was no clear correlation between the percentage of fibres and the elastic modulus.

Mixes containing less than 0.15% of steel fibres did not have significantly different results from the control mix, while those with 0.15% or 0.2% showed considerably higher results. Comparing the mixes reinforced with basalt and polypropylene fibres, fracture energy values of similar magnitude were observed for the proportions of 0.5% and 1.0%.

The addition of small percentages of steel fibres increased the post-crack tensile strength of the mixtures, but only additions over 0.15% significantly increased the fracture energy. It was not possible to establish a relationship between the use of steel fibres and the other types of fibres used in this preliminary stage of the study, confirming that a comparison between steel fibre reinforced concrete and vegetable fibre reinforced concrete would not be appropriate due to their significantly different properties.

#### *4.4.1.5 Flexural tensile strength ($f_{R,j}$) and ($f_n$) [MPa]*

To conclude the analysis performed in the preliminary mix designed, the flexural tensile strength at peak load ($f_n$) and residual strength ($f_{R,j}$) after failure were assessed. The results are presented in Table 4.4, where j from $f_{R,j}$ correspond to values from Table 2.9. The addition of most fibres reduced the peak value obtained for flexural tensile strength ($f_n$), except for the mixtures containing basalt 1.0%, hemp 0.5% and steel 0.15%. For instance, the incorporation of 0.5% of untreated flax fibres reduced the flexural tensile strength of concrete from 4.71 MPa to 3.21 MPa. Moreover, an increase in fibre content also reduced the peak tensile strength, except for basalt and polypropylene, which presented higher $f_n$ with an increase in fibre content. On the other hand, the mix containing steel fibres reached a peak value smaller than the mixes reinforced with hemp 0.5% and basalt 1.0%.

*Table 4.4 – Flexural tensile strength ($f_n$) and ($f_{R,j}$) [MPa], j values from Table 2.9.*

| ***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 | - | - |

Regarding residual tensile strength, the control mix presented values $f_{R,j}$ until j=4, similar to the mixes reinforced with less than 0.15% of steel fibres. However, all the other fibres increased the residual flexural tensile strength to j=5. The mixes containing basalt 0.5%, flax 0.5%, hemp 1.0% and steel 0.2% achieved a net deflection of 2.77mm (j=6), with hemp fibres providing the greatest $f_{R,j}$, followed by steel 0.2%, flax 0.5% and lastly basalt 1.0%.

In conclusion, the incorporation of most fibres decreased the peak flexural tensile strength, except for basalt 1.0%, hemp 0.5%, and steel 0.15%. However, all fibres increased the residual tensile strength. Hemp fibres showed the greatest $f_{R,j}$, followed by steel 0.2%, flax 0.5% and basalt 1.0%. The mix containing basalt and polypropylene presented higher fn with an increase in fibre content, while the mix reinforced with steel fibres reached a smaller peak value than those reinforced with hemp 0.5% and basalt 1.0%. These results suggest that the incorporation of specific types of fibres can improve the residual tensile strength of concrete.

#### *4.4.1.6 Summary*

From the preliminary mix design, it was possible to identify limitations that needed to be addressed for an optimized concrete. The first issue was a reduction in workability caused by flax and hemp fibres, leading to a non-cohesive casting process. To overcome this problem, three measures could be adopted for an optimized mix design: first, the designed slump for the control mix could be increased; second, a plasticizer additive could be used; and third, a reduction in the fibre content could be evaluated.

Mixtures containing steel fibres did not produce results comparable to the other fibres and were not included in the following part of this study.

Another measure to be considered could be the reduction of the maximum aggregate size to 10mm, which could increase the possible applications of the concrete developed in this research to thinner structures such as concrete panels, blocks, and floor leveling.

These results confirmed the range values for the properties of singular fibres, and also highlighted the results and challenges observed by authors mentioned in the literature review. For the FRC mixes, in comparison to the control mix, results for the slump test confirmed the high hygroscopicity of the fibres. The addition of fibres increased the fracture energy, reduced the elastic modulus, and could add some residual tensile strength. Although flax fibres reduced the peak load for compressive and flexural tensile strength, the residual strength observed was superior to the mixes reinforced with basalt and polypropylene fibres. The surface treatments detailed in the next section allowed an evaluation of the enhancement of the properties and the selection of the most relevant treatments for flax and hemp fibres to be used as reinforced concrete in the final part of this study.

### *4.4.2 Optimized FRC mix*

After evaluating the effects of surface treatment on hemp and flax fibres, concrete mixtures were made and tested to have their properties assessed. As mentioned, the control mixture was redesigned after the previous preliminary analysis aiming for higher workability and reduction of maximum aggregate size, to increase the possibilities of applications for the new mix. The results obtained are presented and discussed ahead in this chapter.

### *4.4.2.1 Compressive Strength ($f_{cu}$), Density and Slump*

Firstly, two samples made of three specimens were prepared using the optimised control mix to verify the reproducibility of the values obtained in comparison with previous and future fibre-reinforced concrete mixes. Subsequently, fibres were added to the optimised mix, and tests were conducted on sets of three specimens for each mix, starting with 0.5% flax, hemp, polypropylene, and basalt fibres. Based on the results, new mixes were prepared with smaller amounts of fibres, and the values obtained for slump are presented in Figure 4.13. Results for compressive strength and specific gravity tested at 7 and 28 days are presented in Figure 4.14.

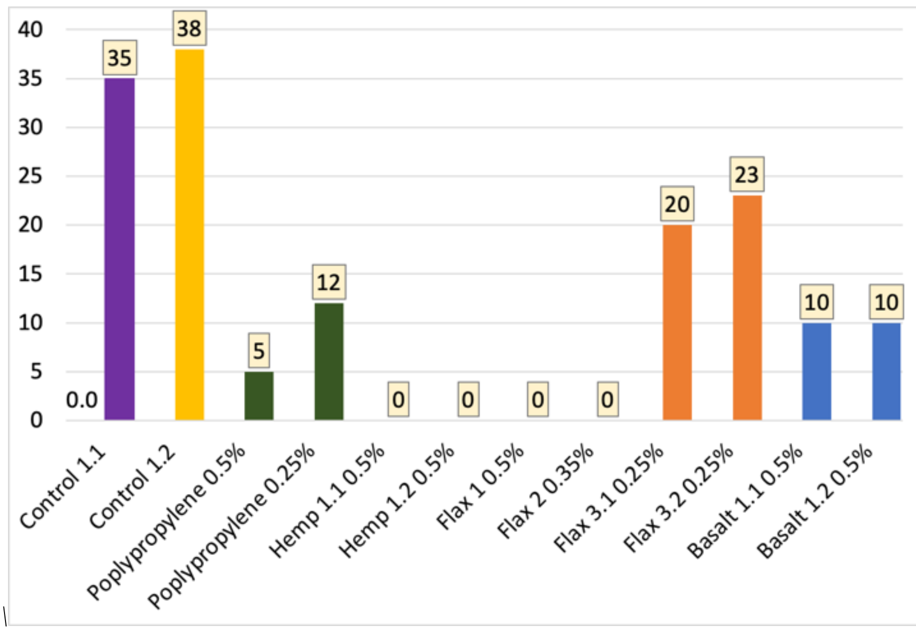



*Figure 4.13 – Slump obtained during mix optimisation.*

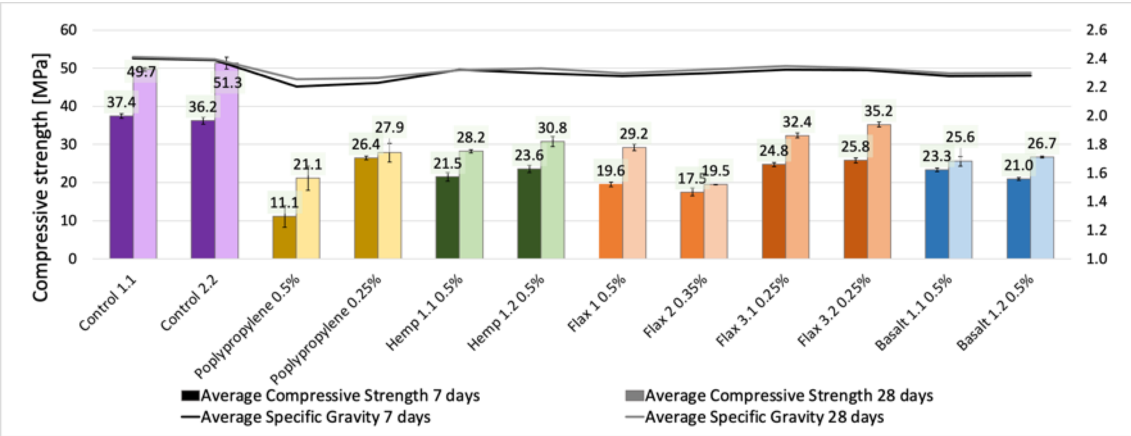



*Figure 4.14 – Compressive strength [MPa] and specific gravity [g/cm$^3$].*

As noted, the optimised concrete mix also experienced a reduction in slump with the addition of fibres, with the mixes containing 0.5% of flax and hemp fibres achieving a slump of zero. A reduction in compressive strength was also observed, including for the mixes reinforced with 0.5% of polypropylene. A new mix containing 0.35% of flax fibres was tested, but results were still unsatisfactory. Finally, the addition of 0.25% of flax fibres was found to achieve a certain level of workability without compromising the compressive strength. A mix containing 0.25% of polypropylene was also prepared for comparison purposes.

At 7 days, the control groups had the highest compressive strength, with Control 1.2 producing the highest value of 51.3 MPa. However, the other mixes had significantly lower values, with the polypropylene mixes having the lowest values. At 28 days, the compressive strength of all mixes increased, with the control groups still having the highest values, but the difference between the control groups and the fibre mixes decreased.

Similarly, in the preliminary stage of the study, the control mix achieved a high compressive strength of C50 after reaching the designed age. However, when fibres were incorporated into all the mixtures, the compressive strength was reduced to less than 32MPa, which was the target for the study. The goal of the study was to propose a satisfactory mixture of vegetable fibre reinforced concrete, with the novel application of flax compared to hemp. For that, the concentration of flax fibres was optimised based on the results obtained. By reducing the percentage by volume of treated flax from 0.5% to 0.25%, it was possible to achieve the desired compressive strength of C32 for the both samples produced in triplicates.

Again, although high variability of properties was observed when testing singular fibres, the results obtained for concrete reinforcement were consistent between samples, noticed by the small error bars from Figure 4.14.

Confirming what was seen in the literature, results show that the addition of fibres to concrete tend to reduce its compressive strength, with the performance varying depending on the type and percentage of fibres used. In smaller quantities, the results indicate a potential of flax fibres, as viable alternatives to traditional fibres like polypropylene and basalt. Additionally, all specimens presented satisfactory fracture types, indicating that the fibre reinforced concrete mixtures were effective in enhancing the durability of the material.

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 through from samples tested at 7 in comparison to those tested at 28 days.

#### 4.4.2.2 *Fracture Energy (Gf), Young's Modulus (E)*

Table 4.5 shows the average values obtained and calculated from the 3-point bending test for Fracture Energy (Gf) and Young's modulus (E).

*Table 4.5 – Fracture energy and elastic modulus of FRC.*

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

At 7 days, the Basalt 0.5% mix showed the highest fracture energy of 1296.88 N/m, which is more than 6 times higher than the control sample. However, its elastic modulus of 11.14 GPa is only 40% of the control. On the other hand, the mix containing 0.5% of flax had a fracture energy of 129.80 N/m and elastic modulus of 25.13 GP, which is 65% and 91% of the control, respectively. The Hemp 0.5% mix had a fracture energy of 144% of the control, and an elastic modulus of 70% of the plain concrete mix.

In comparison to the values previously obtained presented in Table 4.3, 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 7 days. Which could possibly be attributed to the initial adhesion of the mortar in the fibres. However, after 28 days, the same mixture presented an increase of 53% in comparison to the control mix indicating that the hardening process was not compromised.

The mixture containing hemp fibres presented, at 7 days, an increase on the Gf of 44% and after 28 days the percentage rose to 254% of the control confirming that hemp fibres increase the fracture energy of concrete structures.

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 7 days to those conducted at 28 days, contrary to the mix containing flax fibres that at 7 days presented an elastic modulus proportional to 91% of the control and at 28 day the value was reduced to 72%, equivalent to the mix reinforced with 0.5% of hemp fibres.

#### 4.4.2.3 *Flexural tensile strength ($f_{R,j}$) and ($f_{n}$) [MPa]*

Before comparing the results from Table 4.6 to the values obtained in the first mixture (Table 4.4), 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 than the mix tested at 7 days, the values were still considered in this analysis.

Comparison of these results with the preliminary analysis showed that the second control mix had lower flexural tensile strength values at peak load, indicating a slight decrease in this property by reducing the maximum aggregate size. However, the residual tensile strength obtained for the flax and hemp-reinforced mixes at 7 days increased, with residual strength observed at deflections j=7 and j=8.

*Table 4.6 – Flexural tensile strength ($f_n$) and ($f_{R,j}$) [MPa], j values from Table 2.9.*

| 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 7d | 3.95 | 0.30 | 0.40 | 0.57 | 1.48 | 0.11 | - | - | - |
| Basalt 0.5% 7d | 6.02 | 0.35 | 0.49 | 0.75 | 1.73 | 5.13 | 5.92 | - | - |
| Flax 0.25% 7d | 3.07 | 0.38 | 0.55 | 0.90 | 2.30 | 0.97 | 0.44 | 0.21 | 0.15 |
| Hemp 0.5% 7d | 3.66 | 0.41 | 0.63 | 1.04 | 2.68 | 3.06 | 1.95 | 1.39 | 1.54 |
| Control 28d | 4.03 | 0.79 | 1.29 | 2.16 | 0.17 | - | - | - | - |
| Basalt 28d | 9.15 | 0.60 | 0.97 | 1.67 | 4.34 | 7.17 | 3.07 | - | - |
| Flax 0.25% 28d | 4.23 | 0.46 | 0.73 | 1.23 | 3.03 | 1.00 | 0.34 | - | - |
| Hemp 0.5% 28d | 4.60 | 0.59 | 0.97 | 1.73 | 4.09 | 3.66 | 2.08 | - | - |

Confirming a better performance of the optimised mixture, at 28 days, all the mixes showed higher values than those obtained at 7 days, except for the mix reinforced with 0.25% treated flax fibres, which exhibited a residual tensile strength equivalent to the mix containing 0.5% untreated fibres in the preliminary analysis.

### *4.4.2.4 Thermal Conductivity*

To evaluate the insulation related to each mixture, their thermal conductivity was measured following the procedure detailed in 3.4.10 As seen in the literature review, thermal conductivity is the inverse of thermal resistivity, where lower values represent enhanced thermal insulation (Bala and Gupta 2021).

Figure 4.15 shows the experimental results obtained, which, although slightly lower in magnitude than expected, values are compatible with results reported in the literature for the same property evaluated on concrete reinforced with waste tile rubber as a replacement for sand, which ranged from 0.96 to 0.85 [W/mK]. (Bala and Gupta 2021)

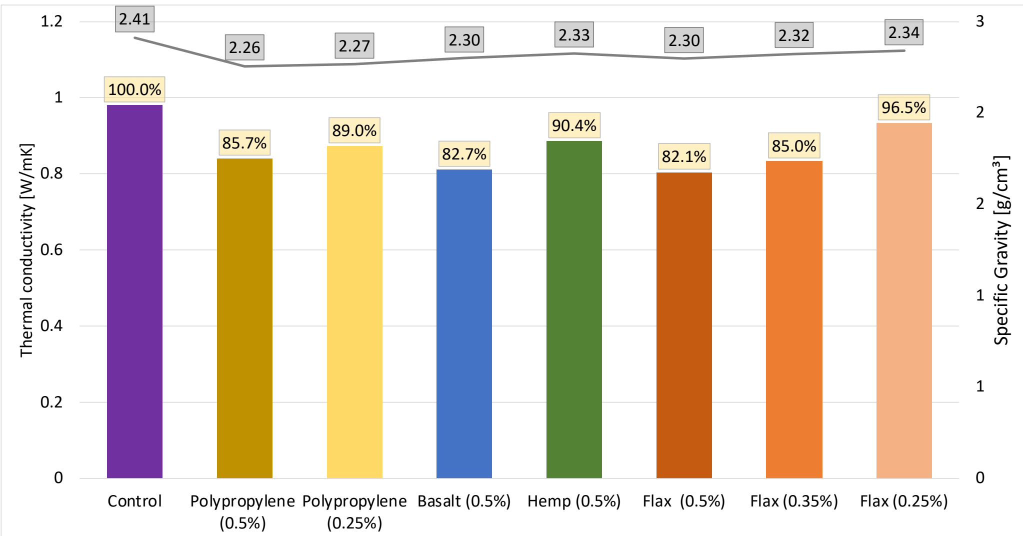



*Figure 4.15 –Thermal conductivity [W/mK] and Specific Gravity [g/cm³]*

As expected, the addition of fibres led to a reduction in thermal conductivity for all samples, as anticipated. Among the various mixtures, the ones reinforced with basalt and flax fibres performed the best. Basalt is a commonly used material in the construction sector for insulation. The mix containing 0.5% of flax fibres also presented favourable results.

Values of specific gravity were once again included to allow a comparison between density and thermal conductivity. As previously observed, samples with higher fibre volumes had lower density. The relationship between density and thermal conductivity further confirms previous research as discussed in the literature review.

### 4.4.2.5 Depth of penetration of water under pressure

Figure 4.16 presents the results of an experiment conducted to measure the water penetrability of the hardened concrete mixtures. In general, the presence of fibres increased water penetration compared to the control mix. However, the addition of 0.25% of flax fibres resulted in values that were virtually similar to the control mix. This suggests that, at this proportion, the cement paste would be able to coat the fibres, making it difficult for water to penetrate into the matrix. On the other hand, the mixes containing hemp fibres exhibited up to 50% higher penetration levels compared to the control and flax 0.25% mixes.

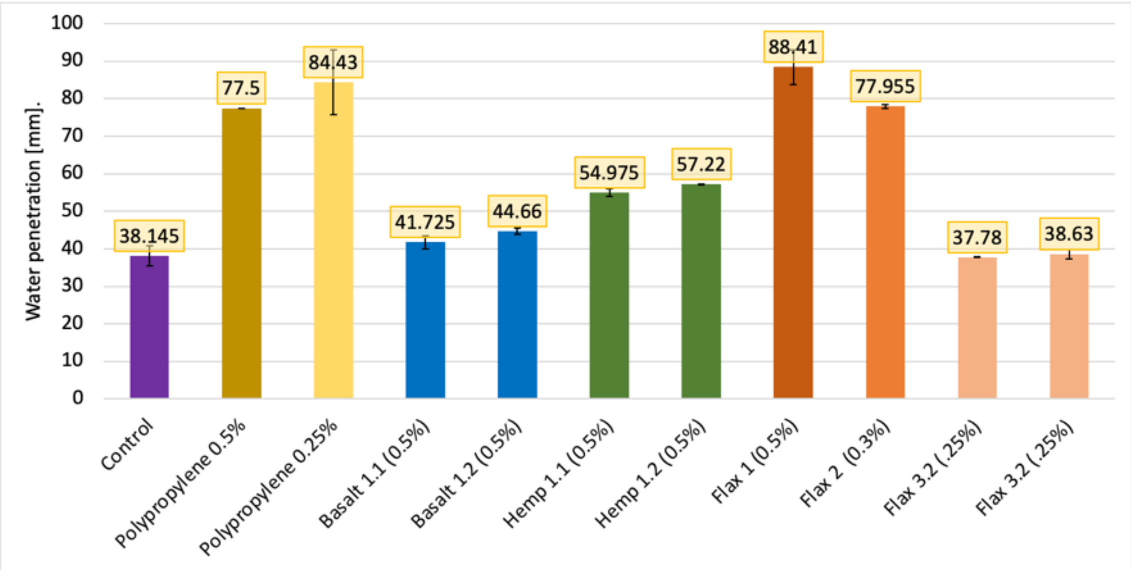
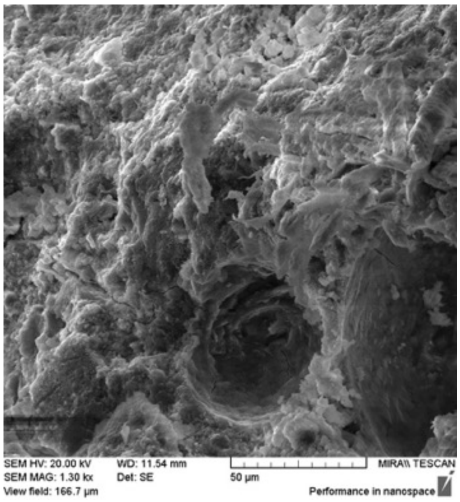



*Figure 4.16 – Water penetration [mm].*

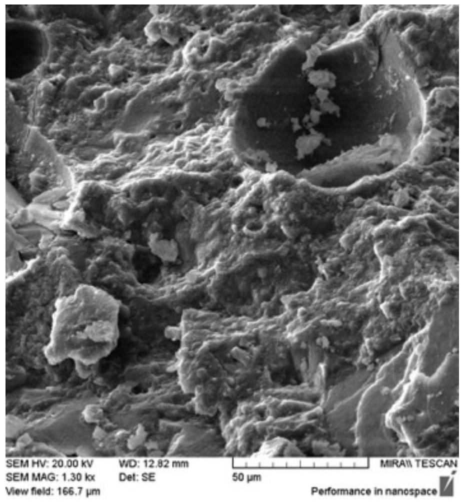
Surprisingly, the mixes containing polypropylene, a synthetic material, presented higher levels of water penetration, which could indicate lower fibre-matrix adhesion.

### *4.4.2.6 SEM Images*

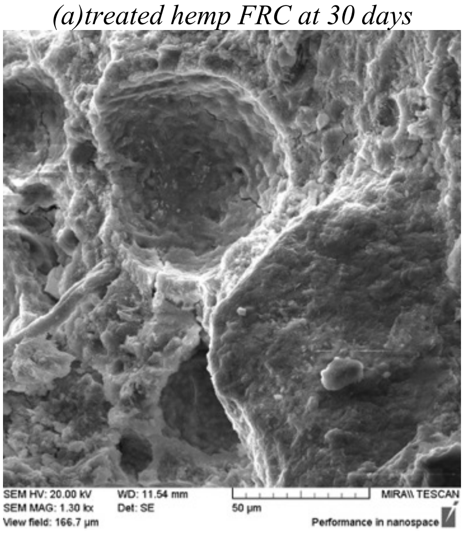
The SEM images presented in Figure 4.17 provide valuable insights into the microstructure of FRC mixes at different ages, allowing for a better understanding of the behaviour of fibres and their interaction with the cement matrix over time.



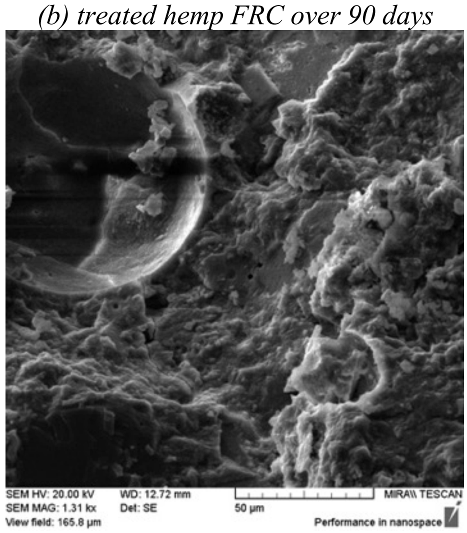
*(a)treated hemp FRC at 30 days*



*(b) treated hemp FRC over 90 days*



*(c) treated flax FRC at 30 days*



*(d) treated flax FRC over 90 days*

*Figure 4.17 – SEM images of FRC at 30 days and over 90 days.*

At 30 days of age, the SEM images reveal a strong presence of fibres with a fibrous aspect, which is expected given the reinforcing effect of the fibres in the fresh concrete. However, after 90 days, the cementitious bond appears much closer, and the presence of fibres is reduced, which may indicate the degradation of the fibres associated with the concrete strengthening.

The reduction of fibres over time can be attributed to several factors, including the continuous degradation of fibres and ageing of the concrete. Previous studies have shown that the degradation of fibres in concrete is a complex phenomenon that depends on several factors such as fibre type, length, and orientation, as well as environmental conditions such as temperature, humidity, and exposure to chemical agents. The SEM images presented in this study confirm the previous findings, highlighting the reduction of fibres over time.

While the SEM images provide valuable insights into the microstructure of the FRC mixes, it was not possible to evaluate the change in the percentage of voids caused by the degradation of the fibres and ageing of the concrete in this study.

## 4.5 Summary and limitations

The experimental study here described allowed an overview of the effects of vegetable fibres as reinforcement of concrete. The study compared the properties of flax and hemp FRC using both untreated and treated fibres. However, due to the COVID-19 pandemic, the acquisition of more flax fibres for additional tests was affected.

The optimized concrete mixture developed using 0.25%vol of treated flax fibres met the designed compressive strength, surpassing mixes containing polypropylene, basalt, and hemp fibres. Additionally, the second mix tested, still presented zero values when reinforced with 0.5% of treated vegetable fibres, and a reduction of 0.25% in the fibre content of flax fibres increased the slump from zero to 23mm, presenting enhanced workability.

Regarding fracture energy, the FRC mixes tested during the preliminary study presented higher fracture energy values, but at 7 days, the optimized mixture showed a reduction in fracture energy by 35%. Nevertheless, at 28 days, the same mixture presented 153% higher fracture energy than the control mix tested at the same age. Mixtures reinforced with basalt fibres presented the highest increase in fracture energy, and all the mixes showed higher values of fracture energy at 28 days compared to 7 days.

All the mixes reduced the stiffness in terms of elastic modulus, confirming that their addition can reduce the concrete brittleness. In terms of flexural tensile strength, the reduction of the maximum aggregate size reduced the maximum tensile strength of the control mix, and at 7 days, the mixture containing only the mix reinforced with basalt fibres was able to increase the peak value. However, at 28 days, all the FRC tested presented higher flexural tensile strength than the control mixture.

The surface treatment performed improved the residual tensile strength of flax and hemp fibres at 7 days. However, the residual tensile strength was reduced from samples tested at 7 days when compared to those tested at 28 days. Nevertheless, the results obtained for the mixes using treated fibres and tested at 28 days presented a similar magnitude to the mixes using untreated fibres and tested at 7 days.

The addition of fibres reduced thermal conductivity, and a direct relationship was observed between the fibre content, thermal conductivity, and density. For water penetration, higher values were obtained for mixes containing a higher volume of fibres, except for polypropylene fibres, which could indicate poor adhesion between the fibre matrix in comparison to natural fibres.

Finally, the SEM images for the vegetable FRC tested indicated a reduction in the fibre content over 90 days compared to 30 days, indicating a continuous degradability of the structure of the fibre in the concrete matrix. Although SEM images could not be performed on the FRC produced with untreated fibres, future work could explore this.

In conclusion, this study demonstrates the potential of vegetable fibres as reinforcement for concrete, particularly flax and hemp fibres. However, further research is required to explore the durability of these fibres in concrete over an extended period. Additionally, it would be interesting to investigate the possibility of combining different fibres to optimize their performance in concrete.

# Chapter Five

# Conclusions and Future Work

# Chapter 5: Conclusions and Future Work

## 5.1 Conclusions

The aim of this study was to evaluate the suitability of vegetable fibres for reinforcing concrete. Based on properties previously reported in research, the experimental phase began with the selection of the vegetable fibres: flax fibres were chosen as a novelty, and hemp fibres as a benchmark seen that they are commonly used for reinforcing concrete. Basalt, polypropylene and steel fibres were also included in the preliminary section of the study when different mixes were produced to assess the performance of untreated flax fibres in comparison to the other fibre reinforced concrete (FRC) produced. Although basalt fibres are often classified as natural due to being produced from mineral raw materials, they require industrial production, like glass fibres, and are also considered synthetic fibres.

Confirming limitations presented by previous authors on the topic, this study expected high variability of results during the characterisation of the properties of vegetable fibres, as well as a reduction in the compressive strength of the fibre reinforced concrete, compared to a plain mixture, and increased values for water penetration, as vegetable fibres have high hygroscopicity. The literature suggests that performing a chemical surface treatment on vegetable fibres would improve their properties, making them more consistent and predictable.

Flax and hemp fibres, the selected vegetable fibres, were then subjected to six different surface treatments for five different lengths of time, totalling 30 different combinations of chemical surface treatments. The most effective treatments were selected for flax and hemp fibres, considering their increase in tensile strength and reduction in the stiffness of the fibres, properties sought by their use as reinforcement in concrete. During this stage, the environmental impact of the chemicals used was also assessed as one of the main motivators for the adoption of vegetable fibres as a replacement for current non-natural fibres commercialized for reinforcement of concrete is to reduce the high environmental footprint from the construction sector.

Among the treatment options, the surface treatment using stearic acid for 4 hours was selected for flax fibres, a natural fatty acid that was able to double the tensile strength of the fibres while increasing the stiffness by 30%. Another treatment that presented similar positive results was the one using EDTA for 4 hours. Due to the limited amount of fibres purchased and the non-availability during the COVID-19 pandemic, only the first treatment was included for the optimized fibre-reinforced concrete production.

For hemp fibres, the treatment that satisfied both parameters of increasing the tensile strength and reducing the elastic modulus was the treatment using NaOH 10% for 24 hours. Although NaOH is an extremely hazardous chemical, it is commonly used by other authors and outstood among the other treatments, being selected for the final part of this study.

Results indicate that less hazardous chemicals can increase the tensile strength of flax and hemp fibres to a greater extent than the common treatment using NaOH, while also reducing the variability in their properties. Although the increase in the elastic modulus was not interesting for this particular study, these findings may be interesting for reinforcing composites that do not require a brittleness analysis.

The effects of an alkaline solution in the tensile strength and elastic modulus of the fibres were also assessed, and the results indicate that the treated vegetable fibres would still have their properties affected in an alkaline environment, such as a concrete matrix. After degradation, results indicate that treated flax fibres would be susceptible to a higher variation of properties in comparison to untreated fibres, presenting also higher stiffness. On the other hand, the results obtained for hemp fibres had lower variability between untreated and treated fibres after exposed to an alkaline environment. Although this analysis was conducted on the fibres, concrete mixes were produced to evaluate their effects in comparison to concrete reinforced with untreated fibres.

Finally, from the optimised fibre reinforced concrete mixtures produced, it was found that a mixture reinforced with 0.25% of treated flax was able to achieve the designed compressive strength values, surpassing the mix reinforced with 0.5% of treated hemp fibres. The workability of fibre reinforced concrete remained low (slump zero) with a reduction of the maximum aggregate size and the addition of plasticiser. However, with the reduction of the flax fibre content from 0.5% to 0.25% per volume, it was possible to obtain slump values up to 23mm.

The addition of fibres was able to increase the fracture tensile strength and reduce the concrete elastic modulus at 28 days. Although at least 50% higher than the control, the mix containing treated vegetable fibres presented significantly lower values of fracture energy compared to the mixture reinforced with basalt fibres. Both flax and hemp fibre-reinforced concrete mixes, at 28 days, presented the lowest stiffness among the mixtures studied - 72% of the control compared to 82% of the control, obtained for basalt FRC.

Positively, both FRC using surface-treated fibres presented increased residual tensile strength. Comparing results from the preliminary analysis and the optimised mixes, increased residual tensile strength was observed for both hemp and flax FRC after surface treatment. Similarly, the addition of fibres in the mixes enhanced the thermal conductivity, and it was observed that the concrete density was reduced according to the volume of flax fibres added.

As expected, the addition of fibres increased the water penetration compared to plain concrete. However, the mixes with 0.25% of flax fibres presented a similar level of water penetration to the control mix, indicating that at this proportion, the cement paste would be able to coat the fibres, reducing the water infiltration.

Finally, from SEM images taken from concrete mixes reinforced with treated hemp and flax fibres at 30 days in comparison to mixes after over 90 days, it was possible to observe a reduction in the fibre content for both fibres, indicating continuous degradation over time, even after the surface treatments were conducted.

Overall, although the high variability in the properties of the treated fibres was still observed, in the concrete matrix, they presented a more consistent behaviour with a reduced standard deviation observed on the duplicated samples produced with three specimens each. There are certainly still opportunities for improvement, and pertinent suggestions for future work are presented in the section ahead.

## 5.2 Future Work

In this study, the suitability of using flax and hemp fibres as vegetable fibre reinforcement for concrete was investigated. Positive results were obtained compared to concrete reinforced with untreated fibres. However, it was not possible to predict the behaviour of the fibres from the surface treatment alone, and further studies are necessary to assess this in depth. A mathematical correlation between the results obtained from the fibre treatments would allow for the estimation of the behaviour of the fibre-reinforced concrete (FRC) produced, creating a reliable parameter to be used during the mixture design method.

Apart from the surface treatments adopted, the treatment using EDTA for 4 hours also presented interesting results for flax fibres and could be a promising treatment for fibres added to the concrete mixture. According to Le Troedec et al. (2008), EDTA treatment separates complex calcium ions and fibres linked with pectin. Instead of coating the fibres, their impact on a molecular level could positively affect the concrete properties.

Finally, an applied study could be conducted with the mixes designed during this work, with the maximum aggregate size reduced to 10mm. Considering the possibility of water penetration, the impact and abrasion resistance of thinner flooring layers or masonry blocks and panels for internal use could also be interesting applications for this material. Another alternative, particularly for the mixture containing 0.25% of treated flax fibres with stearic acid for 4 hours, could be their usage on 3D printers, which has become popular and feasible for the construction industry.

# Chapter Six

# References

# Chapter 6: References

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# Appendices

# Appendix A – Mechanical properties of singular fibres Algorithm

```
%This is a simplified algorithm to calculate the tensile strength,
%elastic modulus and %elongation at break for fibres.
%Do not use this without authorization.
%Author: Ana Caroline da Costa Santos

clc;
clear all;
close  all;


files = dir('*.xlsx');


% List all files from the directory
answers = zeros(length(files):12);
% Matrix base for the results
names = strings(length(files):1);
% String base for the name results (1st column)


maxloadanswers=zeros(length(files),18);

maxtanswers=zeros(length(files),18);

deltalanswers=zeros(length(files),18);

elasticmodeaswrs=zeros(length(files),18);

elbreakawrs=zeros(length(files),18);

numfiles=length(files);
mydata=cell(1,numfiles);

 for k = 1:length(files) %Loop

name = files(k).name;
opts = detectImportOptions(name);
opts = setvartype(opts,'char');

mydata{:,k}=readtable(name,opts);

% T = readtable(name);
mydata{:,k}=table2array(mydata{:,k}(:,:));
T=str2double(mydata{:,k}(:,:));

T(isnan(T))=0;
```

```
[rows,cols]=size(T);


diameter=[];
area=[];
tensile=[];
counterdiam=1;


    for  i = 3:3:cols
    diameter(counterdiam)= T(1,i);
    area(counterdiam,:)= (diameter(counterdiam)^2*pi)./4;

     counterdiam= counterdiam+1;


    end
        x=1;
        load=[];
        lmax=[];
    for  n = 1:3:cols

        load(:,x)=T(:,n);

        lmax(x,:)= max(load(:,x));
        x=x+1;

    end

        for j=1:length(area)
           for l=1:rows

                 tensile(l,j)= load(l,j)/area(j,:);


            end

        end

    [trows, tcols]= size(tensile);
    td=zeros(rows,length(area)+length(area));

    %tenslie vs extension

    extension=T(:,2:3:end);
    td(:,1:2:end)=tensile(:,:);
    td(:,2:2:end)=extension(:,:);
    plot(td);


    tmax=[];
    counter=1;
    deltal=[];
```

```
    elbreak=[];
    diam=[];
    dd=[];

    [tdrows,tdcols]=size(td);
    for  i = 1:2:tdcols

     tmax(counter,:)= max(td(:,i));

    [X Y]=find(tmax(counter,:)==td(:,i));

    deltal(counter,:)=td(X(length(X)),i+1);

    elbreak(counter,:)=  deltal(counter,:)/40;
    dd(counter,:)=  diameter;

    diam=  mean(diameter);


    [maxrows,maxcols]=size(tmax);

    counter= counter+1;

    end


    elasticmode=[];

    counterem=1;
    [erols, ecols]= size(load);


        for  m = 1:1:ecols

         elasticmode(counterem,:)=
(lmax(counterem,:)*0.04/((area(m,:)/1000000)*(deltal(counterem,:)/1000
)));


         counterem =  counterem + 1;

        end

    maxtanswers(k,1:maxrows)= tmax';
    deltalanswers(k,1:maxrows) = deltal';
    elasticmodeaswrs(k,1:maxrows) = elasticmode';
    elbreakawrs(k,1:maxrows) = elbreak';
    ddawrs(k,1:maxrows) = diameter';
    diamawrs(k,1,1) = diam';

    names(k,:) = name;

end
```

```
result =  [names maxtanswers]

result2 =  [names deltalanswers]

result3 = [names elasticmodeaswrs]

result4 = [names elbreakawrs]

result5 = [names ddawrs]


Final = [result result2 result3 result4 result5]
```

# Appendix B – $W_0$ used to calculate Gf in Equation 2.5

```
%This is a simplified algorithm to calculate W0 for fracture energy of
FRC.
%Do not use this without authorization.
%Author: Ana Caroline da Costa Santos


clc
clear all



files=dir('*.xlsx');
% List all files from the diretory
answers = zeros(length(files):3);
% Matrix base for the results
names = strings(length(files):1);
% String base for the name results (1st column)



for k = 1:length(files)
%Loop

name = files(k).name;
T = readtable(name);
T(1,:)=[ ];
T=T(:,[1:6]);


matrix=table2array(T);

x1=matrix(:,1);
x1=str2double(x1'); %converts text into a string that represents real
or complex numeric values
x1=x1(~isnan(x1)); %removes all cells containing NaN values

y1=matrix(:,2);
y1=str2double(y1');
y1=y1(~isnan(y1));

da=max(y1);
db=find(y1==da);
nx1=x1(1,1:db);
ny1=y1(1,1:db);
plot(nx1,ny1);
w01=trapz(nx1,ny1);  %Area under curve

x2=matrix(:,3);
x2=str2double(x2');
x2=x2(~isnan(x2));
```

```
y2=matrix(:,4);
y2=str2double(y2');
y2=y2(~isnan(y2));

da=max(y2);
db=find(y2==da);
nx2=x2(1,1:db);
ny2=y2(1,1:db);
plot(nx2,ny2);
w02=trapz(nx2,ny2);

x3=matrix(:,5);
x3=str2double(x3');
x3=x3(~isnan(x3));

y3=matrix(:,6);
y3=str2double(y3');
y3=y3(~isnan(y3));

da=max(y3);
db=find(y3==da);
nx3=x3(1,1:db);
ny3=y3(1,1:db);
plot(nx3,ny3)
w03=trapz(nx3,ny3);

answer = [w01,w02,w03];

answers(k,:) = answer;
names (k,:) = name;

end

answers;

result = [names answers]
```

# Appendix C – Algorithm for calculation of the Ci for Equation 2.7

```
%This is a simplified algorithm to calculate the Ci value used to
obtain the elastic modulus of FRC according to EN14651.
%Do not use this without authorization.
%Author: Ana Caroline da Costa Santos

clc
clear all



files=dir('*.xlsx');
% List all files from the diretory
answers = zeros(length(files):3);
% Matrix base for the results
names = strings(length(files):1);
% String base for the name results (1st column)
b=0.15;
d=b;
a0=0.025;
A=b*(d-a0);
L=0.5;
%Dimensions of the specimen

for k = 1:length(files);
%Loop

name = files(k).name;
T = readtable(name);
T(1,:)=[ ];
T=T(:,[1:6]);

matrix=table2array(T);

x1=matrix(:,1);
x1=str2double(x1'); %converts text into a string that represents real
or complex numeric values
x1=x1(~isnan(x1)); %removes all cells containing NaN values
x1=x1/L;
%dL/L (strain formula)

y1=matrix(:,2);
y1=str2double(y1');
y1=y1(~isnan(y1));
y1=y1/A;
%F/A Stress formula

da=max(y1);  %limit graph to max Y
db=find(y1==da);
dc=gradient(x1,y1) %limit graph to inflection point
dd=find(x1==dc)
nx1=x1(1,dc:db);
ny1=y1(1,dc:db);
plot(nx1,ny1); %plots graph from zero to max value
```

```
p = polyfit(nx1,ny1,1);
f = polyval(p,nx1);
plot(nx1,ny1,'m',nx1,f,'-');
legend('data','best  fit line');
g=[nx1 f];
h=gcf
exportgraphics(h,'name.png','Resolution',300)


Ci1=p(1);

x2=matrix(:,3);
x2=str2double(x2'); %converts text into a string that represents real
or complex numeric values
x2=x2(~isnan(x2)); %removes all cells containing NaN values
x2=x2/L;

y2=matrix(:,4);
y2=str2double(y2');
y2=y2(~isnan(y2));
y2=y2/A;

da=max(y2);
db=find(y2==da);
nx2=x2(1,1:db);
ny2=y2(1,1:db);
plot(nx2,ny2); %plots graph from zero to max value

p = polyfit(nx2,ny2,1);
f = polyval(p,nx2);
plot(nx2,ny2,'m',nx2,f,'-');
legend('data','best  fit line');


Ci2=p(1);


x3=matrix(:,5);
x3=str2double(x3'); %converts text into a string that represents real
or complex numeric values
x3=x3(~isnan(x3)); %removes all cells  containing NaN values
x3=x3/L;

y3=matrix(:,6);
y3=str2double(y3');
y3=y3(~isnan(y3));
y3=y3/A;

da=max(y3);
db=find(y3==da);
nx3=x3(1,1:db);
ny3=y3(1,1:db);
plot(nx3,ny3) %plots graph from zero to max value
```

```
p = polyfit(nx3,ny3,1);
f = polyval(p,nx3);
plot(nx3,ny3,'m',nx3,f,'-');
legend('data','best  fit line');


Ci3=p(1);



answer = [Ci1,Ci2,Ci3];

answers(k,:) = answer;
names (k,:) = name;

end

answers;

result = [names answers]
```

# Appendix D – Descriptive analysis of tested fibres

*Table D.1 – Descriptive analysis of basalt fibres.*

| ***Parameter*** | ***Diameter*** | ***Tensile Strength*** | ***Elastic Modulus*** | ***Elongation at break*** |
|---|---|---|---|---|
| Mean | 14.01 | 2545.68 | 136.18 | 0.01723 |
| Standard Error | 1.24 | 235.64 | 26.25 | 0.00219 |
| Median | 12.50 | 2680.54 | 110.53 | 0.01818 |
| Standard Deviation | 6.99 | 1268.96 | 141.36 | 0.01218 |
| Sample Variance | 48.88 | 1610266.99 | 19983.09 | 0.00015 |
| Kurtosis | 24.24 | -0.62 | 7.89 | -0.72710 |
| Skewness | 4.66 | 0.11 | 2.65 | 0.32942 |
| Range | 40.00 | 4859.27 | 660.02 | 0.04199 |
| Count | 32 | 29 | 29 | 31 |
| Confidence Level(95.0%) | 2.52 | 482.69 | 53.77 | 0.00447 |

*Table D.2 – Descriptive analysis of flax fibres.*

| ***Parameter*** | ***Diameter*** | ***Tensile Strength*** | ***Elastic Modulus*** | ***Elongation at break*** |
|---|---|---|---|---|
| Mean | 82.40 | 865.96 | 38.52 | 0.02087 |
| Standard Error | 7.31 | 70.11 | 4.19 | 0.00165 |
| Median | 74.65 | 891.50 | 38.50 | 0.02066 |
| Standard Deviation | 43.83 | 420.65 | 25.14 | 0.00990 |
| Sample Variance | 1921.41 | 176946.40 | 631.86 | 0.00010 |

| | | | | |
|---|---|---|---|---|
| Kurtosis | 9.72 | -0.44 | 0.00 | 0.45162 |
| Skewness | 2.69 | 0.17 | 0.34 | 0.17054 |
| Range | 238.40 | 1686.52 | 97.17 | 0.04643 |
| Count | 36 | 36 | 36 | 36 |
| Confidence Level(95.0%) | 14.83 | 142.33 | 8.51 | 0.00335 |

*Table D.3 – Descriptive analysis of hemp fibre.*

| ***Parameter*** | ***Diameter*** | ***Tensile Strength*** | ***Elastic Modulus*** | ***Elongation at break*** |
|---|---|---|---|---|
| Mean | 72.98 | 262.68 | 22.44 | 0.01514 |
| Standard Error | 4.77 | 24.24 | 3.81 | 0.00155 |
| Median | 68.70 | 236.11 | 13.70 | 0.01619 |
| Standard Deviation | 27.38 | 139.24 | 21.57 | 0.00877 |
| Sample Variance | 749.81 | 19386.88 | 465.16 | 0.00008 |
| Kurtosis | 3.05 | -0.47 | 1.99 | -0.63896 |
| Skewness | 1.35 | 0.46 | 1.59 | 0.19334 |
| Range | 135.20 | 573.29 | 86.76 | 0.03096 |
| Count | 33 | 33 | 32 | 32 |
| Confidence Level(95.0%) | 9.71 | 49.37 | 7.78 | 0.00316 |

*Table D.4 – Descriptive analysis of flax fibres for n=10.*

| ***Parameter*** | ***Diameter*** | ***Tensile Strength*** | ***Elastic Modulus*** | ***Elongation at break*** |
|---|---|---|---|---|
| Mean | 76.57 | 815.03 | 44.04 | 0.02082 |

| | | | | |
|---|---|---|---|---|
| Standard Error | 6.52 | 132.26 | 9.48 | 0.00382 |
| Median | 72.90 | 898.13 | 38.28 | 0.01976 |
| Standard Deviation | 20.62 | 418.24 | 29.99 | 0.01209 |
| Sample Variance | 425.09 | 174926.74 | 899.27 | 0.00015 |
| Kurtosis | 1.11 | -0.55 | 0.35 | 0.89670 |
| Skewness | 1.27 | -0.24 | 0.78 | 1.06890 |
| Range | 65.28 | 1342.00 | 95.10 | 0.03831 |
| Count | 10 | 10 | 10 | 10 |
| Confidence Level(95.0%) | 14.75 | 299.19 | 21.45 | 0.00865 |

*Table D.5 – Descriptive analysis of hemp fibres for n=10.*

| ***Parameter*** | ***Diameter*** | ***Tensile Strength*** | ***Elastic Modulus*** | ***Elongation at break*** |
|---|---|---|---|---|
| Mean | 77.29 | 254.47 | 23.25 | 0.01326 |
| Standard Error | 5.65 | 40.71 | 6.20 | 0.00240 |
| Median | 74.21 | 226.38 | 15.82 | 0.01641 |
| Standard Deviation | 17.86 | 128.75 | 19.59 | 0.00760 |
| Sample Variance | 318.90 | 16575.79 | 383.92 | 0.00006 |
| Kurtosis | -0.16 | 0.77 | 2.42 | -1.84147 |
| Skewness | 0.14 | 0.23 | 1.63 | -0.41408 |
| Range | 60.00 | 467.85 | 63.19 | 0.01964 |
| Count | 10 | 10 | 10 | 10 |
| Confidence Level(95.0%) | 12.77 | 92.10 | 14.02 | 0.00544 |

# Appendix E – Properties of flax fibres treated

| *Treatment* | *Duration* | *Flax* | | | |
|---|---|---|---|---|---|
| | | *Max Tensile* | *E* | *ε %* | *Diameter(μm)* |
| EDTA | 4h | 1582.61 | 46.34 | 3.10% | 64.07 |
| | 6h | 1140.33 | 87.52 | 1.40% | 70.20 |
| | 10h | 993.93 | 93.81 | 1.13% | 74.57 |
| | 15h | 1707.17 | 78.74 | 1.99% | 105.38 |
| | 24h | 1050.77 | 54.90 | 1.84% | 73.75 |
| KMnO4 | 4h | 629.79 | 18.54 | 4.45% | 73.64 |
| | 6h | 2366.94 | 138.43 | 1.56% | 47.30 |
| | 10h | 1272.23 | 81.08 | 1.38% | 46.68 |
| | 15h | 1280.52 | 77.35 | 1.38% | 62.36 |
| | 24h | 1899.16 | 116.92 | 1.54% | 57.45 |
| NaOH10% | 4h | 546.84 | 32.51 | 3.66% | 62.67 |
| | 6h | 460.61 | 36.18 | 1.39% | 54.40 |
| | 10h | 453.97 | 7.66 | 4.92% | 56.29 |
| | 15h | 299.90 | 8.28 | 3.95% | 65.62 |
| | 24h | 448.03 | 15.07 | 2.92% | 54.68 |
| NaOH15% | 4h | 238.23 | 34.48 | 1.80% | 65.45 |
| | 6h | 252.25 | 7.82 | 3.16% | 49.91 |
| | 10h | 278.60 | 4.70 | 5.86% | 57.37 |
| | 15h | 502.84 | 15.08 | 4.16% | 47.83 |
| | 24h | 374.45 | 6.41 | 6.06% | 57.83 |

| | | | | | |
|---|---|---|---|---|---|
| NaOH5% | 4h | 362.15 | 19.69 | 3.75% | 56.69 |
| | 6h | 231.92 | 4.53 | 4.92% | 44.99 |
| | 10h | 2153.71 | 143.74 | 1.82% | 49.80 |
| | 15h | 844.77 | 41.59 | 1.76% | 56.05 |
| | 24h | 1442.61 | 63.99 | 2.09% | 85.10 |
| Stearic Acid | 4h | 1739.80 | 53.59 | 3.13% | 44.82 |
| | 6h | 567.12 | 55.67 | 0.90% | 42.45 |
| | 10h | 2343.23 | 133.90 | 1.40% | 49.84 |
| | 15h | 760.82 | 45.19 | 1.73% | 48.46 |
| | 24h | 2078.83 | 117.02 | 1.48% | 36.32 |

# Appendix F – Properties of treated hemp fibres

| *Treatment* | *Duration* | *Hemp* | | | |
|---|---|---|---|---|---|
| | | *Max Tensile* | *E* | *ε %* | *Diameter (µm)* |
| EDTA | 4h | 227.43 | 32.24 | 1.59% | 67.08 |
| | 6h | 384.18 | 28.54 | 1.40% | 68.09 |
| | 10h | 1195.93 | 86.21 | 1.31% | 83.99 |
| | 15h | 539.46 | 52.19 | 1.16% | 93.96 |
| | 24h | 852.23 | 123.78 | 1.10% | 89.19 |
| KMnO4 | 4h | 364.55 | 29.32 | 1.24% | 63.57 |
| | 6h | 552.33 | 36.53 | 1.64% | 57.97 |
| | 10h | 852.15 | 68.85 | 1.18% | 52.52 |
| | 15h | 859.39 | 80.73 | 0.99% | 68.24 |
| | 24h | 910.84 | 79.49 | 1.26% | 89.02 |
| NaOH10% | 4h | 153.44 | 8.61 | 1.90% | 68.34 |
| | 6h | 157.46 | 11.79 | 1.55% | 61.97 |
| | 10h | 223.46 | 10.48 | 2.21% | 45.11 |
| | 15h | 95.46 | 7.11 | 1.38% | 46.64 |
| | 24h | 367.11 | 19.30 | 2.03% | 53.57 |
| NaOH15% | 4h | 101.89 | 7.58 | 1.69% | 70.66 |
| | 6h | 98.71 | 3.89 | 2.73% | 42.53 |
| | 10h | 80.76 | 3.16 | 2.92% | 54.94 |
| | 15h | 109.14 | 6.03 | 2.08% | 75.23 |
| | 24h | 128.73 | 8.21 | 1.81% | 41.84 |

| | | | | | |
|---|---|---|---|---|---|
| NaOH5% | 4h | 232.66 | 15.58 | 1.51% | 87.52 |
| | 6h | 242.85 | 15.79 | 1.45% | 78.91 |
| | 10h | 818.73 | 72.17 | 1.12% | 69.20 |
| | 15h | 505.54 | 33.05 | 1.35% | 60.79 |
| | 24h | 418.51 | 27.34 | 1.48% | 28.76 |
| Stearic Acid | 4h | 389.84 | 29.43 | 1.23% | 38.07 |
| | 6h | 337.63 | 25.46 | 1.38% | 50.46 |
| | 10h | 819.83 | 68.10 | 1.11% | 47.61 |
| | 15h | 372.10 | 32.97 | 1.06% | 57.80 |
| | 24h | 1694.40 | 112.03 | 1.43% | 32.34 |