Influencia de la adición de la fibra de abacá en las características de los aglomerados de cemento y su comportamiento a largo plazo

TESIS DOCTORAL

Memoria presentada por

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List of Publications

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Acronyms and notations

ANOVA	Analysis of variance.
ASTM	American Society for Testing and Materials.
CH_4	Methane.
CO	Colombia.
CO_2	Carbon dioxide.
CrI	Crystalline index.
C-S-H	Calcium silicate hydrate.
CT	Conventional treatment with a single use of NaOH solution.
DTG	First derivative of the TG curve.
EC	Ecuador.
ESPAC	Ecuadorian survey of surface and continuous agricultural production.
FR	Reference abaca alkali treated fiber.
$Frac_{LEACH}$	Partitioning factor for the fraction of fertiliser and manure nitrogen applied to soil that is lost through leaching and runoff.
FT-IR	Fourier transform-infrared spectroscopy.
FWD-1	Alkali treated fiber subjected to WD-1.
FWD-2	Alkali treated fiber subjected to WD-2.
GLO	Global.
GU	General Use.
GWP	Global warming potential.
HR	Hornification.
HS	NaOH-3%
IN	India.
INAMHI	Ecuadorian Institute of Meteorology and Hydrology.
INEN	Ecuadorian Institute of Standardization
IPCC	Intergovernmental Panel on Climate Change.
ISO	International Standards Organization.
JSCE	Japan Society of Civil Engineers.
kg CO_2 eq.	Kilograms of carbon dioxide equivalents.
LCA	Life cycle assessment.
LCI	Life cycle inventory.

LCIA Life cycle impact assessment.

LS Natural latex + silica fume.

MFTHS Alkali treated fiber reinforced mortar.

MSF mortar with no fiber.

n number of samples per treatment.

N_2O Nitrous oxide.

NAF Natural abaca fiber.

NaOH Sodium hydroxide.

NT No treated fiber.

P *P*-value.

PCR Product category rules.

PE Peru.

PPF Polypropylene fiber.

RM Reference Mortar.

RoW Rest of world.

RT Reuse of NaOH solution per 8 cycles of recirculation.

SEM Scanning electron microscope.

T Temperature.

T0 No treatment cycle performed.

T1 Single-use of NaOH solution

T2-T10 Reuse of NaOH solution, from second use to tenth use.

TAF-CT Treated abaca fiber - conventional treatment.

TAF-RT Treated abaca fiber - reuse treatment.

TG Thermogravimetric curve.

TGA Thermogravimetric analysis.

UNE Spanish Association for Standardization.

US United States.

w/d wet/dry.

WD-1 Aging process of wet and dry 1, according to Neves Junior, Ferreira, et al., (2019).

WD-2 Aging process of wet and dry 2 according to Wei et al., (2016).

XRD X-ray diffraction.

Contents

Abstract

Natural fibers have been commonly utilized by researchers as an alternative to steel fibers or synthetic fibers in composite materials. The use of plant-based natural fibers and vegetable waste has garnered global attention due to their affordability, abundance, and environmental friendliness. Among the analyzed natural fibers are coconut, sisal, jute, hemp, and curaua; due to their wide availability, especially in tropical countries, these fibers are cost-effective and possess the significant advantage of being renewable.

Abaca fiber is derived from the pseudostems of *Musa textilis* and is commonly known as Manila hemp. In tropical countries like the Philippines, abaca plants are abundantly found, making the country the largest global exporter, followed by Ecuador, which accounts for 15% of production. Abaca fiber is the first natural fiber to meet the quality requirements for composites used in vehicle exteriors. Since abaca is produced in Ecuador, this fiber was employed to determine its influence on the properties of cement composites in both short-term and long-term assessments. This research focused on analyzing the addition of abaca fiber in mortars used for masonry (coating).

The research is divided into three parts:

The first part involved determining the optimal size, dosage, and treatment available for incorporating abaca fiber into cement mortar. It was established that the mortar to be analyzed would be the typical mixture used in Ecuador, characterized by a cement-to-sand ratio of 1:3. In this phase, three types of fiber treatments were applied: a sodium hydroxide solution at a concentration of 3%, silica fume combined with natural latex, and heat treatment, utilizing fiber sizes of 20, 25, 30, and 35 mm, and dosages of 0.2%, 0.3%, and 0.4%. Thermogravimetric analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FT-IR), and tensile testing were performed on the fiber to determine the optimal performance. For the mortar, the sodium hydroxide treatment enhanced the fiber behavior and was incorporated into the study mortar, assessing its flexural strength and setting time.

The second part of the investigation focused on determining the short-term and long-term mechanical behavior through accelerated aging processes of the mortar reinforced with sodium hydroxide-treated abaca fiber, at a concentration of 3%, with a size of 30 mm and a dosage of 0.2% by total solids weight. After 28 days, the following properties were analyzed: dry bulk density, flexural strength, compressive strength, tensile strength, and shrinkage. The samples underwent two types of accelerated aging through wet/dry cycles, and the same mechanical properties were assessed, in addition to SEM and FT-IR analyses of the treated abaca fiber post-aging.

The third part of the investigation entailed the characterization and life cycle assessment of sodium hydroxide-treated abaca fiber, reusing the treatment solution. This segment was developed due to the significance of sustainability and the assessment of whether the treated plant fiber is environmentally friendly. A study was conducted on the effects of reusing this solution analyzing abaca fibers through SEM, TGA, and tensile testing, enhancing the inventory database and determining the carbon footprint of the treated abaca fiber in comparison with polypropylene fiber. The results indicate that reusing the treated water is effective up to the eighth cycle, with a 25% reduction in carbon footprint, thereby contributing to the assessment of the treatment's effectiveness and its incorporation in masonry mortars.

Resumen

Las fibras naturales han sido comúnmente usadas por los investigadores como una alternativa a las fibras de acero o fibras sintéticas en materiales compuestos. El uso de las fibras naturales de plantas y desperdicios vegetales ha atraído la atención global debido a su asequibilidad, abundancia y la amigabilidad con el medio ambiente. Entre las fibras naturales que han sido analizadas se encuentran las de coco, sisal, yute, cáñamo, curaua; debido a su amplia disponibilidad, especialmente en los países tropicales, estas fibras tienen un costo bajo y presentan la gran ventaja de ser renovables.

La fibra de abacá se obtiene del seudo tallo de la musa textiles y se conoce comúnmente como cáñamo de Manila. En países tropicales como Filipinas, las plantas de abacá se encuentran en abundancia siendo el mayor exportador mundial, seguido de Ecuador con el 15% de la producción. La fibra de abacá es la primera fibra natural en cumplir con los requerimientos de calidad para compuestos usados en los exteriores de vehículos. Debido a que el abacá se produce en Ecuador, se utilizó esta fibra para determinar la influencia que tendría su adición en las características de aglomerados de cemento a corto y largo plazo. Esta investigación se enfocó en analizar la adición de la fibra de abacá en morteros usado para albañilería (revestimiento)

La investigación está dividida en tres partes:

La primera consistió en determinar el tamaño, dosificación y tratamiento óptimo disponible para incorporar la fibra de abacá en el mortero de cemento. Se determinó que el mortero a analizar sería el típico trabajado en Ecuador el cual tiene una relación 1:3 cemento: arena. En esta parte de la investigación se usaron 3 tipos de tratamientos a la fibra: solución de hidróxido de sodio en una concentración del 3%, humo de sílice + látex natural y hornificación, tamaños de fibra de 20-25-30 y 35mm y dosificación de 0.2-0.3 y 0.4%. Ensayos de Termogravimetría, X-RD, SEM, FT-IR y tensión fueron realizados a la fibra para determinar el mejor comportamiento. En cuanto al mortero, el tratamiento de hidróxido de sodio mejoró el comportamiento de la fibra y fue adicionado al mortero de estudio, analizando su flexotracción y tiempo de fraguado.

La segunda parte de la investigación consistió en determinar el comportamiento mecánico a corto y largo plazo mediante procesos acelerados de envejecimiento de ese mortero reforzado con fibra de abacá tratada con hidróxido de sodio en una concentración del 3%, en un tamaño de 30mm y una dosificación del 0.2% del peso total de sólidos. A 28 días se analizó: dry bulk density, flexión, compresión, tracción y shrinkage. Se sometieron las muestras a dos tipos de envejecimiento acelerados por procesos de Wet/Dry a las mismas propiedades mecánicas y adicional se hicieron ensayos a la fibra de abacá tratada y luego de someterse a los procesos de envejecimiento de SEM y FT-IR.

La tercera parte de la investigación consistió en la caracterización y evaluación del ciclo de vida de la fibra de abacá tratada con hidróxido de sodio, reusando dicha solución en el tratamiento de la fibra. Esta parte se desarrolló por la importancia que tiene el tema de sostenibilidad y si efectivamente la fibra vegetal tratada es amigable con el medio ambiente. Para lo cual se hizo un estudio del efecto de reusar esta solución, analizando las fibras de abacá mediante SEM, TGA, tracción, se aumentó la base de dato de inventario y se determinó la huella de carbono de la fibra de abacá tratada comparándola con fibra de polipropileno. Los resultados muestran que reusar el agua tratada es efectiva hasta el octavo ciclo, en términos de huella de carbono se reduce a un 25%, con lo cual esta investigación aporta hasta el ciclo de efectividad del tratamiento y su incorporación en morteros de albañilería.

Chapter 1. Introduction

1.1. Introduction

The use of natural plant fiber as reinforcing agents in composite materials has attracted widespread scientific and industrial interest over the past decade. This interest originates from the natural abundance an low cost of such fibers, as well as their properties (Cai et al., 2016). Natural fibers, such as sisal, jute, cotton, flax, hemp, kenaf, have already been considered as potential alternatives to traditional fibers (steel fiber, polymer fibers, glass fiber) given their environmental friendliness and availability (Wei & Meyer, 2017). Natural fibers can considerably improve the tension performance, flexural properties, crack resistance, and fatigue behavior of such composites (Ganapathy et al., 2019; Stapper et al., 2021; D. Zhu et al., 2023).

Abaca fiber is obtained from the pseudo-stem of *musa textile* which is commonly used as Manila Hemp. Philippines is the largest producer of abaca, followed by Ecuador with the 15% of the worldwide production (Richter et al., 2013). Therefore, this natural fiber was considered among this research. Despite all these advantages, there are concerns that arises when mixed in cementitious mortar, the durability is one of the biggest concerns (Boulos et al., 2019). Hence, two strategies can be pursued for counteracting these concerns, the first one consists on modifying the matrix (X. Li et al., 2007) and the second focuses on modifying the natural fiber itself through chemical, or physical modification in order to increase its stability in cementitious matrices (Z. Zhao et al., 2018).

Considering these two strategies, the one focusing on modifying the fiber have been more used (Ferreira et al., 2015). Treatments consisting on dipping fiber into solution of sodium hydroxide corresponding to alkali treatment have been widely used (Ferreira et al., 2015; E. J. da Silva et al., 2017; Sood & Dwivedi, 2018), hornification (Cid et al., 2020; Ferreira et al., 2017), and others have been used as well (Boulos et al., 2017, 2019).

The treatments modify the fiber itself, it has been proved through TGA, SEM, XRD that the treated fiber shows a variation on its natural composition of pectin, cellulose, hemicellulose and lignin (Boulos et al., 2019), improving the bonding between the fiber and the cementitious matrix (Morton et al., 2010; Oushabi et al., 2017).

Despite all these advantages, the most critical concern is the low durability of fibers, knowing that natural fiber durability depends on the durability of constituent exposed to stress. Hence, deterioration of natural fibers in a cementitious matrix has emerged as the major problem to be solved in order to produce durable natural fiber reinforced cement composites (Arshad et al., 2020). Therefore, methods such as accelerated process of wet/dry could be used as well for estimating the degradation of the material, this remarks an important decision whether to use or not natural fibers in the composite and analyze if after some time, it still contributes to the mechanical properties of the mortar.

As Poletanovic et al., (2021) described in her hemp study subjected to alkali treatment where 10 cycles of w/d alternatively used showed an increase of 20% approximately of compressive and flexural strength compared to reference mortar. Processes of w/d described by Neves Junior, Ferreira, et al., (2019) and Wei et al., (2016) have been used as well for determining the degradation of the material. These processes simulate severe and medium condition of affectation, the main reason to look for this type of process is the natural condition of Ecuador where only two seasons exist: dry and rainy, hence this process results in the most severe exposition for mortars.

The composites are analyzed at 28 days through dry bulk density, flexural strength, compressive strength, dry shrinkage and tensile strength. Then samples are subjected to processes of w/d through the same mechanical properties in order to compare the behavior and results of the composite.

After determining the characteristics of the bio-composite it is important to analyze as well the use of NaOH and whether it contributes positively to the environment. Knowing that NaOH is one of the most widely used chemical treatments for natural fibers (Maiti et al., 2022) where fibers are washed with water to remove any residual alkaline content and then neutralized to prevent further degradation, after this fibers are dried at room temperature or in ovens (Iucolano et al., 2015). However, this procedure can lead to a significant consumption of NaOH, which not only affects production costs but also raises environmental concerns. The quantity of NaOH required per kilogram of natural fiber depends on the solution concentration and fiber type. For example, low-concentration NaOH solutions (1 %) consume 0.15–0.20 kg of NaOH per kg of natural fiber. At common concentrations (3–5 %), NaOH consumption ranges between 0.44 and 1.90 kg of NaOH per kg of natural fiber, while at high concentrations of NaOH (10–50 %), up to 6.67 kg of NaOH per kg of natural fiber may be required.

Reusing NaOH solutions in different applications has proven to be effective in enhancing material properties and offering some environ- mental benefits. In one approach, sisal fibers treated with 10 % NaOH and incorporated into alkali-activated slag-based composites demonstrated improved flexural and compressive strengths with reduced electrical conductivity and density using the residual NaOH solution as an activator (X. Zhao et al., 2024). Another method involves residual bagasse fibers for use in the production of fiber-reinforced bio-composite pellets, where a NaOH recovery process allows for the recycling of up to 61 % of the NaOH used, although the reduction in carbon footprint is 1 % (Ita-Nagy et al., 2020) . For these reasons, circularity measures, such as reducing chemical consumption and wastewater generation, can be implemented to mitigate the environmental impacts associated with the production of treated natural fibers. Several researchers have proposed the use of life cycle assessments (LCA) to assess the effectiveness of cleaner production methods and circular economy alternatives (Yaseen et al., 2024).

All these concerns justify the need of investigating the incorporation of treated abaca fibers in cementitious mortar, its mechanical properties in short and long term, the appropriated dosage, length, size of abaca fiber, and whether it is a green solution and if quantitatively it represents a better option over polypropylene which is one of the most used fibers in construction.

1.2. Objectives

The main objective of this thesis is to analyze the influence of the addition of abaca fiber on the characteristics of cement agglomerates and their long-term behavior.

In order to fulfill the main objective, the following secondary objectives were performed:

I. To analyze the effect of three different treatments on abaca fibers used in cementitious mortars, and how the size and dosage of abaca fibers influence the behavior of the cementitious composite.

II. To compare the mechanical behavior of a mortar with no fiber vs. an alkali treated fiber reinforced mortar applied to masonry in construction industry as well as the mechanical properties of these mortars when subjected to accelerated aging process of w/d

III. To study the effect of recirculation and reuse of NaOH solution for the treatment of natural fibers generating life cycle inventories for the treatment process of natural fibers with NaOH solution as well as quantifying and evaluation the carbon footprint on natural and treated abaca fiber besides synthetic fiber.

Chapter 2. Methodology

2.1. Methodology

The methodology employed in this study is summarized in Figure 2.1. The first phase involved a comprehensive literature review focusing on natural fibers, including their characterization, various treatments, dosages, and lengths utilized in cementitious matrices. Additionally, the review covered the properties of bio-composites, natural and accelerated aging processes of natural fibers, life cycle assessments, and strategies for reusing solutions to reduce carbon footprints. Insights from this literature review informed the experimental design and analysis.

To characterize treated and untreated abaca fibers, a selection of treatment methods was determined based on the literature review and the availability of materials in Ecuador. The treatments applied included hornification, sodium hydroxide (NaOH) at a 3% concentration, and a combination of latex and silica fume. These treatments were evaluated through X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and tensile strength testing. Among the treatments, the sodium hydroxide method yielded the most favorable results.

Following the identification of the optimal treatment for abaca fibers, a reference mortar composition was established based on traditional plaster mortar formulations commonly used in Ecuador, adhering to ASTM and NEC 2015 standards. Experimental mortar samples incorporating treated abaca fibers were prepared with varying fiber dosages (0.2%, 0.3%, 0.4%) and fiber lengths (20 mm, 25 mm, 30 mm, 35 mm). These samples were subjected to flexural and compressive strength tests, which determined the optimal composition: mortar reinforced with sodium hydroxide-treated abaca fibers at a 0.2% dosage and a 30 mm fiber length.

The resulting bio-composite was further characterized by comparing it with a reference mortar (without fiber addition). Comparative tests included evaluations of dry bulk density, flexural strength, compressive strength, tensile strength, and shrinkage. Durability emerged as a critical factor requiring further investigation. Due to time constraints, natural aging could not be pursued; instead, accelerated aging processes were employed to simulate Ecuadorian environmental conditions, including wetting and drying cycles representative of local soil, rainfall, and drying seasons. The aged samples underwent the same characterization tests, and the results were recorded.

The sodium hydroxide-treated abaca fiber-reinforced mortar demonstrated potential as an environmentally friendly bio-composite. However, its performance was benchmarked against polypropylene fiber, a widely used synthetic alternative in construction. A life cycle assessment (LCA) was conducted to quantify the sustainability of the materials in terms of their carbon footprint.

Finally, the study evaluated the reuse potential of the sodium hydroxide solution used in fiber treatment. Three types of abaca fiber samples were tested: untreated natural fiber, treated fiber with conventional sodium hydroxide treatment, and treated fiber using a reused NaOH solution.

The reused solution was applied for up to 10 cycles, and its effectiveness was assessed using titration, TGA, SEM, and tensile strength analysis. These results, along with comparisons to polypropylene fibers, informed the LCA in terms of carbon footprint, underscoring the environmental advantages of the proposed bio-composite.

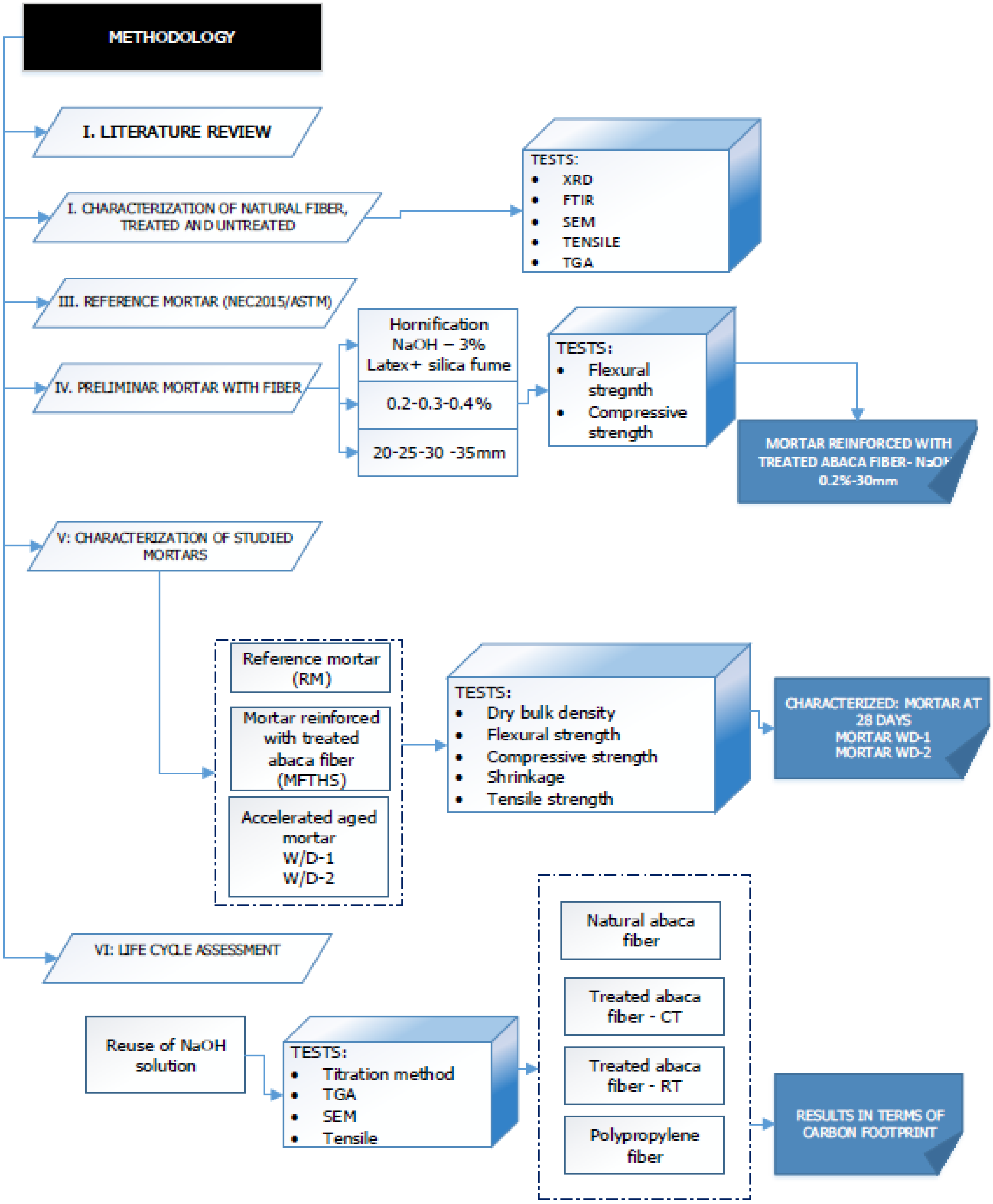

Figure 2.1. Methodology framework used in this study.

To achieve the proposed objective, this doctoral thesis has structured through the following compilation of publications:

1. The effect of different treatments on abaca fibers used in cementitious composites, published in Journal of Natural Fibers (Alcivar-Bastidas et al., 2023).

2. Effect of aging process on mechanical performance of reinforced mortar with NaOH abaca fibers, published in Case Studies in Construction Materials (Alcivar-Bastidas, Petroche, Cornejo, et al., 2024).
3. Characterization and life cycle assessment of alkali treated abaca fibers: the effect of reusing sodium hydroxide (Alcivar-Bastidas, Petroche, Ramirez, et al., 2024).

The first article analyzes the effect of different treatments on abaca fibers considering dosages and fiber length. Three different treatments were applied: hornification, dipping in 3% sodium hydroxide solution and water and natural latex+silica fume with different lengths (20-25-30-35mm) and dosages of 0.2%, 0.3% and 0.4%; in order to achieve the best treatment, length and dosage.

Treated and untreated fibers were subjected to Thermogravimetric analysis (TGA), X-ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FT-IR), tensile test. After analyzing all proposed treatments with dosages and length specified, conventional mortar for plastering used in Ecuador was designed with the following considerations 1:3 cement:sand.

In terms of treatment best results were achieved when applying alkali treatment with the 3% sodium hydroxide solution concentration. The conventional mortar was reinforced with this treated abaca fiber with different lengths and dosages subjected to flexural and compressive strength and best results were achieved with 30mm length and 0.2% dosage.

In the second article considering one of the major concerns within bio composites, its durability a comparative analysis through mechanical properties in traditional mortar MSF and treated abaca fiber mortar MFTHS were performed, comparing its dry bulk density, flexural, compressive, dry shrinkage and tensile strength. Its durability was analyzed after subjecting both type of samples to accelerated aging process of wet/dry. Treated abaca fibers subjected to both process of w/d were analyzed through SEM and FT-IR, in order to compare the internal composition of the fiber. The same mechanical properties were performed to the bio composite after subjecting samples to accelerated process of w/d.

This study resulted in the following conclusions:

- Despite the application of w/d cycles, alkali treated abaca fiber preserves the same mechanical composition.
- When correlating results from mechanical properties, bio composites with treated abaca fiber resulted in better outcomes.

Following the research topic where length, treatment, dosage, mechanical properties at 28 days and after applying accelerated aging process were defined; it was important to evaluate the effects of implementing a circular economy approach through the recirculation of a NaOH solution on the treatments of abaca fibers. It is important to remark that the abaca treatment involves the usage of this solution and after completing the treatment the residue is discarded. This waste water could be used "n" times until it loses its effectiveness. This study analyzed ten reused solutions until efficiency was lost, also polypropylene fiber was used as one of the main synthetic fibers used in construction. In order to measure the effectiveness of the treatment samples were subjected to SEM, TGA, tensile test and the titration method for measuring NaOH solution concentration. After determining the "n" effective treatment, life cycle assessment was performed to natural, treated abaca fiber and synthetic polypropylene fiber measuring the generated carbon footprint. In general terms carbon footprint results show the environmental advantages of abaca fiber over polypropylene, with notable reduction in carbon footprint and higher tensile strength.

Chapter 3.
The effect of different treatments on abaca fibers used in cementitious composites[1]

[1] The result presented in this chapter were published in Alcivar-Bastidas, S., Petroche, D. M., & Martinez-Echevarria, M. J. (2023). The effect of different treatments on abaca fibers used in cementitious composites. Journal of Natural Fibers, 20(1). https://doi.org/10.1080/15440478.2023.2177235

3.1. Introduction

During last years, natural fibers (such as flax, jute, sisal coir and bamboo fibers) have been extensively used as suitable reinforcement in cement-based composites (Benaimeche et al., 2018). Using fibers from natural plants has attracted greater global attention because of its affordability, abundance and eco-friendliness (Jiang et al., 2018). The advantages of using natural fibers in industrial applications relate to their light weight, low cost, nontoxicity, biodegradability and high specific stiffness (Cai et al., 2015).

Abaca fiber is obtained from the pseudo-stem of *Musa textile*. Which is commonly known as Manila hemp. This plant has little non-edible fruits full of seeds, its pseudo-stem can grow up to 6.5 m. In tropical countries like Philippines, abaca plants are available in abundance (Kumar Sinha et al., 2017). Worldwide, the demand of abaca is mainly fed by the Philippines with an 85% and Ecuador with a 15% (Richter et al., 2013). Certain authors have compared mechanical properties of natural fibers with promising results in terms of tensile strength abaca 400 MPa (Onuaguluchi & Banthia, 2016).

Despite all these advantages, many challenges arise concerning the use of natural fibers in cementitious applications, certain drawbacks could be mentioned as poor dispersion in cementitious matrix and probably the most critical, the low durability of fibers in cementitious matrix (Boulos et al., 2019). Therefore, in order to counteract these drawbacks, two strategies could be applied as a way of increasing the durability of the natural fibers in cement paste. The first strategy is to modify the composition of the cementitious matrix in a way that reduces its alkalinity in order to increase the durability of natural fibers in cement paste (X. Li et al., 2007). The second strategy focuses on the natural fiber itself and involves chemical or physical modification in order to increase its stability in cementitious matrices (Boulos et al., 2019; Jiang et al., 2018). This study focused on the second strategy by applying three different treatments on abaca fibers.

This work aims to (1) analyze the effect of three different treatments on abaca fibers used in cementitious mortars, (2) study how the size and dosage of abaca fibers influence the behavior of the cementitious composite. The abaca fibers were subjected to three different treatments: a process of hornification, dipping in 3% NaOH solution and water, and finally a coating of silica fume with natural latex. Scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform-infrared spectroscopy (FT-IR), Thermo gravimetric analysis (TGA), tensile test and flexural strength were used to analyze structural and chemical changes in the fibers before and after applying the treatment.

3.2. Material and methods

3.2.1. *Materials*

The abaca fibers used in this research correspond to a second quality fiber. This classification is based on the color and diameter of the fiber (Simbaña et al., 2020). The cement used is a general use (GU) type, according to ASTM C1157 (ASTM International, 2023b). The sand used in this study was collected from Rio Boliche (Figure 3.1) and its granulometric curve was set as the average between ASTM C144 maximum and minimum ranges (ASTM International, 2018).

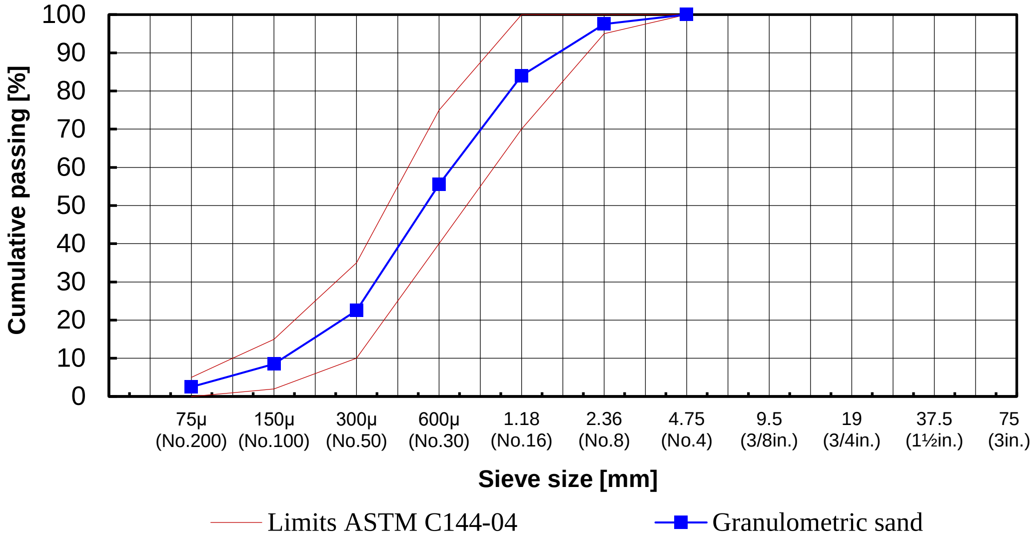

Figure 3.1. Granulometric curve of sand used in this research.

3.2.2. *Abaca treatments*

The length of the fibers were determined based on previous research and were cut into four different lengths: 20, 25, 30, 35 mm (Ferreira et al., 2018; Onuaguluchi & Banthia, 2016). The fibers were subjected to three different treatments: hornification, dipping in 3% NaOH solution and water, and a coating of silica fume with natural latex.

3.2.2.1. Hornification

The process was performed as described by Ferreira et al. (2017) with the main purpose of improving natural fiber properties: The abaca fibers were placed in a container with water (T = 22°C) during 3 hours. The drying process was carried out in a furnace at a temperature increasing temperature rate of 1°C/min to 80°C. Then this temperature was maintained for 16 hours. After 16 hours of drying, the furnace was cooled down to of 22°C in order to avoid possible thermal shock to the fibers. This procedure was repeated 5 times because at this point other natural fibers accomplished better performance and it has been proven that through this method fiber-matrix bonding also improves frictional mechanism (Ferreira et al., 2015, 2017).

3.2.2.2. Dipping in 3% sodium hydroxide solution and water

The process was performed by a procedure previously applied in leaf fibers according to Jiang et al., (2018). A solution of NaOH (3% mass percentage) and water was prepared, fibers were dipped into this solution for 4 hours and then in water for 10 hours. Impregnated abaca fibers were rinsed with water until extruded brown washing became transparent. Then, fibers were dried at 85±1°C for 24 hours in a furnace, the use of temperature led to increase the fiber stiffness and decrease its moisture absorption (Pacheco-Torgal & Jalali, 2011). Fibers were cooled to room temperature and then stored in sealed plastic bags for posterior usage.

3.2.2.3. Natural latex + Silica fume

Firstly, each abaca fiber was immersed in an adherent solution of natural latex for 1 min, according to Silva et al. (2017). At this point, the adherent solution surrounds the abaca fiber generating bonding layers. The silica fume particles act as fillers within the cementitious matrix, reduce the porosity of the composite by filling voids, form more C-S-H, it is also useful for strength development (Júnior Carvalho Machado et al., 2020).

3.2.3. Abaca fibers in cementitious mortars

The cement based mortar matrix consists of a cement to sand ratio 1:3 which corresponds to a typical mortar used in Ecuador (INEN 2615:2012) (INEN, 2015). The ratio between water-cement is fixed after performing a workability test with the flow table according to UNE 1015-3 (UNE, 2000) .

The dosage of fiber was applied in three different percentages 0.2%, 0.3% and 0.4% based on the total weight of solids based on previous literature review (Çomak et al., 2018; Onuaguluchi & Banthia, 2016; Wei et al., 2016), the mortar mixture composition is listed in Table 3.1.

Table 3.1. Mortar mix composition.

Materials	Dosage
Sand	1620 g
Cement	540 g
Water	Flow rate 110±5%
Abaca	0.2, 0.3, 0.4% total solid weight

Figure 3.2 describes the scheme performed in order to develop this research; where abaca fibers were fixed at 25 mm length with a 0.2% of the total solid weight and mixed into the mortar applying three different treatments to find out the optimum treatment (analyzing through flexural strength); once the treatment was determined the optimum length and dosage were analyzed for reaching the best length and dosage.

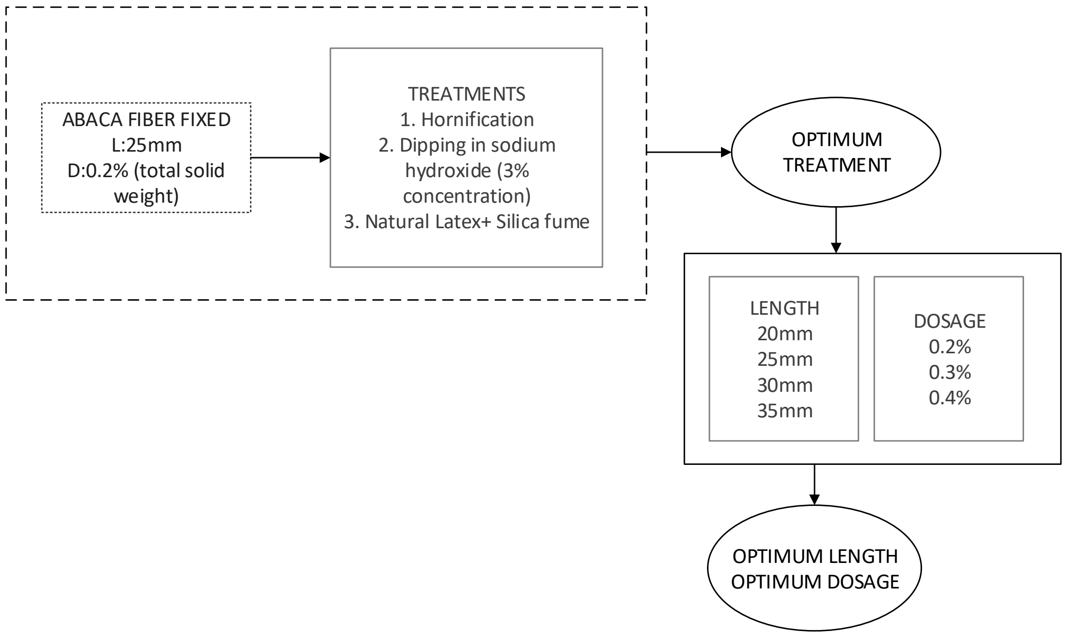

Figure 3.2. Methodology proposed for determining optimum treatment, size and dosage.

Thermogravimetric analysis was performed on no treated and treated abaca fibers. This was performed by comparing the rate of weight loss and the decomposition temperature. The analysis was developed with the use of TGA701 under a ramp rate of 15°C/minute from room temperature to 104°C and 50°C/minute from 104°C to 1000°C. The crystal structure of the no treated and treated abaca fibers was characterized. X-ray diffraction (XRD) was performed to 4 samples of abaca fibers subjected to hornification (HR), NaOH-3% (HS), natural latex + silica fume (LS) and the No treated fiber (NT). This technique is used to identify the crystalline minerals in the natural fibers when their content is higher than 1%. The degree of CI in the cellulosic materials is calculated according to the peak height method. The empirical equation proposed by Segal et al., (1959) for calculating the crystalline index is given in Equation 1.

$$CrI = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

In SEM, samples were prepared in order to analyze the superficial changes as a consequence of the different treatments applied. The three point bending tests on unnotched specimens were carried out according to UNE 196-1 (UNE, 2018) which evaluate compressive and flexural properties of cement based mortar. FT-IR was performed on treated and untreated abaca fibers. This technique is sensible to internal structures since it reflects the characteristic vibrations of atomic groups. To carry out tensile tests, the treated and untreated fibers were split into parcels. This test was performed taking as references the procedure from Cai et al. (2015) and ASTM C1157 (ASTM International, 2023b). The testing machine corresponds to an IBERTEST MOD IBTH-2730. The samples were put in a porta sample that consists of a 300 g which was tied through epoxy on each bottom in order not to interfere with the test. Fibers length between 300-500 mm. The fiber diameters were obtained through SEM (200μm), as showed in Figure 3.3, using the software Motic Images plus 3.0.

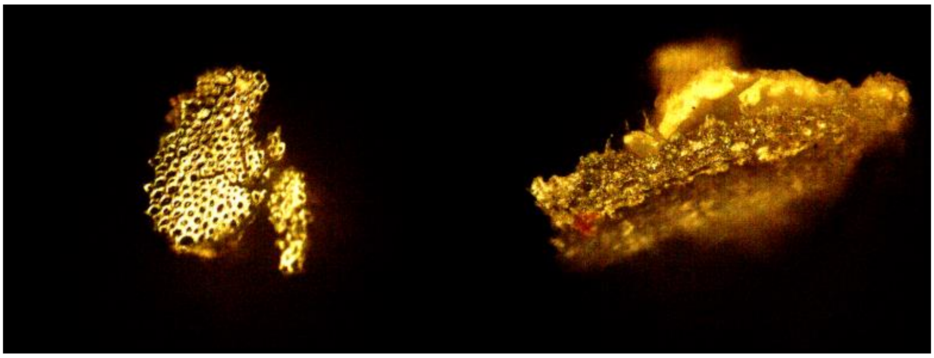

Figure 3.3. Images of abaca fibers through scanning electron microscopy.

Figure 3.4 shows the design of the porta sample for tensile test. The strength at breaking point (N) is gotten from the testing machine, the cross-sectional area of the fiber in the fracture plane is obtained through reflection microscopy, zoom 200, OLYMPUS PME3. This test was performed applying a maximum load of 500 N and a velocity rate of 1 mm/min.

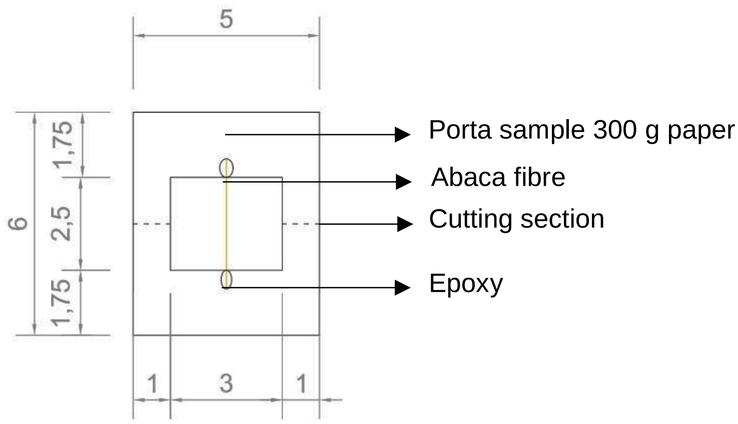

Figure 3.4. Porta sample for tensile test. Units: cm.

3.3. Results and discussion

It is important to understand the natural fibers cell wall in order to predict the mechanical properties of the fiber itself. Considering that all natural fibers are composed of pectin, cellulose, hemicellulose and lignin (Boulos et al., 2019), it turns necessary to analyze the behavior of treated samples within its composition. On the other hand, different dosages and sizes may also influence the behavior of the cementitious mortars.

3.3.1. Characterization of abaca fibers

3.3.1.1. Thermogravimetric analysis (TGA)

Thermogravimetric analysis was carried out to investigate the changes that the abaca fiber may suffer through the proposed treatments. Figure 3.5 shows the curves of the different conditions of abaca fibers subjected to hornification (HR), NaOH-3% (HS), natural latex+silica fume (LS) and to the No treated fiber (NT). Figure 3.5 (a) DTG curve shows an initial peak between 40° C and 100°C (loss in weight about 4%) which corresponds to the vaporization of absorbed water. After this peak, the curve shows two more peaks. According to Y. J. Rueda-Ordóñez and Tannous (2018), thermal disintegration of natural fibers begins with the decomposition of the hemicellulose that starts at 200°C to 400°C, then cellulose between 300°C and 410°C and lignin from 400°C to 800°C. Figure 3.5 (b) Among NaOH, degradation starts at 200℃ corresponding to hemicellulose with a 20% weight loss, then cellulose between 380℃ up to 400℃ with a 65% weight loss and lignin at 680℃ with a 20% weight loss. Figure 3.5 (c) HR degradation starts at 270℃ corresponding to hemicellulose with a 10% weight loss, then cellulose at 320℃ with a 60% weight loss and lignin at 500℃ with a 30% weight loss. Figure 3.5 (d) LS degradation starts at 300℃ corresponding to hemicellulose and 20% weight loss. At 400℃ cellulose degrades with a 5% weight loss and lignin at 500℃ lignin with a 5% weight loss. This behavior means that there are few volatile elements and that's why the variation of weight vs temperature is small, it can be seen a constant velocity for which only one type of material is being liberated. Among these behaviors it can be seen that NaOH and HR treatment have some similarities, for example the weight loss of cellulose is around 60% and lignin at an average of 25%. But it is interesting how the LS treatment varies from all others losing only a 5% weight loss in the cellulose and in lignin. Between these results, it should be analyzed if these behaviors interfere within flexural strength results.

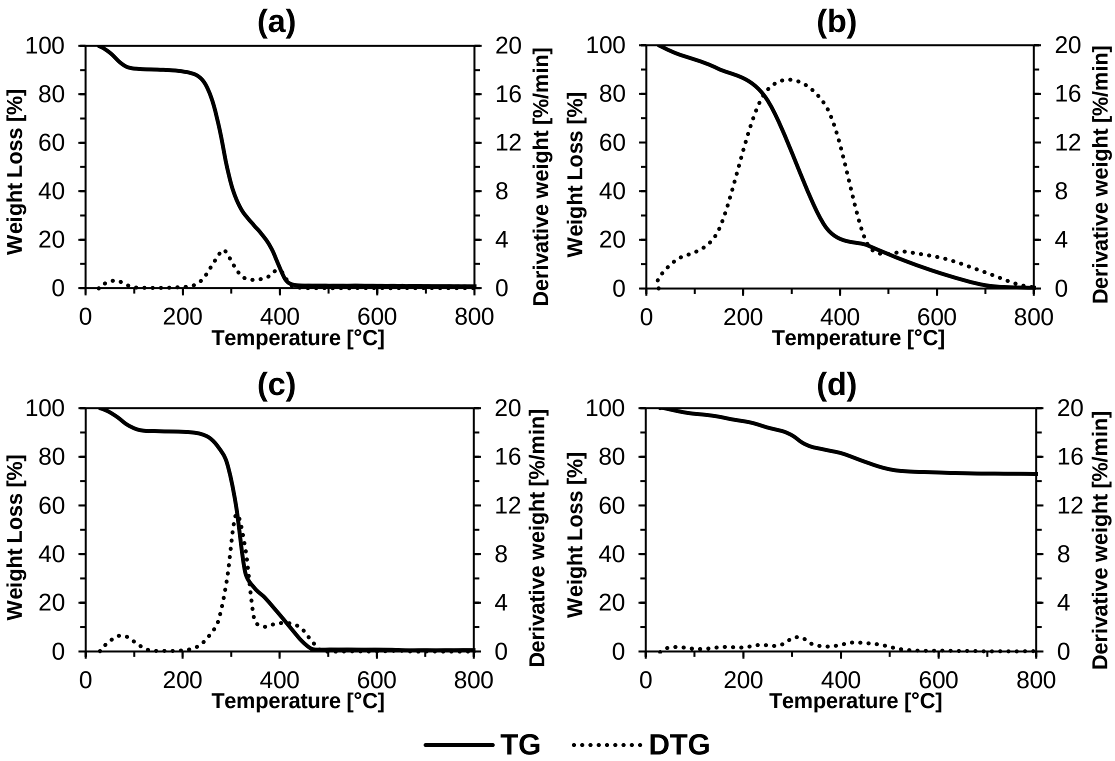

Figure 3.5. TG and DTG curves. (a) NT: No treated fiber. (b) HS: NaOH-3%. (c) HR: Hornification. (d) LS: Natural latex + silica fume. TG: Thermogravimetric curve. DTG: First derivative of the TG curve.

3.3.1.2. X-ray diffraction

X-ray diffractograms for all four samples are presented in Figure 3.6. Two main peaks can be seen through all the analyzed samples at 16.4° and 22.5°, typical of the cellulose which have a considered width referring to an amorphous material (H. S. Kim et al., 2023). There's an unusual peak at 31.34°in the LS sample which refers to a calcium sulfate (Eremin et al., 2016). These results allow the analysis of the cellulose structure and crystallinity. Table 3.2 shows the results of the 4 samples in terms of CrI%, the highest and the lowest index corresponds to the HS (38.46%) and LS (28%) treatment respectively. All treatments have eliminated no crystalline materials on the fiber, including amorphous hemicellulose, lignin and others non cellulosic materials, which has given the fiber the chance of adopting a more crystalline structure (Cai et al., 2016). These results have a certain connection with results from TGA where the weight loss of cellulose and lignin were totally alike within these two treatments hence it would be interesting to analyze if this behavior repeats in terms of flexural strength.

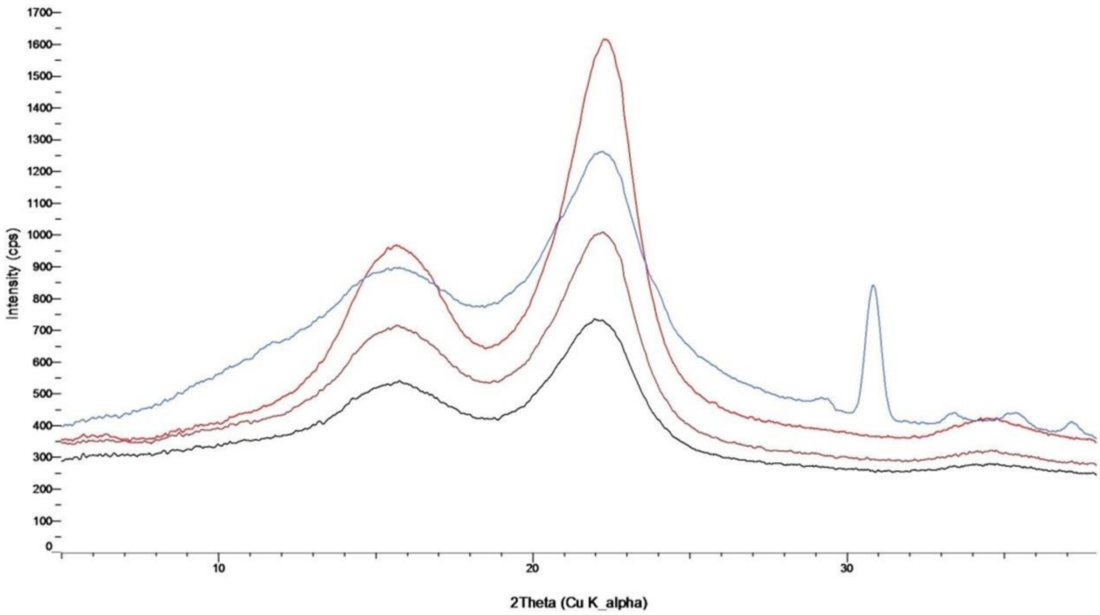

Figure 3.6. Diffractograms for abaca fiber: NT HS, HR, and LS.

Table 3.2. Crystallinity index of NT, HS, HR and LS abaca fiber.

Fiber	I_{total}	I_{am}	CrI (%)
NT	720	500	30.50
HS	1625	1000	38.46
HR	1000	700	30.00
LS	1250	900	28.00

NT: No treated fiber. HS: NaOH-3%. HR: Hornification. LS: Natural latex + silica fume. Itotal: Intensity at 22.1°. Iam: Intensity at 16.0°. CrI: Crystallinity index.

3.3.1.3. Fourier-transform infrared spectroscopy

The chemical composition of all samples was analyzed as seen in Table 3.3. Peaks were formed corresponding to different bondages such as: (-OH), (H-O), (C-H), (C=O), (C=C), (C-H), (C=C), (H-C-H), (C-O), (C-O-C) and (C-O) showed in Figure 3.7, these bondages are associated to elements referred as polysaccharides, cellulose, hemicellulose, pectin and lignin.

Table 3.3. Absorption peaks of abaca fiber submitted to different treatments.

Wavenumer (cm^{-1})	Link (functional group)	Possible assignment
3400	-OH	Cellulose, hemicellulose, lignin

3336	O-H	Polysaccharide
2893	C-H	Cellulose and hemicellulose
1742	C=O	Hemicellulose and pectin
1603	C=C	Lignin components
1514	C-H	Hemicellulose and pectin
1510	C=C	Lignin
1430	H-C-H	Pectin and lignin
1242	C-O	Lignin
1100	C-O-C	Cellulose
1062	C-O	Hemicellulose and lignin
1026	CH_3	
900	Glycosidic links	Polysaccharide
896	C-OH	β glucosidic linkage presence between the monosaccharide
795	Si-O-Si	Symetric stretching modes of SiO4 units

Within the three applied treatments HR, HS and LS, it can be seen in Figure 3.7 that they all look similar, except LS; apparently, this treatment radically transforms the bondage bringing a new value to show, these results are consistent with the ones obtained through TGA and XRD. HR treatment looks similar to NT fiber, this may imply that there is no significant change in the chemical fiber's composition. HS treatment shows at 1742 cm^{-1}, peak that refers to the bondage C=O, where the hemicellulose and pectin disappear, as TGA results confirm, at 1100cm^{-1} another peak the C-O-C shows up referring to cellulose; therefore, it can be said that part of the lignin and pectin are eliminated from the fiber. Hence, the HS treatment removes the binding materials such as hemicelluloses, pectin and lignin, this led to the separation of the abaca fiber bundle into elementary fibers (Cai et al., 2015).

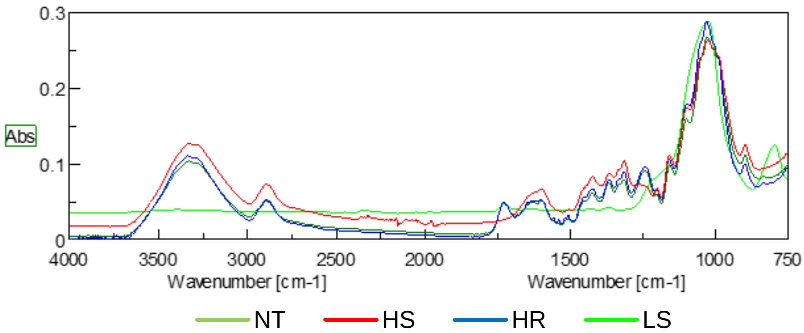

Figure 3.7. FT-IR of abaca fiber submitted to NT, HS, HR and LS.

3.3.1.4. Tensile test

After summiting all samples to tensile strength as seen in Figure 3.8, it was obtained that NT abaca fiber has the lowest strength compared to treated abaca samples. Results were summarized in Table 3.4. According to these results Ecuadorian abaca fibers may seem less resistant compared to Philippines 750 MPa (K. Liu et al., 2013). However, these results may vary depending on the mechanism applied to measure this property, as well as the harvesting and cropping procedure. If we compare the results of tensile tests to previous tests it can be seen that HS and LS treatment have the highest and the lowest results in terms of tensile test, these results are congruent to the ones obtained by TGA, XRD, FT-IR where hemicellulose, cellulose and weight loss vary tremendously. Summarizing, HR is 5.4% more resistant than NT and HS treatment increases its strength in a 26.41%; therefore, it can be said that HS treatment has the best performance in terms of tensile strength.

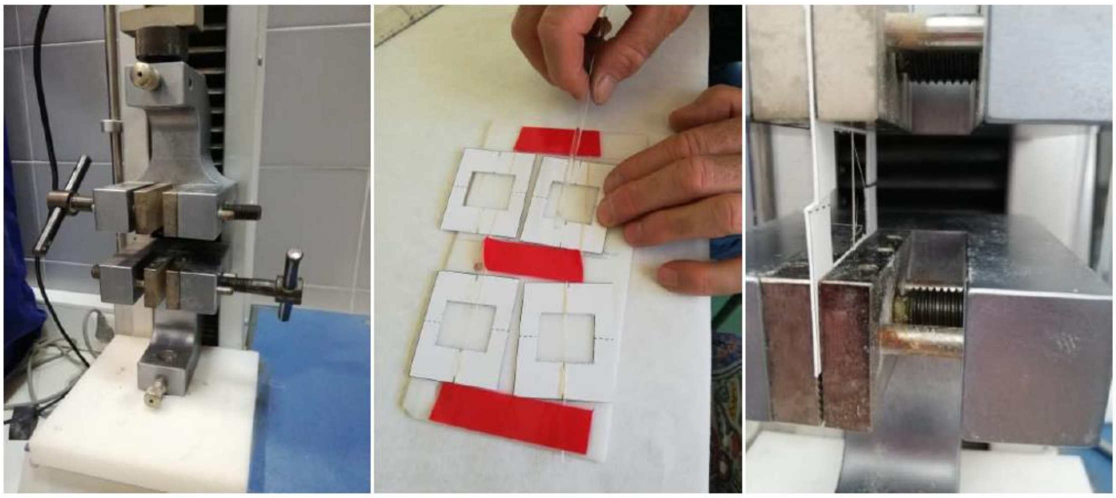

Figure 3.8. Tensile test for abaca fiber NT, HS, HR and LS.

Table 3.4. Tensile test of NT, HS, HR and LS abaca fiber.

Fiber	Tensile test (MPa)
NT	342.33
HS	465.18
HR	361.88
LS	350.90

3.3.1.5. Scanning electron microscopy (SEM)

In Figure 3.9 (a) SEM analysis of 300 µm show the behavior of abaca fibers in all their conditions NT, HR, HS and LS. Abaca fibers experiment a modification of its surface when subjected to proposed treatments. It can be seen how the NT fibers show a similar aspect compared to the HR and HS treatment; on the other hand, LS fibers show a totally different aspect, having as main element Silica fume with almost unnoticeable fibers. Figure 3.9 (b) shows fibers in all proposed conditions at 50 µm, it can be seen changes in the surface's morphology of all of them.

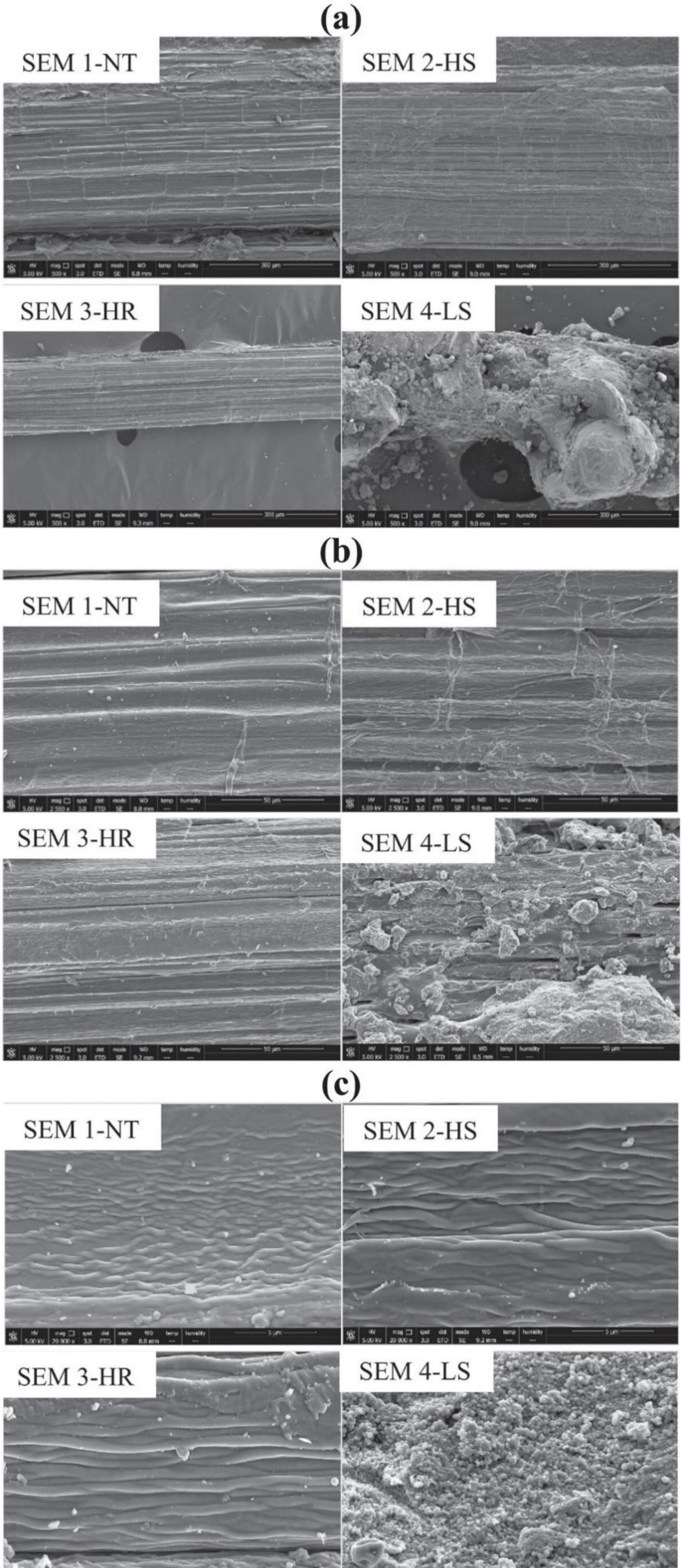

Figure 3.9. Abaca fibers NT, HR, HS and LS respectively, corresponding to (a) 300μm, (b) 50μm and (c) 5μm of backscattered electrons.

HR treatment shows how the morphology of the natural fiber has disappeared, meaning that transversal elements previously seen in NT fiber has been diluted, fiber's roughness has increased as well. As shown with LS, the fiber seems to be present but it is covered by latex and the small rocks that can be appreciated is the silica fume. As shown in Figure 3.9 (c) at 5 µm, the HR and the HS treatment, the fiber suffers a separation, originally it seems to be tight in a bundle, but it looks different. The LS treatment shows small spherical elements corresponding to silica fume. In general, all applied treatments modify the fiber' surface, hence the adhesion must be considered in cementitious matrix.

3.3.2. Abaca fibers in cementitious mortar

One of the most determining tests in terms of masonry mortar is the flexural strength. Therefore, to determine the best treatment (HR, HS, LS), prismatic samples were performed, fixing a size in 25 mm and 0.2% dosage for comparison. Once the best treatment was determined, different sizes and dosages were tested to find out optimum size and dosage. Table 3.5 shows the results after establishing a fixed dosage of 0.2% and a fiber length of 25 mm. At first sight, the LS treatment accomplished better results among all treated fibers, but at 28 days these results were lower than NF mortar. It seems interesting the fact of how the HR treatment didn't increase its strength within the first 15 days, but at 28 days it was 3% higher than NF sample. On the other hand, HS treatment at 7 days showed lower strength compared to NF and LS sample, but at 28 days it showed the highest strength within all samples compared, increasing its strength by a 9%. Compressive strength results of NF sample compared to HR, LS and HS respectively were 18% lower, 16% lower and 7% higher, meaning that at 7 days the HS treatment showed the highest results in terms of compression. This behavior can be seen within the 28 days where LS and NF samples reached the same strength, HR increased by a 2%, but HS treatment showed better results increasing its strength by a 6% %. Using fixed mortar samples of 25 mm length and 0.2% dosage (total solid weight) the best treatment was the 3% NaOH solution (HS treatment). Once the optimum treatment was determined (3% NaOH solution) HS, different lengths and dosages were tested.

Table 3.5. Mortar samples flexural and compressive strength of NF, HS, LS and HR at 7, 14, 28 days.

Treatment	**NF**	**HS**	**LS**	**HR**
Dosage		0.20%		
Fiber length		25 mm		
Flexural strength (MPa)				
7 days	5.7	5.3	5.5	4.3
14 days	6.2	6.7	6.3	4.4
28 days	6.7	8.0	6.5	6.9
Compressive strength (MPa)				
7 days	14.50	15.50	12.50	12.30
14 days	17.80	17.50	17.20	17.10
28 days	19.30	20.50	19.30	19.70

Figure 3.10 shows all dosages and lengths analyzed. The highest flexural strength results in were obtained when abaca fibers were added to the mixture with a 30 mm length and a dosage of 0.2% of the total solid weight, resulting in 8.2 MPa at 28 days; this represents an

increase of 18% compared to NF mortar. It can be seen that as lengths increase flexural strength decreases. In terms of compressive strength, the behavior is similar, at 30 mm length with 0.2% the highest compressive value is achieved, being 8.5% higher than NF.

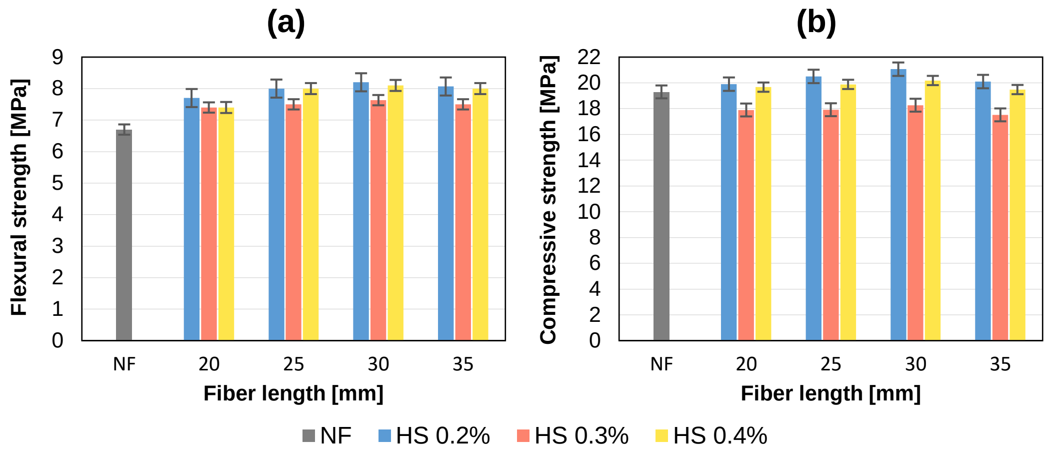

Figure 3.10. Samples of NF, HS with 0.2%, 0.3%, 0.4% and lengths of 20, 25, 30, 35 mm. (a) Flexural strength at 28 days. (b) Compressive strength at 28 days.

3.4. Conclusions

This work presented the effect of three different treatments on abaca fiber: hornification (HR), NaOH-3% (HS) and coating of silica fume with natural latex (LS). From this investigation it can be concluded that:

- Fixing an initial dosage and length of 0.2% of the total solid weight and 25 mm, best results were obtained from fibers treated with NaOH HS treatment.
- All three treatments modify the fiber itself, removing lignin and hemicellulose from the surface of natural fibers as proven through TGA, XRD, FT-IR and SEM analysis.
- HS fiber (NaOH) had better behavior in terms of flexural and compressive strength, when the fiber was analyzed through this treatment it was seen that hemicellulose, cellulose and lignin were lost and bondages of H-C-H were formed.
- Silica fume with natural latex treatment (LS) showed the poorest results in terms of tensile test and flexural strength, this could be because once the treatment was applied, internally few volatile elements remained compared to HR and HS treatment, meaning that internally the composition was modified, but this variation wasn't as different as the others, only 5% weight loss compared to 65% and 30% in HS and HR respectively
- HR treatment, in terms of flexural strength, within the first days showed the highest results, but after reaching the full strength, it was seen that results were lower than HS treatment.
- NaOH samples with different dosages and lengths were analyzed (0.2-0.3-0.4% and 20-25-30-40 mm). Results in terms of flexural strength show better behavior when abaca fiber's length are 30 mm with a 0.2% dosage of the total solid weight.

Chapter 4.
Effect of aging process on mechanical performance of reinforced mortar with NaOH abaca fibers[2]

[2] The result presented in this chapter were published in Alcivar-Bastidas, S., Petroche, D. M., Cornejo, M. H., & Martinez-Echevarria, M. J. (2024). Effect of aging process on mechanical performance of reinforced mortar with NaOH abaca fibers. Case Studies in Construction Materials, 20, e03122. https://doi.org/10.1016/j.cscm.2024.e03122

4.1. Introduction

Concrete is one of the most used materials by man (Petroche & Ramirez, 2022) and its main component, cement, is responsible for over 8% global CO_2 emissions (Guo et al., 2021; Salas et al., 2016). The search for alternative materials to reduce the carbon footprint has led to the study of alternatives such as geopolymers (Almutairi et al., 2021; de Oliveira et al., 2022; Salas et al., 2018), fly ash (Kurda et al., 2020; Yaseen et al., 2022; L. Zhang et al., 2023), metakaolin (Machner et al., 2018a, 2018b; Ren et al., 2023), slag (Awoyera & Adesina, 2019; Khair et al., 2024; Shobeiri et al., 2023; Snellings et al., 2022), silica fume (Lou et al., 2023), calcined clay (Avet & Scrivener, 2018; Scrivener et al., 2018; Sharma et al., 2021), construction/mine waste (Borges et al., 2023; R. B. da Silva et al., 2022; Mazurana et al., 2022), carbon capture (Junior et al., 2021; Neves Junior, Dweck, et al., 2019; Woydich et al., 2023) and bio composites (Abdalla et al., 2023; Choi, 2022; Riofrio et al., 2022; San Andrés et al., 2023; Sheeba et al., 2023).

The use of vegetable fibers constitutes a very interesting alternative as raw materials for cement-based composites in terms of environmental conservation, energy, and resources having a low cost and obtained from renewable sources (Cheung et al., 2009; M. Li et al., 2020; Lilargem Rocha et al., 2022; Luo & Netravali, 1999; Singh et al., 2023; Vasiliki Pachta et al., 2014). Natural fibers have been commonly used through years; in Ancient Greece, wood and straw fibers were particularly used in order to increase volume stability in mortars (Kesikidou & Stefanidou, 2019), they are generally used to avoid shrinkage cracking phenomena, increase brittle materials fracture toughness and develop strain-hardening behavior (Candamano et al., 2021). Studies demonstrate that natural fibers have higher toughness, improved impact and higher deformation capacity (Benaimeche et al., 2018; Çomak et al., 2018; de Lima et al., 2022; Ramakrishna & Sundararajan, 2005; Tolêdo Filho et al., 2003).

A variety of natural fibers are available in the nature such as: flex, hemp, jute, sisal, isora, ferula, bamboo, okra, guaruman, abaca, coir which are used as reinforcement in different matrices polymeric or cementitious (Ardanuy et al., 2015; Azevedo, Lima, et al., 2022; Azevedo, Rocha, et al., 2022; Azevedo et al., 2020; Ferreira et al., 2015; Lima, Oliveira, et al., 2023; Lima, Tambara Júnior, et al., 2023; Scalici et al., 2016; Song et al., 2021; Sood & Dwivedi, 2018). As it is well known, the use of natural fibers, as an alternative to synthetic ones, as reinforcement of composite materials have received growing attention according to their specific properties, price, because of those reasons, natural fibers began to be used as reinforcement of composite materials in engineering (Scalici et al., 2016). The development of this type of materials may introduce new chances for improving the farming business where good quality cellulosic fibers species could be achieved; these vegetable fibers can be obtained from agricultural by products which may generate an additional value to agro-industrial products (Mármol & Savastano, 2017).

Between all available natural fibers around the world, there is abaca, also known as Manila hemp, this fiber has called researchers attention not only because of its flexibility and high tensile strength, but also because its cultivation reduces soil erosion levels and rehabilitates biodiversity in tropical areas. Abaca fiber is important to Ecuador's economy because it represents the 0.2% of the primary exports. Since 2010 this number has increased constituting more than $150 million in its economics (Banco Central del Ecuador, 2018), representing the 15% of the worldwide consumption (Kumar Sinha et al., 2017; Richter et al., 2013). According to Lomerio & Oloteo (Lomerio & Oloteo, 2000), there are more than 200 different varieties of abaca, Ecuador has two varieties: Bungalanon and Tangongon that differ from the ones in Philippines because of its farming and cultivation process (Araya-Gutiérrez et al., 2023). Abaca has been widely used in ropes, bags, paper, including the automobile industry because of its mechanical properties (Agung et al., 2011; Simbaña et al., 2020).

As other natural fibers, abaca fiber could be used as composites reinforcement (Ashik & Sharma, 2015). It is known that fibers as reinforcement for concrete are used to provide ductility so that they support the action of tensile loads (George et al., 2020), but its use may be affected in terms of adhesion and durability; therefore, in order to improve the necessary adhesion between fibers and matrix, several chemical treatments can be used (Sepe et al., 2018), this is also helpful concerning to durability in which the natural fiber suffers a chemical or physical modification for increasing its stability in cementitious matrices (Boulos et al., 2019; Jiang et al., 2018).

Studies from Fidelis et al., (2016) summarizes different treatments and their advantages, particularly focusing on controlled alkaline treatment. Among various treatments, alkali treatment stands out as one of the most popular due to its costs efficiency and favorable outcomes in enhancing adhesion. Studies have utilized different percentages of sodium hydroxide, including 3%, 5%, 10% and 15% (Cai et al., 2016; Jiang et al., 2018), showing promising results in terms of increased crystallinity, tensile strength and Young's modulus when compared to untreated fibers.

Despite all these advantages, many challenges arise concerning the use of natural fibers in cementitious applications, perhaps the most critical is the low durability of fibers in cementitious matrix, knowing that natural fiber durability depends on the durability of constituents exposed to stress. In the case of natural weathering, most stress factors are a combination of temperature and humidity variations (Zukowski et al., 2018), the considerably lower resistance to the high alkaline environment of the cementitious matrix can reduce the reinforced effect of these fibers and lead to premature failure of the composites (Ferreira et al., 2021; Z. Fu & Yao, 2022; Wei, 2018; Wei & Meyer, 2015).

As a result the deterioration of natural fibers in a cement matrix has emerged as the major problem to be solved in order to produce durable natural fiber reinforced cement composites (Arshad et al., 2020).

One of the methods that has been used for modifying the surface of the fiber is hornification, but the process of hornification cycles of wet/dry (w/d) could be used as well for estimating the degradation of the material, the main reason for analyzing the degradation is the lack of data on the behavior of the bio composite within its service life, years and years should be waited in order to analyze whether the fiber is still contributing to the mechanical properties of the mortar. For this reason, applying cycles of w/d are a good opportunity to estimate the degradation of the material. As Poletanovic et al., (2021) described in her hemp study subjected to alkali treatment where 10 cycles of w/d alternatively used showed an increase of approximately 20% of compressive and flexural strength compared to reference mortar. Leng et al., (2023) analyzed the performance of bamboo subjected to accelerated aging process through w/d cycles, applying 8 cycles alternatively, achieving size changes of samples after subjected to w/d, mortars reinforced with bamboo had better results in terms of flexural and compressive strength.

Therefore, durability is an important topic that must be analyzed. It is important to remark according to the literature available to date, accelerated cycles of w/d to treated alkali fiber has only been done before for hemp by Poletanovic et al., (2021); not procedure of accelerated w/d cycle to treated abaca fiber or another natural fiber has been performed. In terms of bio composites, it is important to contribute by increasing the catalogue of available natural fibers and its behavior as bio composites since the availability of natural fibers vary according to the region.

For this reason, the aim of this research is to (1) compare the mechanical behavior of a mortar with no fiber (MSF) vs. an alkali treated fiber reinforced mortar (MFTHS) applied to masonry in construction industry and (2) to compare the mechanical properties of these mortars when subjected to accelerated aging process of w/d.

4.2. Material and methods

4.2.1. Materials

4.2.1.1. Natural fiber and alkali treatment

Abaca fiber is derived from the pseudo-stem of *Musa textile*, commonly referred as manila hemp. This plant produces small, inedible fruits with seeds, and its pseudo-stem can reach a height up to 6.5 meters. The fiber was obtained at a national plantation located in Santo Domingo, Ecuador. The dosage was determined after evaluating various quantities in a preceding study conducted by same authors (Alcivar-Bastidas et al., 2023), ranging from 0.2 to 0.4%, along with differing fiber lengths 20 mm, 25 mm, 30 mm. The optimal outcome in terms of flexural strength, measured at 8.2 MPa, was obtained with a 0.2% concentration and a fiber length of 30 mm treated with 3%NaOH solution. This selection was influenced by findings from Lee & Choi, (2022) where chopped abaca fiber of comparable lengths and concentrations was employed.

Figure 4.1 (a) shows scanning electron microscope (SEM) results of no treatment and, Figure 4.1 (b) shows NaOH treatment abaca fiber at 5 μm of backscattered electrons, its surface has been modified and according to Alcivar-Bastidas et al., (2023) better bonding and results were achieved. Fibers were dipped into this solution for 4 hours and then in water for 10 hours. Impregnated abaca fibers were rinsed with water until extruded brown washing became transparent, as described by Jiang et al., (2018).

(a) **(b)**

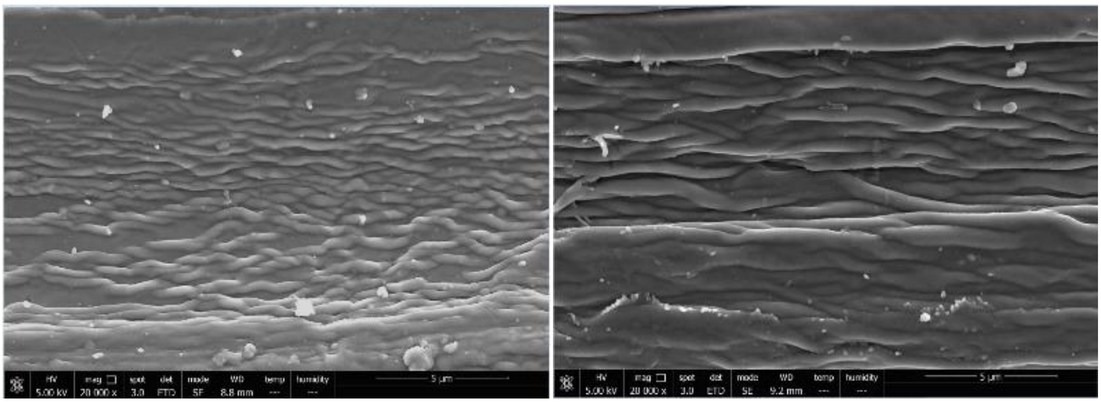

Figure 4.1. Abaca fibers (a) no treatment, and (b) NaOH treatment, corresponding to 5µm of backscattered electrons.

4.2.1.2. Matrix

All samples, mortar with no fiber (MSF) and alkali treated fiber reinforced mortar (MFTHS) were designed according to UNE-EN 196-1 (UNE, 2018) and its composition is listed in Table 4.1. The cement used in this research is a general use (GU), its specification is indicated at ASTM C1157 (ASTM International, 2023b), its bulk oxide composition and other properties are shown in

Table 4.2. Natural sand was used and its granulometric curve was set as the average between ASTM C144 (ASTM International, 2018) maximum and minimum ranges; matrix ratio relation cement: sand 1:3 by weight, with 110±5 mm of the flow rate, following the procedure for workability test of flow table according to UNE-EN 1015-3 (UNE, 2000).

The flow rate range 110±5 mm was maintained for both types of mortars, MSF and MFTHS. However, due to the presence of fiber, additional water was necessary to achieve the same workability. In this study, the fixed flow rate enabled the control of mixture workability.

Table 4.1. Mortar mix composition for 1 m^3.

Materials	**Sand**	**Cement**	**Water**	**NaOH Abaca Fiber**
			Flow rate 110±5 mm	**0.2% total solid weight**
MSF	1458 kg	486 kg	274 kg	--
MFTHS	1458 kg	486 kg	301 kg	4.37 kg

Table 4.2. Bulk oxide composition and other properties of GU cement.

Materials	Amount
SiO_2 [%]	16.9
Al_2O_3 [%]	3.05
Fe_2O_3 [%]	1.72
CaO [%]	58.6
MgO [%]	0.71
SO_3 [%]	2.41
K_2O [%]	0.13
Na_2O [%]	0.08
TiO_2 [%]	0.17
P_2O_5 [%]	0.25
LOI [%]	15.9
Specific gravity [g/ml]	2.85
Blaine [m^2/kg]	526
Compressive strength [MPa]	
1day	7.7
3 days	17.1
7 days	22.4
28 days	28.3

4.2.2. Methods

4.2.2.1. Accelerated Aging processes

With the pass of the time there are factors that may improve or decrease certain mechanical properties. Previous researchers (Stapper et al., 2021; Wei & Meyer, 2014, 2015, 2017) have analyzed different processes for simulating the degradation of cement composites. Therefore, in this research samples were subjected to two types of w/d cycles as accelerated aging processes, simulating severe and medium condition of affectation, the main reason for analyzing w/d cycles is because Ecuador is a country with only two seasons: dry and rainy, hence this process results in the most severe exposition for mortars. Two w/d cycle methodologies are proposed within the context of these conditions, and they follow the procedure as shown in Figure 4.2.

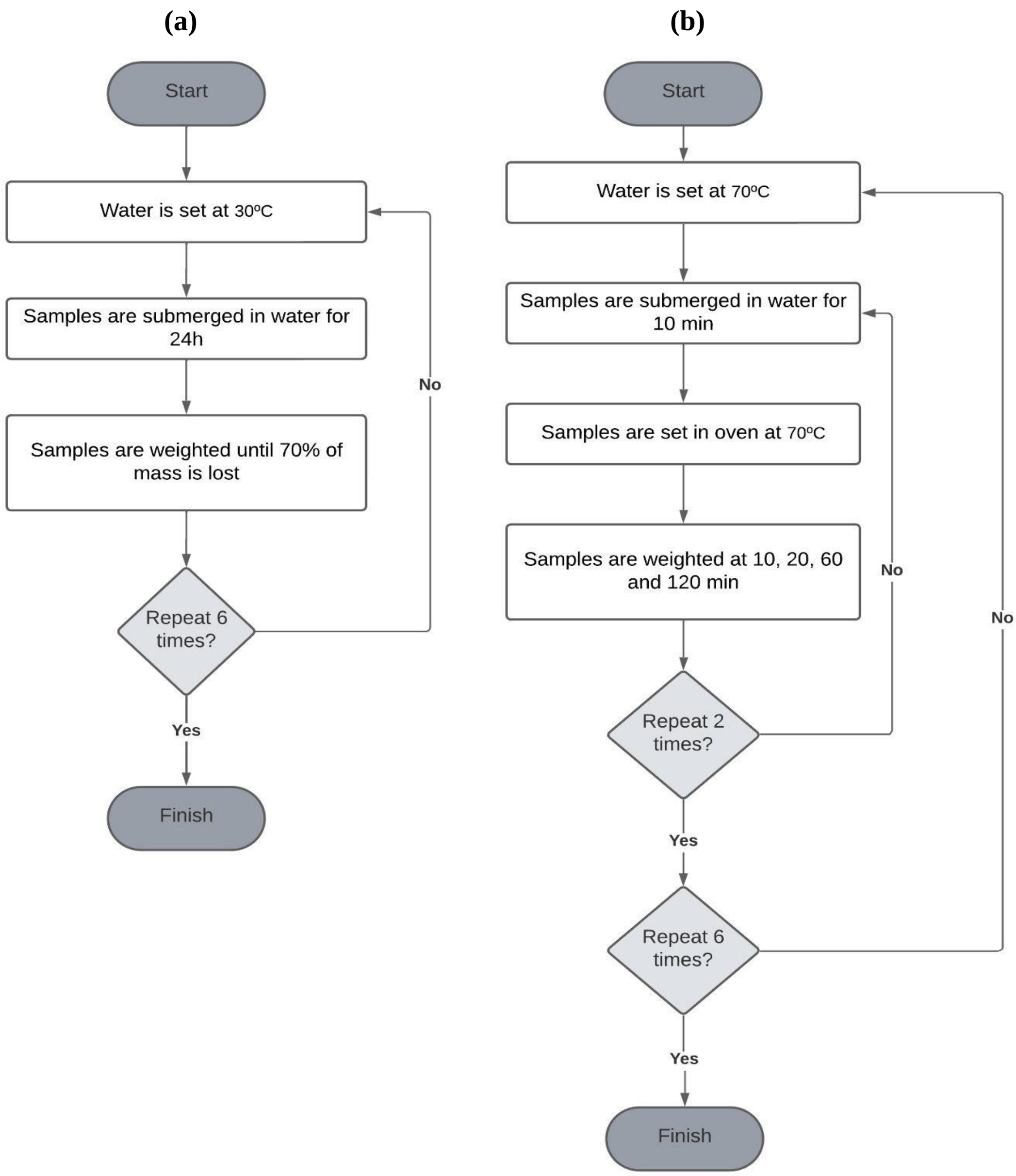

Figure 4.2. (a) Aging process of wet and dry 1 (WD-1), according to Neves Junior, Ferreira, et al., (2019). and (b) aging process of wet and dry 2 (WD-2) according to Wei et al., (2016).

The first w/d cycle (WD-1) is the one proposed by Neves Junior, Ferreira, et al., (2019), with 6 cycles of w/d where samples were subjected to an accelerated aging test after 28 days of age. The aging cycles started with the specimens completely saturated in water at 30±1℃. The cycle had a duration of 3 days (1 day submerged in water for complete saturation followed by 2 days of drying inside the forced ventilated chamber). The chamber was set for a temperature of 36±1℃ and wind speed of 0.5m/s. Considering that after 48h of drying the specimens presented a mass loss of about 70% of the mass gained during its saturation. Figure 4.3 summarizes the process of WD-1.

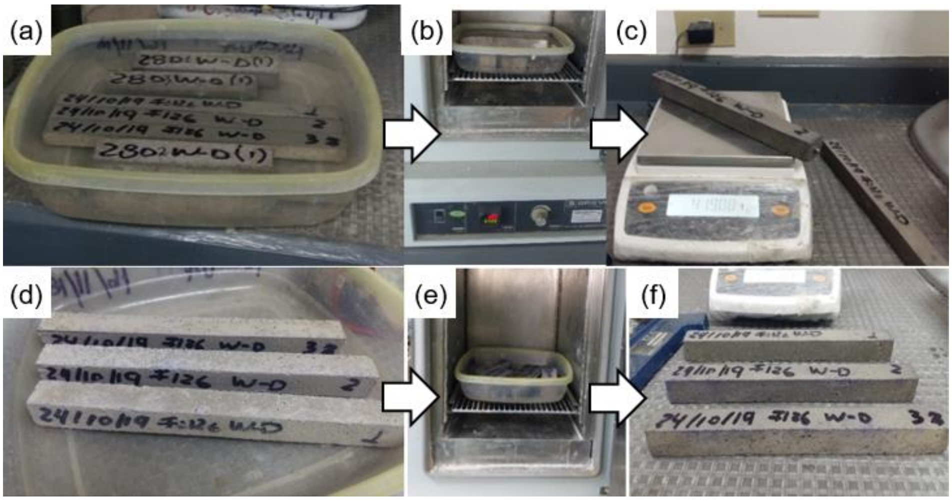

Figure 4.3. Aging process of Wet and Dry 1, according to Neves Junior, Ferreira, et al., (2019). (a) 24 h in water, (b) oven at 30°C, (c) weight test, (d) 48 h dried. (e) oven at 30°C, (f) weight test.

The second w/d cycle (WD-2) is the one proposed by Wei et al., (2016), but applying also 6 cycles of w/d. For this process, specimens were submerged in sealed tap water at 70℃ and then oven dried in a circulating air environment at 70℃ alternately. In order to determine the duration of each cycle, mass change was determined as a function of the immersing or drying time. Increase and decrease of specimen mass subjected to wetting and drying cycles were measured every 10 min for the first 2 hours, every 1 hour subsequently and every 3 (for wetting) or 4 (for drying) hours after 12 hours. Samples were considered at equilibrium when the mass change was less than 2% over 3 consecutive measurements, resulting in 6 cycles. Samples within this research were subjected to both cycles analyzed through dry bulk density, shrinkage, compressive, flexural and tensile strength. Figure 4.4 summarizes the process of WD-2.

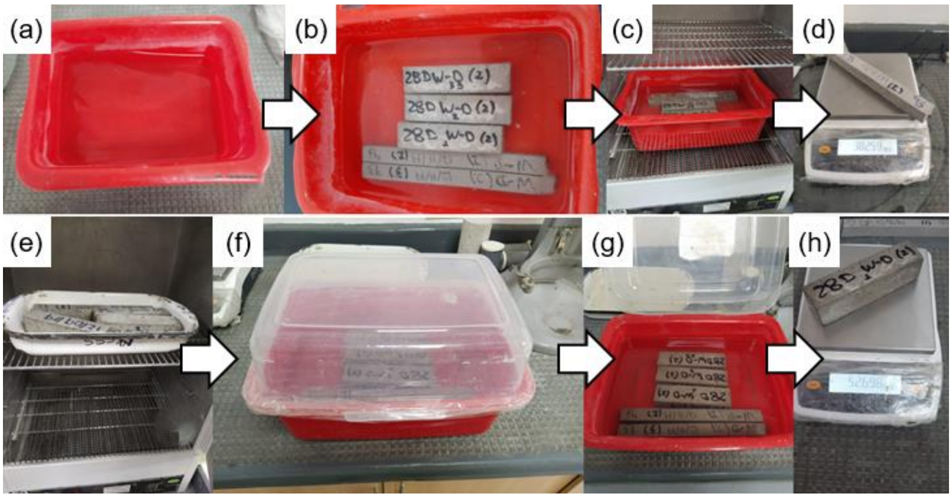

Figure 4.4. Aging process of Wet and Dry according to Wei et al., (2016). (a) water at 70°C, (b) submerged in water for 10 minutes (c) oven at 70°C, (d) weight test. (e) oven at 70°C, (f) submerged in sealed 70℃ water (g) measured after 10, 20, 60, 120 minutes, (h) weight test.

4.2.2.2. Tests samples

4.2.2.2.1. Microstructural analysis to accelerated aging w/d alkali treated fiber

SEM and FT-IR were performed to the alkali treated fiber subjected to accelerated aging cycles of w/d, named as: FWD-1 and FWD-2, according to the w/d process applied. FWD-1 and FWD-2 refer to the abaca fiber samples that underwent an accelerated aging process of w/d. This process was employed to observe the direct effects of aging, specifically WD-1 and WD-2, on the fibers.

This technique (FT-IR) is sensible to internal structures since it reflects the characteristics vibrations of atomic groups and SEM analyze the superficial changes as a consequence of the accelerated aging w/d cycles applied.

4.2.2.2.2. Macro properties of the bio composite

Mortar with no fiber (MSF) and alkali treated fiber reinforced mortar (MFTHS) were analyzed at 28 days through: dry bulk density, flexural, compressive, tensile strength and shrinkage as follows.

Dry bulk density was determined after curing samples, firstly the weight of each water saturated specimen is recorded. Then, the specimen is maintained at 105°C in oven until one weight variation is further recorded. Finally, density is computed as UNE-EN 1015-10 recommendations (UNE, 2020a) and the density mean values.

For flexural and compressive strength, prisms 40x40x160 mm^3 for each type were subjected to three-point bending under load control with loading rate equal to 44 N/s following the UNE-EN 1015-11 and 196-1 recommendations (UNE, 2018, 2020b). Drying shrinkage of mortars measured according to the method described in ASTM C157 (ASTM International, 2017), the length change of the specimen was measured using a vertical comparator for each type. The length was measured at 3, 7, 20, 28, 31, 33, 36, 40, 43, 46 days to later calculate the shrinkage. And tensile strength Figure 4.5, was performed following the Japan Society of Civil Engineers (JSCE) recommendations (Japan Society of Civil Engineers, 2008) using the Shimadzu Autograph AGS-X with 50 KN capacity, allowing obtaining maximum load results and displacements.

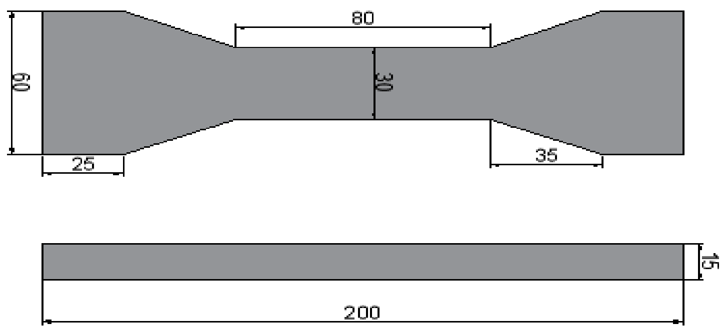

Figure 4.5. Tensile strength measurement for samples according to JSCE recommendations (in mm).

4.2.2.2.3. Macro properties of the bio composite after subjecting samples to accelerated process of w/d WD-1 and WD-2

Samples at 28 days of mortar with no fiber (MSF) and alkali treated fiber reinforced mortar (MFTHS), subjected to cycles of w/d according to Neves Junior, Ferreira, et al., (2019) and Wei et al., (2016) WD-1 and WD-2 respectively, were also tested through tests described in Table 4.3.

Table 4.3. Tests and standards used.

Tests	Standards	References
Dry bulk density	UNE-EN 1015-10	(UNE, 2020a)
Flexural strength	UNE-EN 1015-11	(UNE, 2020b)
Compressive strength	UNE-EN 196-1	(UNE, 2018)
Dry Shrinkage	ASTM C157	(ASTM International, 2017)
Tensile strength	Japan Society of Civil Engineers (JSCE)	(Japan Society of Civil Engineers, 2008)

4.3. Results and discussion

Results have been divided in three parts, the first part corresponds to the microstructural analysis to accelerated aging w/d alkali treated fiber; the second part, corresponds to the comparison of MSF vs MFTHS mechanical properties of the bio composite. And finally, the mechanical properties of the bio composite after subjecting samples to accelerated process of w/d WD-1 and WD-2.

4.3.1. Microstructural analysis to accelerated aging w/d alkali treated fiber

4.3.1.1. Scanning electron microscopy (SEM)

FR, FWD-1 and FWD-2 are shown in Figure 4.6. SEM analysis of 100µm backscattered electrons show the behavior of all three samples; in these images FR and FWD-1 exhibit similarities, but are not identical, there appears to be fiber bonding, and a flatter surface is observed in FR. Similarly, FWD-1 shows some flattening, although not as pronounced as in FR. However, in FWD-2 a rougher surface is evident, and the bonding pattern seems to change. This may occur since WD-2 is more severe if compared to WD-1.

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

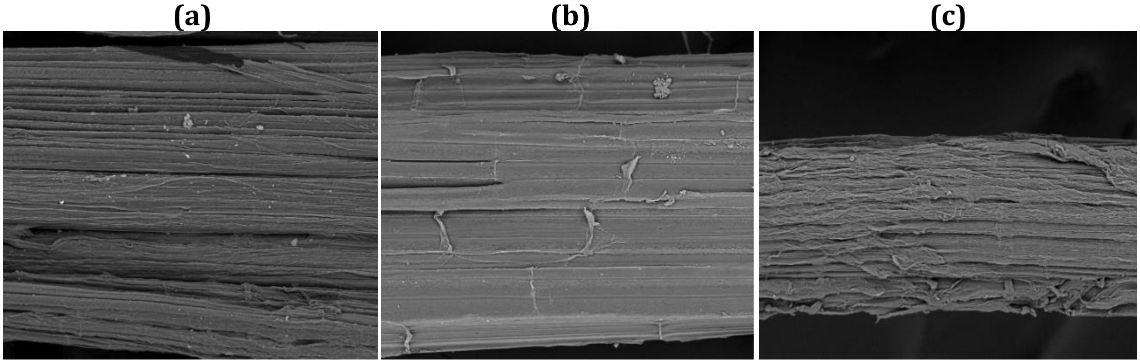

Figure 4.6. FR (a), FWD-1 (b) and FWD-2 (c) subjected to SEM 100µm backscattered electrons.

4.3.1.2. Fourier-transform infrared spectroscopy (FT-IR)

The chemical composition of all samples is listed in Table 4.4. Figure 4.7 shows the comparison between three samples. At first view it can be seen that the chemical composition does not vary significantly between samples, reference abaca alkali treated fiber (FR) in red show a peak between 3450 cm^{-1} corresponding to stretching bands of intermolecular and intramolecular hydrogen bonds, between FWD-1 and FWD-2 the peaks occur at 3433 and 3437 cm^{-1} respectively presenting the same composition. At 2920 cm^{-1} a peak of FR, FWD-1and FWD-2 is visible representing the stretching vibration bands of the C-H group in cellulose. Another important peak occurs at 1640 cm^{-1} producing the stretching vibration band of the free hydrogen-bound hydroxyl, demonstrating the hydrophilic nature of the natural fiber.

Table 4.4. Absorption peaks of abaca alkali treated fiber subjected to processes of WD-1 and WD-2.

Wavenumber (cm^{-1})			**Link (functional group)**	**Possible assignment**
FR	**FWD-1**	**FWD-2**		
3449.05	3433.8	3437.41	-	Stretch bands of intermolecular and intramolecular hydrogen bonds.
2921.67	2916.36	2922.04	C-H	Stretching vibration bands of the C-H group in cellulose.
2852	2858.2	2851.06	$C-H_2$	Stretching vibration band of the $C-H_2$ group due to the presence of meicellulose.
1745	---	---	C=O	C=O stretching vibration of p-coumaric acids of lignin and/or hemicellulose (small shoulder pad).
1640.21	1636.1	1634.26	-	Band stretching vibration of free and hydrogen-bonded hydroxyl, demonstrating the hydrophilic nature of the natural fiber.
1462	1461.3	1467.1	C=C	Vibration caused by the deformations of the C=C bonds of the aromatic ring of lignin.
---	1319.9	1315.1	C-O	C-O bond vibrations.
1269.43	1233.5	1269.99	C-O	Stretching vibration of C-O bonds of acetyl group in lignin.
1044.26	1044.08	1042.57	C-O	Stretching of C-O bonds of hydroxyl and ether groups in cellulose.
784.66	771.7	771.42	C-H	C-H bond stretching vibration of lignin.

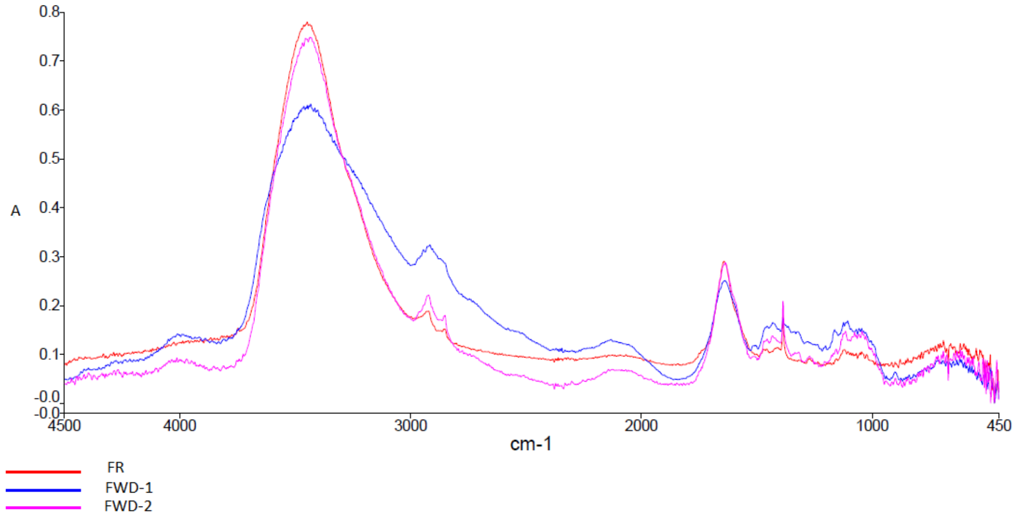

Figure 4.7. FT-IR of abaca alkali treated fiber (FR), and w/d aging process WD-1 and WD-2 (FWD-1, FWD-2).

There is no great difference between FR, FWD-1 and FWD-2 in chemical terms, hence aging process of w/d does not vary the effect of abaca alkali treated fiber even if different procedures applied to the fiber.

4.3.2. Mechanical properties of the bio composite

Samples were subjected to different mechanical properties as showed in Table 4.3, results are summarized in Table 4.5. Determining the mechanical properties of the bio composite is important in order to measure the influence of the alkali treated fiber with the cement matrix.

Table 4.5. Results of mortar samples with and without treated abaca fiber at 28 days.

Tests	MSF	MFTHS
Dry bulk density [Kg/m^3]	1854	1917
Flexural strength [MPa]	6.54	8.2
Compressive strength [MPa]	19.33	26.69
Shrinkage at 28 days [%]	-0.0627	-0.0542
Tensile strength [MPa]	2.87	3.89

In terms of dry bulk density, mortars using the same water content and design composition achieved similar dry bulk density values as mortar Reference Mortar (RM). Samples were measured every 15 minutes until the mass difference was ≤0.2%. MFTHS results were slightly higher (between 3-4%), this could be due to the fact that it contains an additional 0.2% of the total solid weight of abaca fiber resulting in a more compact mortar.

In terms of flexural strength, the MSF mortar reached a 6.54 MPa, but after the addition of treated abaca fiber, MFTHS increased 22% of its original strength resulting in 8.2 MPa, augmenting its early age of flexural strength (Figure 4.8 (a)). These results are comparable to other natural fibers behavior where flexural strength has been improved as summarized by Sood & Dwivedi, (2018).

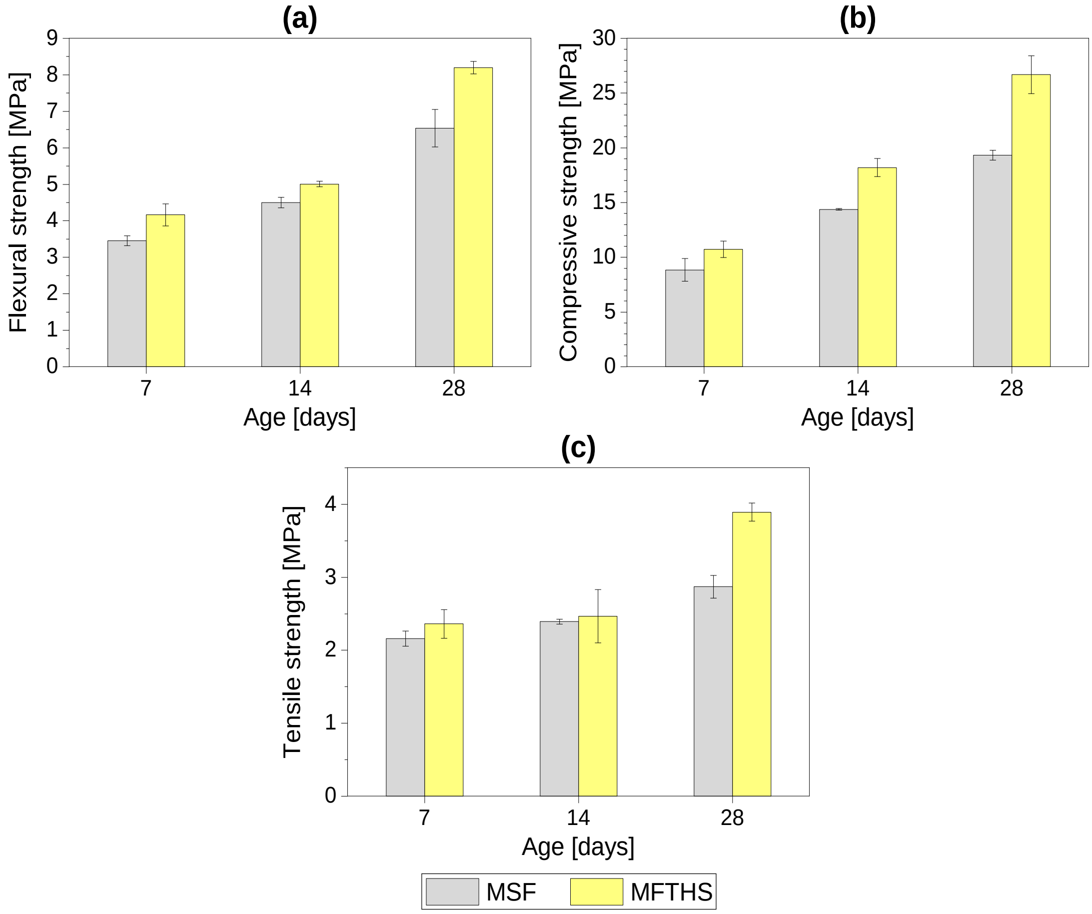

Figure 4.8. Results of mechanical properties (a) flexural strength, (b) compressive strength, (c) tensile strength. MSF: mortar with no fiber. MFTHS: alkali treated fiber reinforced mortar.

It is important to analyze that during the flexural test, RM experienced compression on the top and tension on the bottom surfaces, leading to cracks at the bottom, and a visible crack located near the mid-spam of the sample, regarding to MFTHS, up to certain load the cement mortar mainly absorbed the stress before cracks appearance. Even at early ages, the MFTHS results were higher than RM (Figure 4.8 (a)) Hence, it can be stated that treated abaca fiber slowed down the propagation of cracks due to the bridging effect on both sides of the flexural cracks

The same pattern occurs when analyzing through compressive strength, MSF reached a 19.3 MPa, and MFTHS increased its strength by a 30% (26.69 MPa). The addition of treated abaca fiber in the cementitious mortar improved its strength allowing a better bonding between the matrix and the fiber. Figure 4.1 (b) shows alkali treated abaca surface, the adhesion of the fiber to the matrix depends on numerous factors such as: length, diameter, ratio and roughness. Abaca's roughness allows this bonding resulting in better mechanical strength. Also, MFTHS may have the ability to constrain the growth and widen the cracks in the mortar matrix by bridging micropores and cracks, requiring a greater applied load to propagate cracks when subjected to uniaxial compressive loading. This pattern occurs since early ages as showed in Fig 8.b, where at 7 and 14 days, the compressive strength of MFTHS are higher than MSF.

Shrinkage is an important phenomenon that has a direct relation with the amount of free water and the porosity of the cementitious material. This response occurs after an evaporation of the free water stored in the capillary pores due to a low environmental relative humidity. Within this research, samples were cured by air simulating the most critical scenario (MSF/MFTHS AIR). Values of drying shrinkage were measured for calculation of drying shrinkage rate after 1 up to 45 days. Figure 4.10 shows the results of MSF (RM) and MFTHS (RM) samples subjected to shrinkage, the shrinkage of mortar reinforced with treated abaca fiber illustrates the excellent efficiency in fostering shrinkage reductions. The shrinkage of MFTHS diminishes if compared to MSF, that could expand and generate more cracks. The presence of treated abaca fiber in the reinforced mortar may have inhibited the formation, growth and widening of cracks and micro-cracks due to the bridging mechanism between the pores and cracks. Also, the modified surface of treated abaca fiber could provide a better bonding between the fiber and the mortar matrix resulting in minimizing stress concentrations and minimizing local strains in the mortar matrix.

Samples were subjected to tensile strength, acceptable samples for MSF and MFTHS at 28 days reached 2.87 MPa and 3.89 MPa respectively (Figure 4.8 (c)); MFTHS samples showed an increase of 26% in its strength compared to MSF (Figure 4.8 (b)), this behavior could be given by the addition of abaca alkali treated fiber where a better bonding and cohesion is displayed, delaying the tensile cracks and restrict the crack propagation that typically occurs in the middle portion of mortar specimens under uniaxial tensile force. Hence, a higher load after forming a major crack due to the development of a bridge between the treated abaca fiber and the mortar matrix throughout the rupture zone is more resisted. Pattern than repeats since early ages as shown in Figure 4.8 (c).

4.3.3. Mechanical properties of the bio composite after subjecting samples to accelerated process of w/d WD-1 and WD-2

As described in section 4.2.2.1 samples were subjected to two different processes of w/d cycles, applying 6 cycles, the main reason for performing maximum 6 cycles is that only a2% variation mass is allowed in order for the samples to be considered at equilibrium.. The comparison of w/d cycles is important since this procedure could modify the mechanical behavior of the bio composite.

Table 4.6 summarizes the results obtained from subjecting various types of fibers such as: sisal, pine, eucalyptus, jute and bamboo to different w/d cycles. Results show that mechanical properties decline due to the severity of the process after undergoing these cycles; however, if RM and MFTHS are compared following the application of these cycles on the samples, better results are achieved with treated natural fiber.

As Table 4.6 shows, it is important to analyze the effect of applying these cycles to the samples, comparing the mechanical behavior of RM and MFTHS.

Table 4.6. The effect of different cycles of wet/dry in natural fibers mechanical properties.

Reference	Bio product	MSF	MFTHS
(Ferreira et al., 2015)	Sisal fiber	Dry/wet 16+5h/3h 80°C/22°C Cycles: 10	Better tensile strength and stiffness compared to RM.
(Mendes et al., 2021)	Sisal fiber	Dry/wet 15h/3h T:80°C/22°C Cycles: 5	Better tensile strength compared to RM.
(Ballesteros et al., 2015)	Pine fiber and unbleached eucalyptus	Dry/wet 7h/15h T:60°C/22°C Cycles: 4	Better results in terms of water absorption, bulk density, modulus of rupture, modulus of elasticity compared to RM.
(Ferreira et al., 2017)	Sisal, jute and curauá fiber	Dry/wet 16+5h/3h T: 80°C/22°C Cycles: 10	Better results in terms of tensile strength compared to RM.
(Cid et al., 2020)	Bamboo	Dry/wet 48h/24h T:50°C/22°C Cycles: 10	Better results in terms of modulus of elasticity compared to RM.

4.3.3.1. Dry bulk density

The dry bulk density values for all studied mortars are shown in Figure 4.9 (a). These results show that after applying both processes WD-1 and WD-2 to MSF and MFTHS, alkali treated fiber reinforced mortar have higher density, resulting in a more compact and dense material. Even if both samples were treated with the same procedure of w/d, alkali treated fiber reinforced mortar that were subjected to WD-2 have higher density (2013 Kg/m^3) compared to WD-1 (1997 Kg/m^3).

Apparently, the effect of submerging and drying samples at high temperature (70°C) result in a more compact and dense mortar when referring to MFTHS.

In both w/d processes it can be seen that MFTHS samples show higher density, hence previous treatment applied to the fiber might have contributed to improve the particle packing and enhanced interfacial bonding.

4.3.3.2. Flexural strength

Figure 4.9 (b) shows the results of flexural strength after applying WD-1 and WD-2 to both samples. It can be seen that both procedures diminish flexural strength if it is compared to 28 days' samples, it is certain that aging processes are very aggressive to the samples (temperatures of 70°C, immersion of samples for 24 hours, drying of 48 hours), hence this decrease of strength is suitable.

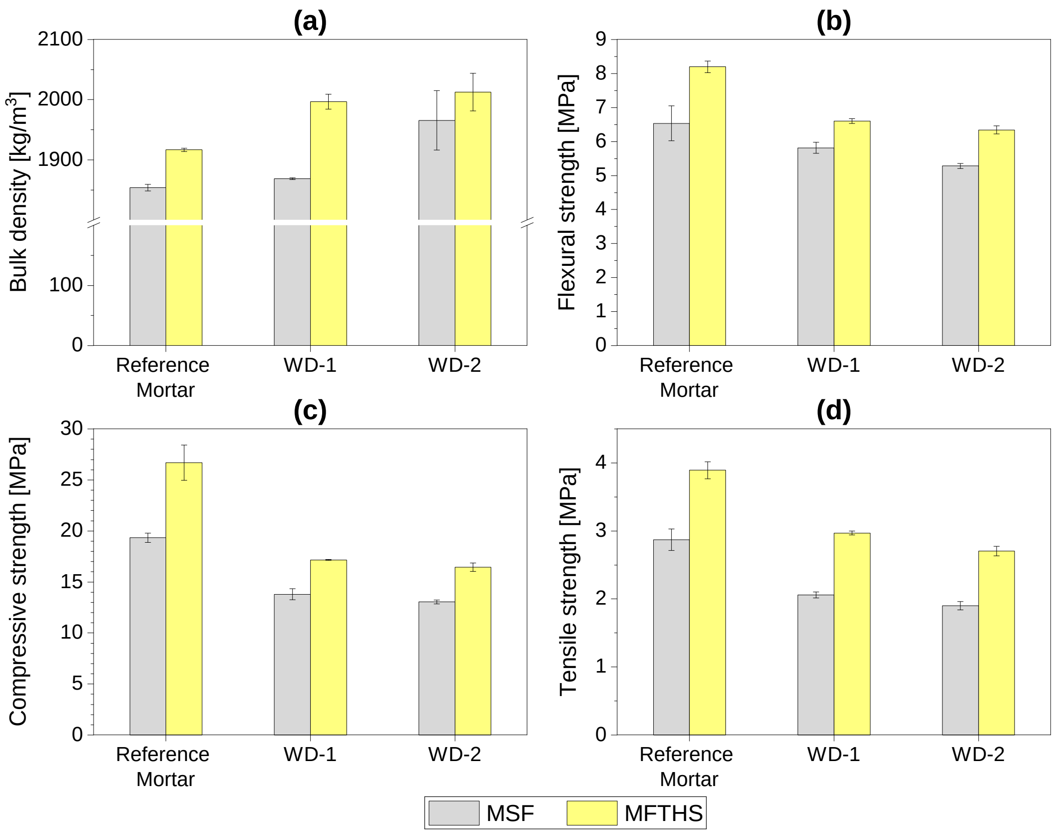

Figure 4.9. Results of mechanical properties at 28 days for (a) dry bulk density, (b) flexural strength, (c) compressive strength, (d) tensile strength. MSF: mortar with no fiber. MFTHS: alkali treated fiber reinforced mortar. WD-1: wet/dry cycle method 1 (Neves Junior, Ferreira, et al., 2019). WD-2: wet/dry cycle method 2 (Wei et al., 2016).

Regarding Figure 4.9 (b) it can be seen that MFTHS, samples subjected to both processes WD-1 and WD-2 have higher results in terms of flexural strength, 12% and 17% respectively if compared to MSF, it can be stated that the addition of abaca alkali treated fiber improves internal bonding and the matrix enhances load transfer and prevents fiber-matrix debonding or pullout during loading (Miah et al., 2023). However, results show that WD-2 has lower strength compared to WD-1, apparently the 6 cycles applied at 70°C of immersion and oven dried affects more than keeping samples to lower temperatures (30°C).

4.3.3.3. Compressive strength

Analyzing compressive strength in Figure 4.9 (c), it shows the same tendency as in flexural strength, where MSF and MFTHS samples subjected to WD-1 and WD-2 are lower in terms of strength when compared to 28 days, this is logical due to the great impact of aging processes where the water absorption by the matrix could lead to swelling and dimensional changes that could weaken the bond between the matrix and the fiber. On the other hand, MFTHS samples show higher strength in terms of compressive strength by both processes than MSF, 19.5% and 21% respectively. Poletanovic et al., (2021) the findings indicate that subjecting alkali treated hemp to w/d cycles led to an enhancement in compressive strength compared to MSF, although it decreased in comparison to the 28-day mark (Poletanovic et al., 2021). Incorporating alkali-treated abaca fiber enhances internal bonding, leading to a denser and more compact matrix structure, thereby reducing void presence and enhancing material density, as previously investigated (Bheel et al., 2021).

4.3.3.4. Tensile strength

In terms of tensile strength, the same behavior occurs, as shown in Figure 4.9 (d). It can be seen that MSF and MFTHS when subjected to WD-1 and WD-2 decrease its strength when compared to samples at 28 days without aging processes. In both aging processes mortars reinforced with treated abaca fiber show higher strength in terms of tensile strength approximately 30% in both processes, as Wei et al., (2016) results after the addition of fiber in different percentages. It can be said that the presence of treated abaca fiber contributes to better performance of these mortars, the addition of fiber could if not treated properly affect this bonding or not being able to stand the heat of treatment. In both cases, it can be seen that tensile strength is higher when fibers were involved.

4.3.3.5. Shrinkage

The behavior of MSF and MFTHS after applying both aging process of WD-1 and WD-2 is as Figure 4.10 shows. At first sight, it is important to remark that within 28 days the main problem is to counteract the shrinkage that is natural because of the water loss, which is improved with the used of abaca alkali treated fiber. After 28 days, aging processes affect directly to the sample since they are subjected to aggressive cycles of w/d and different temperatures in short term. Within this research samples reached 28 days, at this day mark process of w/d WD-1and WD-2 were applied to those samples. Shrinkage was measured up to 28 days and after finishing the cycles of w/d, during the process of WD-1 and WD-2 the percentage of mass loss was measured at each cycle.

MFTHS show better behavior to these processes whether WD-1 or WD-2; in both scenarios the expansion of these mortars as a result of aging process is more controlled and reduced if compared to MSF. The average expansion of MSF subjected to WD-1 is around 0.0316% while MFTHS-WD-1 is 0.011%, between ranges of 0.0215%-0.0503% and 0.0026%-0.00246% respectively.

The same pattern occurs with WD-2, the average expansion of MSF is 0.0691% and 0.0466% for MFTHS within a range of 0.0607%-0.0779% and 0.0398%-0.0574% respectively. After subjecting samples to process of WD-1 and WD-2, a different behavior occurred, expansion in samples. It is well known that cement paste shrinks due time, leading to shrinkage cracking in the cement mortar due to a volume change which could be caused by a variation in temperature, within both process of w/d, samples were subjected to aggressive changes of temperature in short periods of time; as it is seen in Fig 10 samples that weren't subjected to these aggressive cycles of w/d keep a slope that tend to maintain, but all samples that were subjected to WD-1 and WD-2 vary and tend to expand, according to some literature review, shrinkage cracking of cement based materials can be improved by incorporating fibers (Amran et al., 2022). which augment the dimensional stability and control cracking. These results show that the adhesion of treated abaca fiber improves the shrinkage behavior allowing the formation of cracks, but in a more controlled way resulting in a better performance.

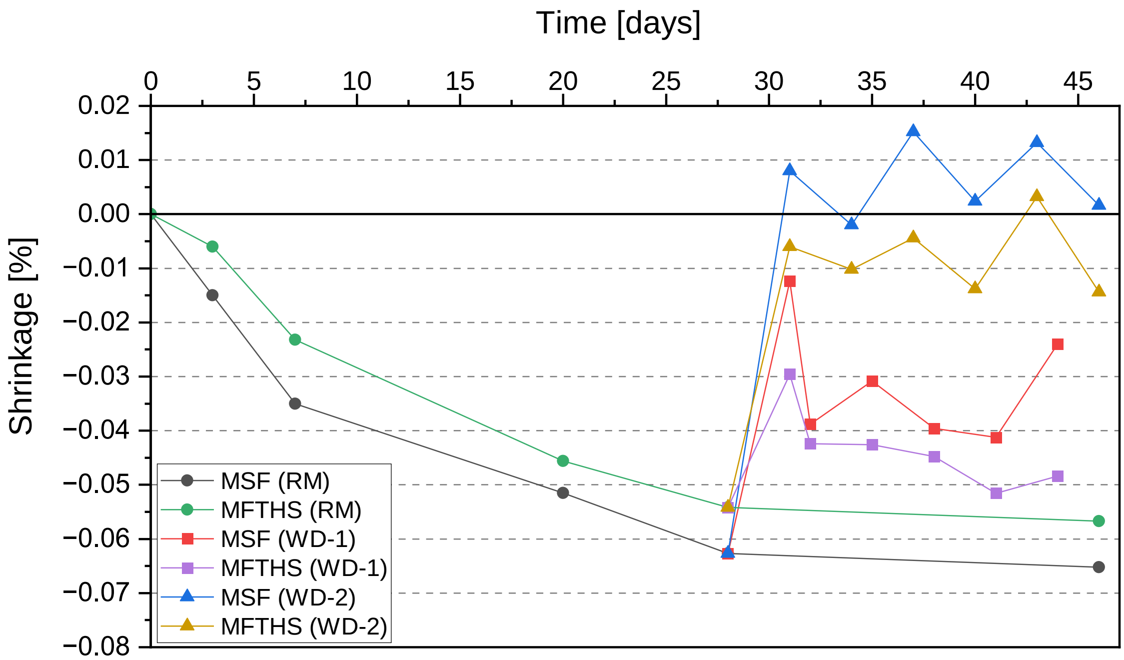

Figure 4.10. Results of shrinkage in RM: reference mortar. MSF: mortar with no fiber. MFTHS: alkali treated fiber reinforced mortar. WD-1: wet/dry cycle method 1 (Neves Junior, Ferreira, et al., 2019). WD-2: wet/dry cycle method 2 (Wei et al., 2016).

4.4. Conclusions

This study presented the analysis of a treated alkali abaca fiber when subjected to accelerated aging process of w/d. As well as the comparison on: mechanical properties between a reference mortar (RM) and an alkali treated fiber reinforced mortar (MFTHS) at 28 days, and after subjecting both type of samples to accelerated aging process of w/d WD-1 and WD-2. From this investigation, it can be concluded that:

- Despite the usage of accelerated aging process of w/d WD-1 and WD-2, the alkali treated fiber preserves the same chemical composition as FT-IR results show, resulting in minimum variation peaks. These results can be correlated with SEM analysis where no great variation was displayed between samples.
- From both aging processes of w/d, results show that WD-2 is more aggressive to samples, even if 6 cycles were applied to each one, the higher temperature and the dipping time of WD-2 diminished its properties if compared to WD-1 for MFS and MFTHS.
- When correlating results from flexural, compressive and tensile strength (Fig 9) to results from Fig 6, it's apparent that better outcomes are achieved with MFTHS. However, within WD-1 and WD-2 better results are attained with WD-1. This behavior may be attributed to surface modifications and fiber bonding, which appear to be enhanced when subjected to WD-1.
- The addition of alkali treated abaca fiber to the bio composite resulted in a 5% more compact sample, all mechanical properties increased compared to reference mortar, resulting in a 20% more resistant mortar in terms of flexural strength, 28% in terms of compressive strength and 26% in terms of tensile strength

- In terms of shrinkage, after subjecting both samples to most critical scenario (not curing), results show that the addition of alkali treated abaca fiber improved with the volume expansion and loss of water of the cementitious mortar resulting in a more controlled behavior.
- After applying accelerated aging process of w/d WD-1 and WD-2 results in terms of dry bulk density, flexural, compressive and tensile strength show that even after applying w/d cycles, MFTHS WD-1 increased their mechanical properties resulting in a 6% more compact mortar, 11% more resistant to flexural strength, 20% higher for compression and 30% higher for tensile strength, and MFTHS WD-2 resulted in a 2% more compact mortar, 17% more resistant to flexural strength, 21% higher for compression and 30% higher for tensile strength. In terms of shrinkage the tendency on this behavior is reflected.

Chapter 5. Characterization and life cycle assessment of alkali treated abaca fibers: the effect of reusing sodium hydroxide[3]

[3] The result presented in this chapter were published in Alcivar-Bastidas, S., Petroche, D. M., Ramirez, A. D., & Martinez-Echevarria, M. J. (2024). Characterization and life cycle assessment of alkali treated abaca fibers: the effect of reusing sodium hydroxide. Construction and Building Materials, 449, 138522. https://doi.org/10.1016/j.conbuildmat.2024.138522

5.1. Introduction

The application of natural fibers has brought benefits to various industries, and their use has been growing (Ismail et al., 2022). Natural fibers possess key properties such as low density, sustainability, high availability in nature, cost-effectiveness, reduced dependence on non-renewable energy/material sources, and relatively high specific strength (Pickering et al., 2016). Natural fibers are essential for the production of comfortable and breathable garments in the textile industry (Islam et al., 2020). Natural fibers are used in the automotive and aerospace industries to develop lighter and biodegradable components (Awais et al., 2021). They are used in the fabrication of composites and reinforcement of construction materials (Araya-Letelier et al., 2021; Laverde et al., 2022; Saini et al., 2024; Trochoutsou et al., 2021), offering the additional benefit of potentially reducing the carbon footprint of cement composites (Ali et al., 2022). These examples reflect the growing interest in the use of natural fibers as sustainable alternatives in various industrial applications.

Natural fibers are compared to polymer fibers because both address key challenges regarding traditional cementitious matrices, considering that concrete has low tensile strength and poor energy dissipation capacity, which makes it prone to cracking (A.Abusogi & Bakri, 2022); among polymer fibers, polypropylene (PP) is particularly valued in the concrete industry owing to its ease of processing and cost-effectiveness (Deng et al., 2022).

Natural fiber treatments significantly contribute to improving their intrinsic properties and expanding their versatility in various industrial applications. These treatments primarily aim to improve the adhesion and compatibility between natural fibers and polymeric matrices in composite materials (Castoldi et al., 2022; Lin et al., 2023). Despite these advantages, many challenges arise concerning the use of natural fibers in applications, such as poor dispersion in the matrix and, most critically, the low durability of fibers in the matrix (Boulos et al., 2019). Hence, two strategies have been developed regarding whether to modify the matrix (X. Li et al., 2007) or fiber by applying different treatments (Boulos et al., 2019; Z. Zhao et al., 2018).

Among the treatments intended for composite applications, alkaline treatment is notable, in which the fibers are subjected to a sodium hydroxide (NaOH) solution to modify their surface and enhance adhesion with polymeric matrices (Samanth & Subrahmanya Bhat, 2023; Yan et al., 2016). Additionally, treatments with silane coupling agents aim to establish chemical bonds between the fibers and resins to optimize the load transfer and improve the mechanical properties of the composite (X. Li et al., 2007; L. Zhao et al., 2024). Other treatments, such as thermal, enzymatic, and peroxide treatments, have also been used to adjust the characteristics and compatibility of natural fibers with matrices. These treatments enable the production of natural fiber-reinforced composites with specific properties tailored to diverse applications (Vo & Navard, 2016).

5.1.1. *NaOH consumption in the alkali treatment of natural fibers*

Alkaline treatment or mercerization is one of the most widely used chemical treatments for natural fibers (Maiti et al., 2022). This treatment has been shown to be effective for fibers from coconut, sisal, jute, banana, bagasse, flax, oil palm, and abaca (Samanth & Subrahmanya Bhat, 2023). This process usually involves immersing the fibers in alkaline solutions, typically NaOH, at solution concentrations ranging from 1–50% (Das & Chakraborty, 2006; K. Liu et al., 2014); the treatment can be conducted at room temperature or elevated temperatures (Pickering et al., 2007; K. Zhang et al., 2018), with exposure times varying from 30 min to 72 h (S. V. Prasad et al., 1983; Vilay et al., 2008). Subsequently, the mercerized fibers are washed with water to remove any residual alkaline content and then neutralized to prevent further degradation (Iucolano et al., 2015; Shahril et al., 2022). Finally, the fibers are dried at room temperature (Alcivar-Bastidas et al., 2023; Rajeshkumar, 2020) or at temperatures ranging from 50 to 105°C (Vilaseca et al., 2007; Yan et al., 2016), for varying periods of time, typically between 6 and 72 h (Cao et al., 2006; Sarkar & Ray, 2004).

However, this procedure can lead to a significant consumption of NaOH, which not only affects production costs but also raises environmental concerns (S. Kim et al., 2008). The quantity of NaOH required per kilogram of natural fiber depends on the solution concentration and fiber type. For example, low-concentration NaOH solutions (1%) consume 0.15–0.20 kg of NaOH per kg of natural fiber. At common concentrations (3–5%), NaOH consumption ranges between 0.44 and 1.90 kg of NaOH per kg of natural fiber, while at high concentrations of NaOH (10–50%), up to 6.67 kg of NaOH per kg of natural fiber may be required. This consumption also depends on the type of natural fiber and fiber solution ratio, as shown in Table 5.1.

Consequently, the main environmental challenge of alkaline treatment of natural fibers is the production of sodium hydroxide (NaOH) through electrolysis, which is an energy-intensive process (S. Kim et al., 2008). This has a significant environmental impact on the treatment processes. Additionally, when fibers are removed from the alkaline bath, the generation of wastewater adds environmental concerns in terms of the treatment itself and the disposal of effluents (Dam & Bos, 2004). Together, these factors create the need for more sustainable practices in the alkaline treatment of natural fibers.

Several studies have focused on the reuse of NaOH solution from the natural fiber surface treatment process as part of a circular economic strategy. This approach aims to reduce both NaOH consumption and wastewater generation (Ita-Nagy et al., 2020; Juradin et al., 2023; S. Kim et al., 2012; X. Zhao et al., 2024). A 40% NaOH solution effectively removed lignin from empty palm fruit bunch fibers, and the solution was reusable for multiple lignin extractions (S. Kim et al., 2012). A recent study explored the reuse of NaOH solution in the treatment of Spanish broom fibers with 15% NaOH. They concluded that the maceration effect reduced the lignin content in the fibers, and the same alkali solution could be used for at least five preparations (Juradin et al., 2023).

Table 5.1. Sodium hydroxide consumption per 1 kg of natural fiber in the reviewed literature.

Fiber	Fiber: Solution ratio[a]	Alkaline treatment (%)														Reference
		1	2	3	4	5	6	7	8	9	10	12	15	20	5	
		Sodium hydroxide (kg)														
Abaca	1:28	-	-	0.8	-	-	-	-	-	-	-	-	-	-	-	(Alcivar-Bastidas et al., 2023)
Abaca	1:33	-	0.6	-	-	-	-	-	-	-	-	-	-	-	-	(Iucolano et al., 2015)
Abaca	1:30	0.3	-	-	-	1.4	-	-	-	-	-	-	-	-	-	(K. Liu et al., 2014)
Abaca	1:15	-	-	-	-	0.7	-	-	-	-	1.3	-	1.9	2.5	-	(Punyamurthy et al., 2012)
Alfa	1:15	0.1	-	0.4	-	0.7	-	0.9	-	-	-	-	-	-	-	(Benyahia et al., 2013)
Bagasse	1:20	0.2	-	0.5	-	0.9	-	-	-	-	-	-	-	-	-	(Cao et al., 2006)
Bamboo	1:20	-	0.3	-	-	-	1.1	-	-	-	1.8	-	-	-	-	(K. Zhang et al., 2018)
Bamboo	1:15	-	-	-	-	-	-	-	-	-	1.3	-	1.9	2.5	5	(Das & Chakraborty, 2006)
Banana	1:15	0.1	-	-	-	-	-	-	-	-	-	-	-	-	-	(N. Prasad et al., 2016)
Coir	1:23	-	-	-	-	1.1	-	-	-	-	-	-	-	-	-	(S. V. Prasad et al., 1983)
Mauritius hemp	1:20	-	-	0.5	-	-	1.1	-	-	1.6	-	2.1	2.6	-	-	(Shahril et al., 2022)
Hemp	1:06	-	-	-	-	-	-	-	-	-	0.5	-	-	-	-	(Pickering et al., 2007)
Hemp	1:15	-	-	-	-	0.7	-	-	-	-	-	-	-	-	-	(Gupta et al., 2018)
Hemp	1:55	-	-	-	-	-	-	-	-	-	5	-	-	-	-	(George & Bressler, 2017)
Hemp	1:20	-	-	-	-	0.9	-	-	-	-	-	-	-	-	-	(Sawpan et al., 2011)
Hemp, Flax	1:40	-	-	1.1	-	1.9	-	-	2.9	-	3.6	4.2	5.2	6.6	-	(Ouajai et al., 2004)
Jute	1:15	-	-	-	-	0.7	-	-	-	-	-	-	-	-	-	(Sarkar & Ray, 2004)
Jute	1:15	-	-	-	-	0.7	-	-	-	-	-	-	-	-	-	(Ray et al., 2001a, 2001b)
Kenaf	1:20	-	-	-	-	0.9	-	-	-	-	-	-	-	-	-	(S. Kim et al., 2008)
Palm (Phoenix sp.)	1:20	-	-	-	-	0.9	-	-	-	-	1.8	-	2.6	3.3	-	(Rajeshkumar et al., 2020,
Doum palm	1:20	0.2	0.3	0.5	0.7	-	1.1	-	1.4	-	-	-	-	-	-	(Zannen et al., 2014)
Royal palm	1:25	-	-	-	-	1.1	-	-	-	-	-	-	-	-	-	(Goud & Rao, 2011)
Pineapple, ramie,	1:100	-	1.9	-	-	-	-	-	-	-	-	-	-	-	-	(Munawar et al., 2008)
Sisal	1:25	-	0.4	-	-	-	-	-	-	-	-	-	-	-	-	(Rong et al., 2001)
Sisal	1:20	-	-	-	-	-	-	-	-	-	1.8	-	-	-	-	(X. Zhao et al., 2024)

[a] By mass No reported: -

Reusing NaOH solutions in different applications has proven to be effective in enhancing material properties and offering some environmental benefits. In one approach, sisal fibers treated with 10% NaOH and incorporated into alkali-activated slag-based composites demonstrated improved flexural and compressive strengths with reduced electrical conductivity and density using the residual NaOH solution as an activator (X. Zhao et al., 2024). Another method involves residual bagasse fibers for use in the production of fiber-reinforced biocomposite pellets, where a NaOH recovery process allows for the recycling of up to 61% of the NaOH used, although the reduction in carbon footprint is 1% (Ita-Nagy et al., 2020).

For these reasons, circularity measures, such as reducing chemical consumption and wastewater generation, can be implemented to mitigate the environmental impacts associated with the production of treated natural fibers. Several researchers have proposed the use of life cycle assessments (LCA) to assess the effectiveness of cleaner production methods and circular economy alternatives. (Peña et al., 2021; Petroche & Ramirez, 2022; Yaseen et al., 2024).

According to the FAO (FAO, 2022), abaca fiber ranks as the sixth most produced natural fiber worldwide, with an annual production of 107 kt in 2022, ahead of jute, flax, hemp, kenaf, and sisal. Abaca plays a crucial role in the production of paper, monetary paper, textiles, ropes, packaging, and in the automotive industry because of its strength and durability (FAO, 2012; Simbaña et al., 2020). It shows promising results in construction applications in the reinforcement of cement composites, where it enhances tensile strength, crack resistance, and durability (Alcivar-Bastidas, Petroche, Cornejo, et al., 2024; Lee & Choi, 2022). Despite their significant industrial importance, the environmental impact of abaca fibers remains underexplored compared to that of other fibers such as jute, flax, hemp, and kenaf (Faist Emmenegger, Bajaj, et al., 2018; La Rosa & Grammatikos, 2019; Wötzel et al., 1999; Zampori et al., 2013; Zhou et al., 2018). Therefore, it is essential to generate a detailed life cycle inventory and environmental profile for abaca to better understand and quantify its environmental implications across various applications.

The objectives of this research are (i) to study the effect of recirculation and reuse of NaOH solution for the treatment of natural fibers and analyze its influence on the properties of abaca fibers, (ii) to generate life cycle inventories for the treatment process of natural abaca fibers with NaOH solution, (iii) to quantify the carbon footprint of the NaOH treatment process for abaca fibers and the proposed recirculation and reuse of residual NaOH solution as a circular economy scenario using a life cycle perspective, and (iv) to evaluate the carbon footprint results in the context of polypropylene fibers as a conventional synthetic alternative.

5.2. Materials

5.2.1. Abaca fibers

Abaca natural fibers obtained from the Manila hemp plant (*Musa textilis*) are known for their strength, durability, and versatility (K. Liu et al., 2013). Their water resistance and ability to withstand biological degradation make them particularly suitable for applications that require robustness and longevity (Alcivar-Bastidas, Petroche, Cornejo, et al., 2024;

Dittenber & GangaRao, 2012). In the construction industry, when applied to cement-based composites, they act as a reinforcement, improving the tensile and flexural strength (Anthony et al., 2020; Tampi et al., 2020) and potentially serving as a replacement for synthetic fibers. The abaca fibers used in this study were cultivated in Ecuador, the second-largest exporter of abaca worldwide (FAO, 2022).

5.2.2. Abaca NaOH treated fiber

Alkaline treatment of the natural abaca fibers was performed as described by Alcivar-Bastidas et al., (2023). This involved preparing a 3% NaOH solution in distilled water and immersing the natural fibers in a fiber:solution ratio of 1:28 by mass for 4 h. Subsequently, the fibers were removed from the solution and washed with tap water until the rinse water became clear. Finally, the fibers were dried in an oven at 85 ± 1 °C for 24 h, after which they were stored and packaged at room temperature. The materials and quantities used are listed in Table 5.2. Natural abaca fibers and abaca fibers treated with 3% NaOH have a length of 30 mm, which is the optimal size for application in masonry mortar cement composites (Alcivar-Bastidas et al., 2023).

Table 5.2. Material quantities for abaca fiber alkali treatment with 3% NaOH solution.

Material	Weight (g)
Input	
Deionized water (solution)	1000
NaOH	30
Abaca natural fiber	36.1
Tap water (wash water)	4326
Output	
Abaca treated fiber	28.7

5.2.3. Polypropylene fiber

Polypropylene fibers are synthetic fibers manufactured by the extrusion of polypropylene. These fibers act as reinforcements to improve the mechanical properties of cement-based composites (Jia et al., 2021; Labib, 2018). In this study, the polypropylene fibers were made of 100% virgin material. The fibers were made according to the ASTM C1116 standard (ASTM International, 2023a), for which the specifications were as follows: 19 mm length, 0.03–0.05 mm diameter, tensile strength between 300–350 MPa, 910 kg/m^3 density, and high resistance to alkalinity (Sika Colombia, 2022).

5.3. Methods

5.3.1. Reuse of NaOH solution in alkali treatment of abaca fiber on a laboratory scale

As part of the circular economy initiative, a scheme for the recirculation and reuse of the NaOH solution is proposed, as shown in Figure 5.1, where the solution is reused after fiber immersion. An experimental testing framework was defined, and treatment 1 (T1) was initiated, as described in Section 5.2.2.

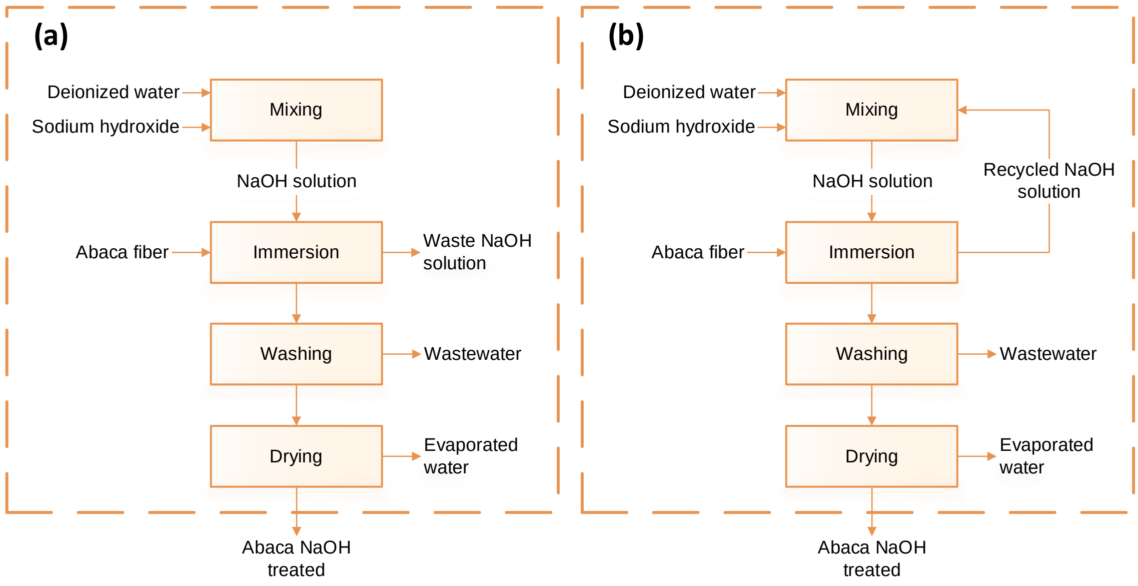

Figure 5.1. Flow chart of alkaline treatment of abaca fiber. (a) Conventional treatment. (b) Proposal for recirculation and reuse of NaOH solution.

Subsequently, the residual NaOH solution became the initial solution for the second cycle of treatment (T2), maintaining the material proportions shown in Table 5.2, a fiber:solution ratio of 1:28, and a fiber:wash water ratio of 1:120 by mass. This procedure was repeated until the maximum number of treatments with the same solution was achieved.

5.3.1.1. Titration Method for measuring NaOH solution concentration

The standard Titration Method described in the Standard Methods for the Examination of Water and Wastewater was used to determine the NaOH solution concentration in all samples, as described in Section 2320 B. Titration Method (American Public Health Association et al., 2023).

The method involves titrating the sample with standard sulfuric acid (H_2SO_4) and chemical indicators (phenolphthalein). The volume of acid used was recorded, and the alkalinity was calculated using a formula that accounts for the volume of acid, its normality, and sample size. The result was expressed in milligrams per liter as calcium carbonate ($CaCO_3$), providing a reliable measure of the water's buffering capacity. Finally, a known solution of NaOH was measured and compared to the $CaCO_3$, and a conversion factor was used to determine the concentration of NaOH in percentage (%).

5.3.2. Fiber properties tests

5.3.2.1. Thermogravimetry/Derivative Thermogravimetry Analysis (TGA/DTG)

Thermogravimetric analysis was conducted on untreated and treated abaca fibers. This was performed by comparing the rate of weight loss and decomposition temperature to observe changes in the fiber mass. This test indicated the thermal decomposition, moisture content, and thermal stability of the fibers. The analysis was developed with the use of SDT Q600 Simultaneous Thermal Analyzer equipment, under a ramp rate of 15°C/minute from room temperature to 104°C and 50°C/minute from 104°C to 1000°C. This test was performed as described in the studies by Alcivar-Bastidas et al., (2023), Shahril et al., (2022), S. Fu et al., (2017), and Kathirselvam et al., (2019). The effectiveness of thermal treatments on natural fibers, such as the removal of lignin and hemicellulose, has been observed (Akram Khan et al., 2011; Ouajai & Shanks, 2005; Oushabi et al., 2017).

5.3.2.2. Scanning Electron Microscope (SEM)

To analyze the morphology of the fibers, an SEM test was performed using a FEI Inspect® scanning electron microscope, and the recommendations from similar studies by Wei et al., (2016, 2017) were followed. This test visually provides the surface roughness, porosity, and fiber wall structure, allowing an examination of the removal of substances such as impurities, wax, hemicellulose, lignin, and fatty acids due to alkaline treatment (N. Prasad et al., 2016; Shahril et al., 2022).

5.3.2.3. Tensile test

The tensile test used to determine the tensile strength of untreated abaca fibers and alkaline-treated fibers with different treatment cycles was performed according to Cai et al., Cai et al., (2015, 2016) and Alcivar-Bastidas et al., (2023). This involved placing a fiber in a sample holder, which was secured with epoxy at the bottom. It was then subjected to tension at a velocity of 1 mm/min using a universal testing machine Shimadzu AGS-X with a load cell of 500 N. The tensile strength at breaking point was obtained from the testing machine, and the diameter was obtained by the SEM test described above using Motic Images Plus 3.0. Also, an analysis of variance (ANOVA) was performed, with $\alpha=0.05$ and 30 samples (n=30) for each scenario. This analysis was performed to determine if there were significant differences in tensile strength between the treatments (Romli et al., 2012). It is considered essential to statistically evaluate the behavior of the natural abaca fiber (NAF), single-use of NaOH solution (T1) samples and the solution reuse treatments, with the main purpose of determining the extent to which the use and recycling processes remained effective.

5.3.3. Life cycle assessment

Life Cycle Assessment (LCA) is a methodology used to evaluate the environmental impacts associated with a product's life cycle, from raw material extraction through production, use, disposal, and recycling (Salas et al., 2016). By conducting LCA, it is possible to identify areas where improvements can be made to reduce the environmental footprint of products or processes. ISO 14040 and ISO 14044 are LCA standards that provide the principles and framework for conducting LCA studies (International Organization for Standardization, 2006a, 2006b). This framework includes (i) defining the goal and scope and establishing objectives and boundaries; (ii) conducting a life cycle inventory (LCI) and collecting data on inputs and outputs; (iii) performing a life cycle impact assessment (LCIA) and evaluating environmental impacts; and (iv) interpretation and drawing conclusions for decision-making and improvement.

5.3.3.1. Goal and scope definition

The natural abaca fiber and alkali-treated abaca fiber within this study are considered for use in cement-based composites, in accordance with ASTM C1116 (ASTM International, 2023a), as an alternative to polypropylene fibers; therefore, they are defined as construction products. A cradle-to-gate scope is defined according to Product Category

Rules (PCR) for construction products in EN 15804:2012 (European Committee for Standardization, 2012). These include A1, raw material supply, A2, transport, and A3, manufacturing.

Abaca is produced globally, with the Philippines being the leading producer, accounting for 63.56% of abaca production, followed by Ecuador at 34.50%, and other countries at 1.94% (FAO, 2022). In this study, the processes of abaca cultivation, abaca fiber production, and alkaline-treated abaca fiber production were associated with the geographic scope of Ecuador. The polypropylene fiber was associated with the geographical scope of the US, which is the origin of the fibers according to the manufacturer.

5.3.3.1.1. System boundaries

Four systems are studied:

i. Natural abaca fiber (NAF) production (Figure 5.2. (a)): The system covers two phases: abaca and fiber production. Abaca production involves seedling production, growth until the seedlings are transplanted into the crop, and forming groups of stems that grow from the base (Figure 5.3. (a)). The abaca plant matures within a period of 18–24 months and can be harvested thrice a year (Lalusin & Villavicencio, 2015). Harvesting involves cutting the stems approximately 10 cm from the ground with an outward beveled cut to prevent the plant from rotting (Simbaña et al., 2020). After cutting, the leaves are removed and the harvested pseudostems are grouped. Tuxiying then begins, which involves cutting the pseudostem until it reaches the central zone (tuxy) containing the biomass to be transformed into a fiber (Göltenboth & Mühlbauer, 2010). In the second phase of fiber production, the tuxies are stripped using a mechanism consisting of a diesel engine, pulleys, and blades (Figure 5.3. (b)). Finally, the fibers are dried in the sun for 1–3 days (Figure 5.3.c).

ii. Treated abaca fiber – conventional treatment (TAF-CT) production (Figure 5.2. (b)): This system covers natural abaca fiber production plus conventional 3% alkali treatment as described in Section 5.2.2.

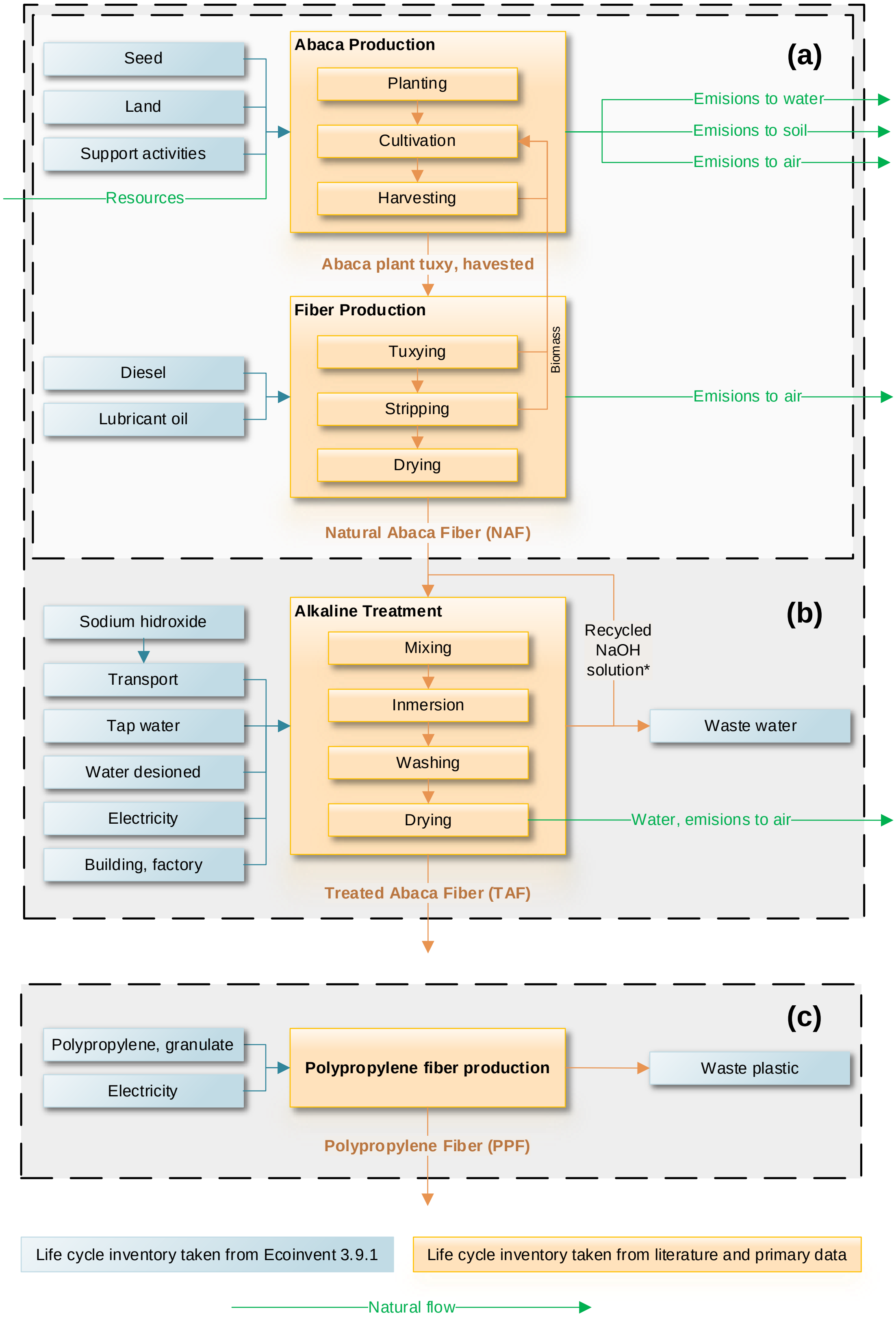

Figure 5.2. Cradle-to-gate system boundary for the production of (a) abaca fiber, (b) abaca fiber treated and (c) polypropylene fiber. * Included in reuse case study.

iii. Treated abaca fiber – reuse treatment (TAF-RT) production (Figure 5.2. (b)): This system is a variant of TAF-CT. This includes NAF production plus 3% alkaline treatment with recirculation of the NaOH solution, as proposed in Section 5.3.1.

iv. Polypropylene fiber (PPF) production (Figure 5.2. (c)): This process begins with the input of polypropylene granulates, which are produced through the polymerization of propylene monomers (Yin et al., 2016). This product serves as the primary raw material for the fiber manufacturing process. Electricity is crucial for powering extrusion, spinning, and other machinery involved in converting polypropylene into fibers (Van den Heede et al., 2018).

The functional unit was 1 kg of fiber, with four different scenarios depending on the system boundaries: 1 kg of natural abaca fiber, 1 kg of abaca fiber treated with a 3% NaOH solution, 1 kg of abaca fiber treated with a reused 3% NaOH solution, and 1 kg of polypropylene fiber.

Figure 5.3. Abaca fiber production stages in Santo Domingo-Ecuador: (a) group of abaca trees, (b) stripping process, and (c) drying process.

5.3.3.2. Life Cycle Inventory

5.3.3.2.1. Natural abaca fiber production

The Ecoinvent LCI calculation tool for crop production (Ecoinvent, 2018) was used to model the life cycle inventory (LCI) of abaca production. It employs various models to calculate emissions from field activities, land transformation and occupation, irrigation, and carbon uptake by plants (Faist Emmenegger, Délerce-Mauris, et al., 2018). This tool requires agricultural and meteorological inputs from the plantation sites.

The agricultural input was obtained from the Ecuadorian Survey of Surface and Continuous Agricultural Production (ESPAC 2022) (Instituto Nacional de Estadística y Censos INEC, 2023c). This survey used the multiple frame sampling methodology, and for abaca production, a sample of farms that represented 24.35% of the total abaca fiber produced was considered (Instituto Nacional de Estadística y Censos INEC, 2023a). An expansion factor was then applied to obtain the total results for all items surveyed for all abaca fibers produced in Ecuador (Instituto Nacional de Estadística y Censos INEC, 2023b). The information surveyed included planted and harvested areas, harvested tons, use of irrigation, and the quantity and type of fertilizers and pesticides (Instituto Nacional de Estadística y Censos INEC, 2023c). According to the ESPAC survey, there are seven types of abaca plantations that vary according to the use of fertilizers and pesticides, depending on the organic or inorganic origin of these substances. The predominant plantation does not involve the use of fertilizers or pesticides, representing 54.93% of the Ecuadorian abaca harvest (Instituto Nacional de Estadística y Censos INEC, 2023a). Owing to its representativeness, the current study was limited to modeling this type of plantation.

Meteorological data were obtained from the Ecuadorian National Institute of Meteorology and Hydrology (Instituto Nacional de Meteorología e Hidrología INAMHI, 2017). The selected meteorological station is located in the province of Santo Domingo, which accounts for 90% of Ecuadorian abaca production (Instituto Nacional de Estadística y Censos INEC, 2023a).

To generate inventories of biomass and organic waste, the average biomass distribution in the abaca plant (Figure 5.4. (a)) was obtained from Cortez et al., (2015): leaves (16.36%), pseudo-stem waste (63.79%), and tuxy (19.85%). Seven farms in Santo Domingo-Ecuador were visited, from which it was evident that organic residues such as leaves, pseudo-stem waste, and stripping waste were left in the plantation. According to farmers, this is a common practice in Ecuador. The biomass serves as a nutrient source for new plants (Choo et al., 2011; Wiloso et al., 2015). Owing to the absence of anaerobic conditions, CH_4 emissions were not generated (Roibás et al., 2016). In this study, the waste biomass was considered to remain within the system; this approach has been used in studies of other plantations under similar conditions (Choo et al., 2011; Cortez et al., 2015; Roibás et al., 2016).

To model the inventories of energy in biomass and carbon dioxide uptake by biomass for abaca production, the Ecoinvent LCI calculation tool for crop production requires a reference crop. In this study, banana (*Musa* spp.) was selected as the base crop because of its close botanical relationship with abaca (*Musa textilis*), as both belong to the *Musaceae* family (Mamun et al., 2015; Richter et al., 2013). Additionally, they are cultivated in the same region in Ecuador (Ortiz-Ulloa et al., 2021) and exhibit similar biomass distribution patterns (Figure 5.4); in the case of banana, the main product is the fruit, while for abaca is the tuxy. As the biomass distribution is a critical input for the Ecoinvent LCI calculation tool (Faist Emmenegger, Délerce-Mauris, et al., 2018), this similarity in biomass distribution makes banana an appropriate reference crop .

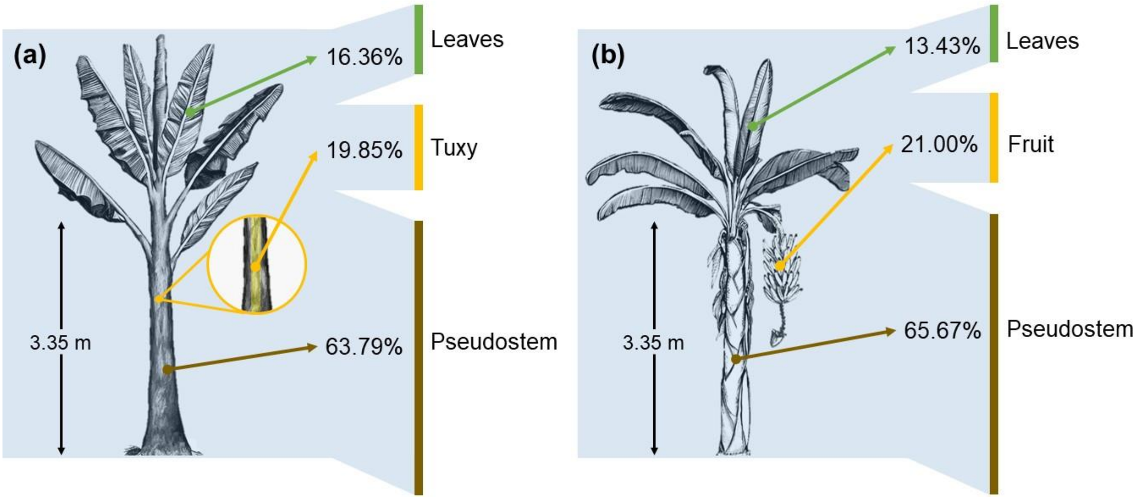

Figure 5.4. Average distribution of biomass of (a) abaca plant (*Musa textilis*) (Cortez et al., 2015; Göltenboth & Mühlbauer, 2010) and (b) banana plant (*Musa* spp.) (Fiallos-Cárdenas et al., 2022; Ortiz-Ulloa et al., 2021).

Dinitrogen monoxide (N_2O) from the crop residues for abaca production was calculated using the IPCC Guidelines (IPCC, 2019), yield from ESPAC 2022 (Instituto Nacional de Estadística y Censos INEC, 2023c), biomass distribution from Cortez et al., (2015), and moisture and nitrogen content (Armecin, 2008; Armecin & Gabon, 2008). Indirect N_2O emissions from leaching and runoff were estimated using IPCC Guidelines (IPCC, 2019). The leaching-runoff factor was calculated according to Franke et al., (2013) and replaced $Frac_{LEACH}$ in the IPCC calculations. This approach was used in a previous carbon footprint study of bananas in Ecuador (Roibás et al., 2016).

Finally, the inventory of fuel and lubricant consumption for the fiber stripping and drying process was collected from the seven farms visited. The fuel was transformed into energy units using the factors for Ecuadorian diesel (density: 850 kg/m^3; heat capacity: 40.8 MJ/kg) (MERNNR et al., 2020).

5.3.3.2.2. Treated abaca fiber production

The inventory of raw materials for the abaca alkaline treatment fiber process using a 3% NaOH solution and the proposal for recirculation and reuse of the NaOH solution were taken from the results provided in this study, in Section 5.2.2 and Section 5.4.1, respectively. Facilities and energy inventories for the alkaline treatment fiber process were taken from the "mercerizing process, textile – IN" detailed in Ecoinvent 3.9.1 (Wernet et al., 2016) and Faist Emmenegger, Bajaj, et al., (2018). This process was adapted to Ecuadorian electricity, and the LCA process of Ramirez et al., (2019, 2020) was used. The NaOH supplier closest to Guayaquil is located in Callao, Peru (Quimpac, 2021). Sodium hydroxide and sodium chloride from Ecoinvent 3.9.1 were adapted for Peruvian electricity.

5.3.3.2.3. Polypropylene fiber production

The life cycle inventory of raw materials for polypropylene fiber production was obtained from studies by Yin et al., (2016) and Van den Heede et al., (2018). Processes for polypropylene granulate production, electricity, and waste treatment were obtained from Ecoinvent 3.9.1 (Wernet et al., 2016).

The main parameters and assumptions of this study are summarized in Table 5.3.

Table 5.3. Summary of main parameters and assumptions in this study.

Phase	Parameters and assumptions
Agricultural phase	Geographical scope: Ecuador. LCI models by Ecoinvent LCI calculation tool for crop production. Base crop: Banana. Agricultural inputs: ESPAC 2022 (Instituto Nacional de Estadística y Censos INEC, 2023c). Limited to modeling without fertilizers and without pesticide plantation. Meteorological data: INAMHI 2017 (Instituto Nacional de Meteorología e Hidrología INAMHI, 2017). Biomass distribution: Cortez et al., (Cortez et al., 2015). Moisture and nitrogen content: Armecin et al., (Armecin, 2008; Armecin & Gabon, 2008). Waste management: Organic residues are left in the plantation. Waste biomass remains within the system in anaerobic conditions, CH_4 emissions are not generated by waste biomass (Roibás et al., 2016). N_2O emissions: IPCC Guidelines (IPCC, 2019) and Franke et al., (2013).
Alkali treatment	Geographical scope: Ecuador. Electricity: Ecuador (Ramirez et al., 2019, 2020). Facilities and energy consumption: Ecoinvent 3.9.1 (IN), adapted with Ecuadorian electricity. Sodium hydroxide and sodium chloride production: Callao, Peru (Quimpac, 2021) and Ecoinvent 3.9.1 (US), adapted with Peruvian electricity. Changes in NaOH and wastewater inventories due to the reuse of NaOH solution.
Polypropylene fiber production	Geographical scope: United States. Polypropylene granulate: Ecoinvent 3.9.1 (US). Electricity and waste treatment inventories: Yin et al., (2016) and Van den Heede et al., (2018).

IN: India US: United States

5.3.3.2.4. Life Cycle Impact Assessment

The methodology used for LCIA was the ReCiPe Midpoint (H) version 1.13 (Huijbregts et al., 2017). The impact category of global warming potential (GWP100), also known as the carbon footprint, was selected because it is considered the most critical indicator of climate change (Skodvin, 1999). OpenLCA 2.1.1 (GreenDelta, 2023) was used for LCA calculations.

5.4. Results and discussion

5.4.1. Reuse of NaOH solution in alkali treatment of abaca fiber on a laboratory scale

Table 5.4 shows the experimental results of reusing NaOH solution in the alkaline treatment of natural abaca fibers. Following the methodology described in Section 5.3.1, by maintaining the same proportions of fiber:solution and fiber:wash water materials, each washing cycle preserved a similar behavior as the conventional treatment of a single-use NaOH solution (T1).

Table 5.4. Experimental inventory of materials in 10 cycles of NaOH solution reuse for alkaline treatment.

Cycle	Initial NaOH solution (g)	Abaca fiber (g)	Washing water (g)	Wet fiber (g)	Dry treated fiber (g)	Final NaOH solution (g)	NaOH in final solution (%)
T0	--	--	--	--	--	--	2.98
T1	1030	36.1	4326.0	333.59	28.70	719.34	2.96
T2	719.34	25.2	3021.2	243.92	20.04	512.59	2.95
T3	512.59	17.9	2152.9	162.89	13.83	370.29	2.93
T4	370.29	13.0	1555.2	106.74	10.05	271.77	2.91
T5	271.77	9.5	1141.4	85.05	7.37	196.38	2.89
T6	196.38	6.9	824.8	59.66	5.52	142.56	2.88
T7	142.56	5.0	598.7	44.80	3.99	103.38	2.86
T8	103.38	3.6	434.2	31.64	2.92	74.78	2.47
T9	74.78	2.6	314.1	23.85	2.07	52.57	0.84
T10	52.57	1.8	220.8	16.79	1.52	36.91	0.31

The 10 reuse cycles resulted in a loss of sodium hydroxide solution owing to the treatment process (fiber washing and evaporation), ranging from 26.61% to 30.16%. As a consequence of alkaline treatment, natural fibers experience mass reduction owing to the removal of impurities, and wax, hemicellulose, and lignin are removed (Abdal-hay et al., 2012; Valášek et al., 2021; Yan, 2012). This behavior was observed in T1, with a mass reduction of 20.5%; from T2 to T9, similar values between 18.9% and 20.4% were obtained. T10 exhibited the lowest mass loss of 15.6%.

Washing cycles were stopped at T10 because the solution was lost. Initially, there was a certain amount of solution, considering the 1:28 fiber:solution ratio; however, as the washing cycles continued, the solution was lost, which resulted in inadequate fiber treatment solution for washing cycle T11. After 10 cycles of NaOH reuse, there was a 70.11% reduction in NaOH consumption per kilogram of treated abaca fibers.

The titration test was performed on all samples from T0 to T10, as shown in Table 5.4, where the initial solution concentration in T0 was 2.98% and the NaOH concentration in T10 was 0.31%. The initial value of 2.98% can be attributed to the manual procedure of dissolving NaOH in the solution. After applying the first procedure of using and recycling the water solution, the NaOH concentration in T1 was measured to be 2.96%, which closely resembled the initial concentration of T0. Between T2 and T4, the NaOH concentration showed a slight decrease, ranging from 2.95% to 2.91%, indicating that the recycling process effectively maintained the concentration at levels similar to those at T0.

From T5 to T7, the average NaOH concentration stabilized at 2.88%, which remained within an acceptable range, suggesting that the washing and recycling procedures were still effective. It is important to mention that at T8, the NaOH concentration decreased to 2.47%, representing 17% of the initial concentration, which could be due to the impurities retained throughout the washing and recycling processes. Between T8, T9, and T10, sharp changes occurred: the value for T9 decreased to 0.84%, and T10 decreased to 0.31%. The NaOH concentration further decreased, indicating a substantial loss of NaOH, which could affect the chemical processes required to modify the properties of the natural fibers.

This noticeable reduction in NaOH concentration between T9 and T10 was likely due to the cumulative effects of washing and recycling. This reduction may result in insufficient removal of impurities such as hemicellulose, cellulose, and pectin, which are crucial for the proper adhesion of the fiber to the matrix. The significant decrease in the concentration between T8 and T9, by approximately 1.63% (55% of the total alkali concentration), may represent a critical threshold at which the effectiveness of the treatment diminishes, potentially compromising the overall outcome.

5.4.2. Influence of the reuse of NaOH solution on the abaca fiber properties

5.4.2.1. Thermogravimetry/Derivative Thermogravimetry Analysis (TGA/DTG)

Thermogravimetric analysis was performed to investigate the effect of different cycles of washing and reusing NaOH solution on the composition of the fiber in terms of thermal stability. Figure 5.5. (a) shows the curves of all ten samples subjected to different washing and reuse cycles, including the natural fiber without treatment, described as natural abaca fiber (NAF) in Fig 5. b. The DTG curve of the NAF fiber shows an initial peak at 60℃ (mass loss around 4%), which corresponds to the vaporization of absorbed water (Rueda-Ordóñez & Tannous, 2018). After this peak, there are two more peaks at 280℃ and 400℃; according to Shahril et al., (2022), between 200℃ and 300℃, the expulsion of hemicellulose lignin and an insignificant amount of cellulose from the fiber occurs. Between 300℃ and 410℃, eliminations of cellulosic components led to mass loss and implied the omission of lignin and wax (Kathirselvam et al., 2019).

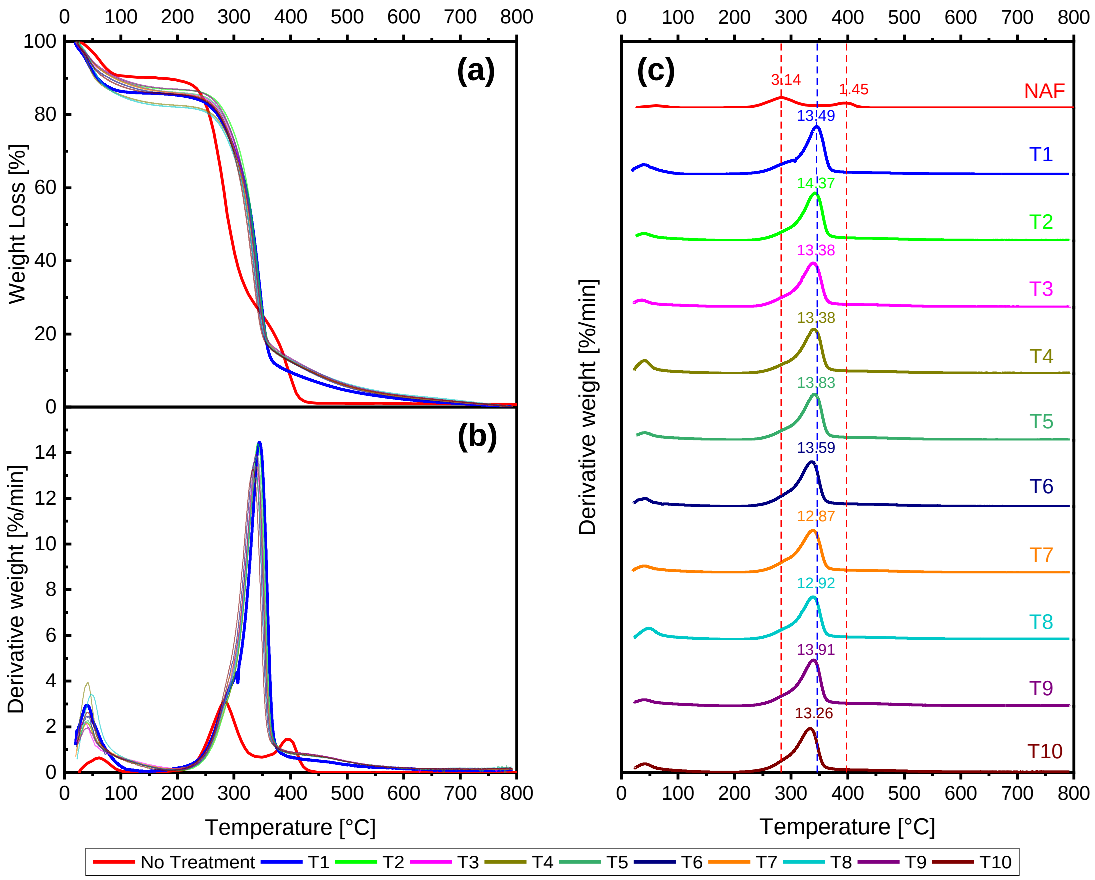

Figure 5.5. Thermogravimetric analysis (a) and derivative thermogravimetric analysis (b–c) of natural abaca fiber (NAF) and treated abaca fiber (T) in 10 cycles of NaOH solution reuse (1–10).

Figure 5.5.c shows the DTG curves of samples T1–T10, where all samples show similarities in their behavior and two main peaks are visible between 40°C and 340°C.

Sample T1 experiences a mass loss of 3% at 38°C owing to vaporization of absorbed water and approximately 60% mass loss at 335°C owing to elimination of hemicellulose and cellulosic components, with lignin being eliminated from the samples (Beroual et al., 2021). During the process of using the recycle solution, the same water solution of 3% NaOH was used to determine the process efficiency by the non-cellulosic material residue content in the cellulosic fibers after the process. The main difference among samples T2, T3, T4, T5, T6, T7, and T8 occurs within 300–400°C, which is the interval corresponding to the fraction of hemicellulose and cellulose, where an evident peak is formed for all treated samples. This corresponds to a loss in mass of a 60%. Samples T9 and T10 experience similar behavior, with a mass loss of 5% at 42°C, and a mass loss of 65% at 340°C, corresponding to the elimination of hemicellulose and cellulose. Comparing the treated and untreated samples, it is evident that hemicellulose and cellulose were lost in all samples with different mass losses. Lignin was still present at 400°C in the untreated samples, while in the treated samples, it was lost. For all samples, the washing and reusing solution of NaOH eliminated the amorphous cellulose, hemicellulose, and lignin proportion in the fiber, and the heat flow required by the fiber sample to decompose was comparatively higher in the alkali-treated fibers than in the untreated samples (S. Fu et al., 2017). In addition, there was no significant variation among the treated samples with respect to mass loss and temperature of occurrence, suggesting that the same alkali solution could be used for at least 10 preparations.

5.4.2.2. Scanning Electron Microscope (SEM)

In Figure 5.6, SEM analysis of samples at 100 μm shows the behavior of untreated and treated samples using the washing and reusing NaOH solution. Visual techniques were used to perform the analyses. The visualization of the specimens was performed by the measurement of the reflected electrons from the surface of examined samples. As observed in the figure, NAF samples present impurities and undesirable globular protrusions at irregular intervals, while treated samples from T1–T8 do not. It is known that alkali treatment improves the surface roughness by removing the hydroxyl coat (OH-), no cellulosic materials, and wax from the surface of the fiber; by removing the hydroxyl coat, the fiber becomes more hydrophobic and reduces the water absorption (Cai et al., 2015).

Treated samples T1, T2, T3, T4, T5, T6, and T7 do not show impurities or globular protrusions; hence, the alkali treatment with washing and reusing NaOH appears effective in removing undesirable residues from the surface (Valášek et al., 2021). In addition, fibrillation and breakdown of the fiber bundle into elementary fibers are visible in these samples, which may occur because pectin, lignin, hemicellulose, and cellulose are removed from the abaca fibers during alkali treatment (Cai et al., 2016).

Up to sample T8, the surface roughness of the natural fiber increased with the disintegration of the hemicellulose and lignin structure, which increased the contact between the real surface area and the environment; hence, it improved the adhesive interface between the fibers and the matrix (Sreenivasan et al., 2014). T8 shows a few impurities on its surface, but they can be considered negligible because the fibers are not bundled and are still separated into elementary fibers. However, T8 represents a turning point where the process of using and recycling the solution could be considered effective.

In contrast, sample T9 exhibited elementary fibers and the presence of impurities. It could be said that at this point, washing and reusing the NaOH solution was no longer effective. Up to T8, the samples showed the same pattern; however, a transition regarding the efficacy of the procedure began at T9. Samples T9 and T10 contained impurities, dust, and wax that were not cleaned during the process. Again, the fibers were bonded to the fiber bundle, decreasing the interfacial adhesion (Punyamurthy et al., 2012).

Through SEM, it can be seen that samples NAF, T9, and T10 show similarities; even if alkali treatment was applied to all samples, including T9 and T10, the recycled water treatment was only effective up to sample T8, where minor impurities were observed. These results show that for T9 and T10, the natural fibers did not react with the treatment, showing impurities, and they seem to bond. Hence, the effectiveness of the alkali treatment through washing and recycling NaOH solution was lost at T9. Thus, from T1 to T8, the behavior was similar, and the alkali treatment of the washing and recycling technique was effective up to this point.

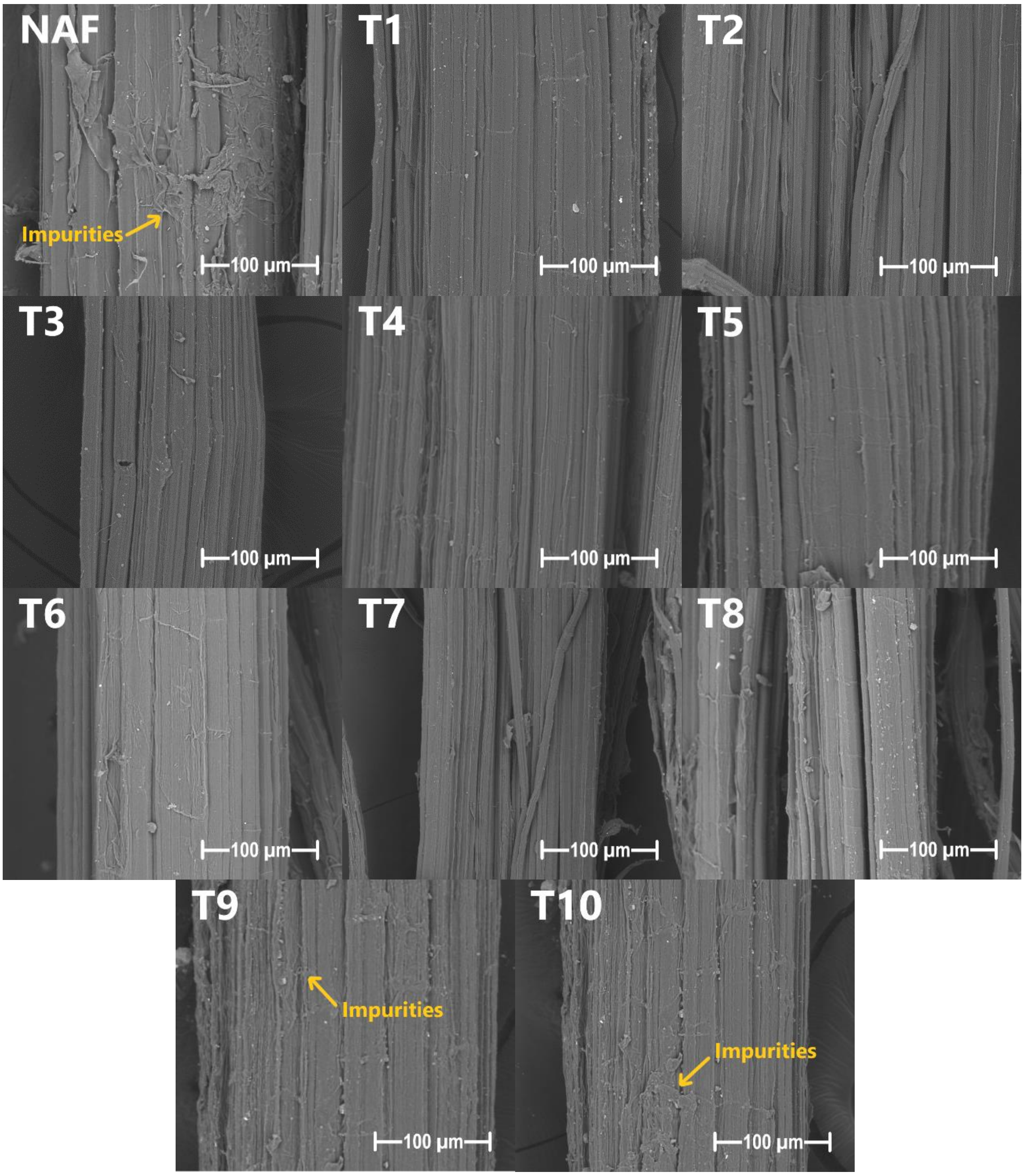

Figure 5.6. Scanning electron microscope (SEM) results of natural abaca fiber (NAF) and treated abaca fiber (T) in 10 cycles of NaOH solution reuse (1–10).

5.4.2.3. Tensile strength

The samples subjected to tensile stress are shown in Figure 5.7. It is seen that NAF reached a 342.92±30.23 MPa tensile strength, which is similar to the result obtained by Valášek et al., (2021), with 326±38 MPa; even if both abaca fibers are from different countries, Ecuador and Philippines, respectively, the results are alike. After applying the alkali treatment, a consistent increase in the tensile strength was observed from T1 to T10 compared to that of the untreated natural abaca fiber (NAF). This suggests that alkali treatment, which involves using and recycling water, initially enhances the strength of the fibers. To evaluate the statistical significance of these improvements, an ANOVA analysis was conducted on the tensile strength results across the samples.

Alkali treatment of natural fibers is known to improve the mechanical properties of the fibers themselves and their composites (Alcivar-Bastidas, Petroche, Cornejo, et al., 2024; Cai et al., 2016; Cao et al., 2006). However, the effect of alkali treatment on abaca fibers can vary depending on the concentration of the NaOH solution, exposure of the samples to this solution, and the time and temperature of the drying cycle.

For instance, Cai et al., (2015), use concentration percentages of 5%, 10%, and 15%, but with different dipping and drying time, results vary; in the first scenario with 30 min dipping time and 2 h drying at 80℃, tensile strength increased by 12%, 11%, and 11%, respectively. In the second scenario, with 2 h dipping time and 2 h drying at 80℃, tensile strength just increased by 8% with a 5% concentration, and with 10% and 15% concentration, it decreased by 5% and 6.5%, respectively (Cai et al., 2016). Y. Liu et al., (2019), used a 3% concentration solution, dipping time of 90 min, and 40 min drying at 75℃, and observed a tensile strength increase of 5.35%. In this research, using 3% concentration, a dipping time of 4 h, and 24 h drying at 85℃, in the conventional alkaline treatment (T1), tensile strength increased 38%, from 342.92 MPa to 473.32 MPa ($P < 0.001$).

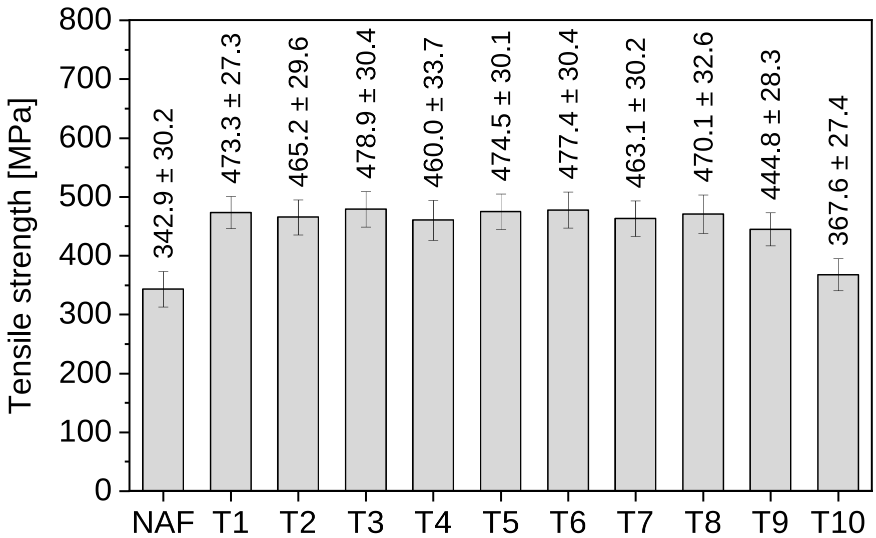

Figure 5.7. Tensile strength results of natural abaca fiber (NAF) and treated abaca fiber (T) in 10 cycles of NaOH solution reuse (1–10). Values are mean ± standard deviation (n = 30).

In this study, washing and recycling NaOH solution on the samples may have affected the tensile strength. T2 increased by 35% compared to NAF ($P < 0.001$).

Samples T1–T8 showed an increase of over 34% compared to NAF. The ANOVA analysis yielded a *P*-value (*P*) greater than 0.05 ($P = 0.156$), indicating that the tensile strength results for these samples are statistically comparable, meaning that any observed differences in strength are not significant enough to be considered meaningful. Essentially, the alkali treatment was consistently effective across these samples (T1–T8), enhancing the fiber strength in a statistically similar manner. Although there were some variations compared to T1 in the tensile strengths of T3 ($P = 0.457$), T5 ($P = 0.878$), T6 ($P = 0.587$), and T8 ($P = 0.683$), these differences were within a range that did not affect the overall statistical comparability; therefore, alkaline treatment remained effective until T8.

However, a critical turning point was identified between T8 and T9. In sample T9, the tensile strength increased as well, but this increase was 29% compared to NAF, which differs from the previous samples ($P < 0.001$), and the ANOVA analysis between these two samples showed a P-value of less than 0.05, indicating that the results for T9 are not statistically comparable to those for T8. This suggests that the treatment process, particularly the recycling of the alkali solution, began to lose its effectiveness after the eighth cycle. The tensile strength of T9, although still higher than that of the untreated NAF, showed a significant decrease with respect to the earlier trend of improvement. This marked reduction in effectiveness is further reinforced with sample T10, which still shows a low increase in tensile strength of 7%, with values of 367.56 MPa compared to 342.92 MPa for NAF. The ANOVA results ($P < 0.001$) confirmed that T8, T9, and T10 are statistically different. This suggests that although the treatment still contributes to some enhancement of the fiber strength, this enhancement is no longer consistent.

Moreover, although the tensile strength of T9 represents a 29.72% increase compared to that of NAF and T10 shows a 7% increase, the ANOVA analysis indicates that these increases are not statistically comparable to those of the earlier samples (T1–T8). This research identified T8 as the critical turning point for the effectiveness of alkali treatment in reinforcing abaca fiber tensile strength. Beyond this point, the process of using and recycling the alkali solution no longer yielded statistically significant improvements. Despite some tensile strength improvement, the diminishing returns observed at T9 and T10 suggest that the treatment process should be limited to eight cycles for optimal effectiveness.

5.4.3. Life Cycle Assessment

5.4.3.1. Life Cycle Inventory

Table 5.5 shows the results of the Ecoinvent LCI calculation tool for crop production applied to the abaca production. This tool assesses biogenic interactions such as energy in biomass, carbon dioxide uptake by biomass, and land use. It also includes activities such as establishing orchards, planting, and trellis system. In outputs, the tool calculates inventories of emissions to soil, water, and air.

In this study, where the scope was limited to calculating the GWP100, emissions to the air were critical. The only air emission calculated by the tool, due to the nature of the cultivation (without fertilizers and pesticides), was N_2O, and was replaced by the results calculated using the IPCC for N_2O from crop residues and leaching/runoff at 2.64E-05 and 3.19E-06 kg N_2O per kg of harvested abaca plant tuxy, respectively.

Table 5.5. Input and output flows of abaca production (tuxy). Data Source: Ecoinvent LCI calculation tool for crop production (Ecoinvent, 2018) using ESPAC data input (Instituto Nacional de Estadística y Censos INEC, 2023a).

Flow	Process/Category	Amount	Unit
Input			
Carbon dioxide, in air	Resource/in air	0.34	kg
Energy, gross calorific value, in biomass	Resource/biotic	3.96	MJ
Establishing orchard	Market for establishing orchard | APOS, U - GLO	2.89E-03	Item(s)
Fruit tree seedling, for planting	Market for fruit tree seedling, for planting | APOS, U - GLO	2.89E-03	Item(s)
Land use change, perennial crop	Market for land use change, perennial crop | APOS, U - EC	3.06E-06	ha

Occupation, permanent crop, non-irrigated	Resource/land	0.61	m2*a
Planting tree	Market for planting tree | APOS, U - GLO	2.89E-03	Item(s)
Transformation, from permanent crop, non-irrigated	Resource/land	3.06E-02	m2
Transformation, to permanent crop, non-irrigated	Resource/land	3.06E-02	m2
Trellis system, wooden poles, soft wood, tar impregnated	Market for trellis system, wooden poles, soft wood, tar impregnated | APOS, U - GLO	6.11E-05	ha
Output			
Abaca plant tuxy, harvested	This Study	1	kg
Cadmium	Emission to soil/agricultural	-2.86E-08	kg
Cadmium, ion	Emission to water/ground water	2.74E-10	kg
Cadmium, ion	Emission to water/surface water	6.50E-09	kg
Chromium	Emission to soil/agricultural	-8.40E-07	kg
Chromium, ion	Emission to water/ground water	1.22E-07	kg
Chromium, ion	Emission to water/surface water	6.00E-07	kg
Copper	Emission to soil/agricultural	-7.47E-06	kg
Copper ion	Emission to water/ground water	1.44E-07	kg
Copper, ion	Emission to water/surface water	6.06E-06	kg
Dinitrogen monoxide	Emission to air/low population density	2.96E-05	kg
Lead	Emission to soil/agricultural	-2.34E-07	kg
Lead	Emission to water/ground water	7.16E-10	kg
Lead	Emission to water/surface water	1.15E-07	kg
Mercury	Emission to soil/agricultural	-1.48E-08	kg
Mercury	Emission to water/ground water	2.82E-11	kg
Mercury	Emission to water/surface water	6.47E-09	kg
Nickel	Emission to soil/agricultural	-9.02E-07	kg
Nickel, ion	Emission to water/surface water	6.79E-07	kg
Nitrate	Emission to water/ground water	1.17E-02	kg
Phosphate	Emission to water/ground water	1.12E-05	kg
Phosphate	Emission to water/surface water	4.68E-05	kg
Phosphorus	Emission to water/surface water	4.85E-04	kg
Zinc	Emission to soil/agricultural	-1.04E-05	kg
Zinc, ion	Emission to water/ground water	3.96E-07	kg
Zinc, ion	Emission to water/surface water	3.25E-06	kg

GLO: Global EC: Ecuador

The total N_2O is 2.96E-05 kg N_2O per kg of harvested abaca plant tuxy, which also represents 0.48 kg N_2O ha^{-1}, lower than other studies reported for banana crops of 1.35–1.6 kg N_2O ha^{-1} for unfertilized soils (Xu et al., 2022; Yang et al., 2003; T. Zhu et al., 2015).

Carbon dioxide, in air, captured during abaca plants growth is 0.34 kg per kg of harvested abaca plant tuxy, similar to that obtained by adapting the studies of Cortez et al., (2015) and Armecin & Gabon, (2008), which reported 0.34 kg of organic carbon per kg of harvested abaca plant tuxy. According to the results of ESPAC 2022 (Instituto Nacional de Estadística y Censos INEC, 2023c), the fiber production of abaca per hectare in this study is 1.3 tons, similar to yield values for Ecuador of 1.2–1.3 tons per hectare from FAO (FAO, 2022), higher than the range of 0.5 to 1 ton reported in other studies (Cortez et al., 2015; Dam & Bos, 2004).

In fiber production, tuxy is stripped using the mechanism shown in Figure 5.3.b, which involves diesel operation at a consumption rate of 0.02 liters of diesel per kg of dry fiber according to the farmers' survey. This value differs from Göltenboth & Mühlbauer, (2010), who reported higher consumption values ranging from 0.05 to 0.08 liters of diesel per kg of dry fiber. In terms of energy, the stripping mechanism uses 0.6936 MJ/kg of dry fiber (Table 5.6), which is close to the reported range by González-García et al., (2010), of 1.2–1.49 MJ/kg of dry fiber for the scutching of hemp/flax fiber using electricity.

Table 5.6. Input and output flows of abaca fiber production.

Flow	**Process/Category**	**Amount**	**Unit**	**Data Source**
Input				
Abaca plant tuxy, harvested	This Study	12.17	kg	(Cortez et al., 2015)
Diesel, burned in agricultural machinery	Market for diesel, burned in agricultural machinery | APOS, U - GLO	0.6936	MJ	This study
Lubricating oil	Market for lubricating oil | APOS, U - RoW	0.002	kg	This study
Output				
Fiber, abaca	This Study	1	kg	This study
Water	Emission to air/low population density	6.51E-03	m^3	(Cortez et al., 2015)

GLO: Global RoW: Rest-of-World

Table 5.7 shows a comparison between all inventories: one cycle (T1) of conventional alkali treatment of abaca fibers (CT) and the option of treatment with the reuse of NaOH solution (RT) in eight washing cycles (T1–T8). The RT proposal generated a 70% reduction in NaOH and deionized water consumption, as well as the same reduction in NaOH transportation. Wastewater generation was reduced by 14%. There was a slight increase of 0.88% in wash water consumption. In both cases, the reduction in the mass of abaca fibers after treatment remained at an average of 20.5%, similar to that observed in other studies (Cai et al., 2015, 2016).

Table 5.7. Input and output flows of abaca alkali treated fiber production.

Process/Category	**Unit**	**CT**	**RT**	**Data Source**
Input				
Market for building, hall | APOS, U - GLO	m^2	1.56E-05	1.56E-05	

Electricity, at supply, 2018 mix - EC	kWh	1.55	1.55	(Faist Emmenegger, Bajaj, et al., 2018; Ramirez et al., 2019, 2020; Wernet et al., 2016)
Fiber, abaca	kg	1.26	1.26	This study
Chlor-alkali electrolysis, membrane cell | sodium hydroxide, without water, in 50% solution state | APOS, U - PE [a]	kg	1.05	0.325	This study
Market for tap water | APOS, U - CO	kg	150.73	152.07	This study
Market for water, deionised | APOS, U - RoW [b]	kg	33.8	10.50	This study
Market for transport, freight, sea, bulk carrier for dry goods | APOS, U - GLO [c]	tkm	1.34	0.414	This study
Output				
Fiber, abaca treated	kg	1	1	This study
Direct disposal of wastewater from textile production | APOS, U - GLO	kg	0.165	0.142	This study
Water, emission to air/low population density	m^3	1.06E-02	1.06E-02	This study

[a] Sodium hydroxide and sodium chloride production are adapted with Peru's electricity.
[b] 50% sodium hydroxide solution state water was considered and removed from the flow.
[c] Sodium hydroxide transport. CT: Conventional alkali treatment (one cycle);
RT: Reuse of NaOH solution per eight cycles of recirculation EC: Ecuador
PE: Peru CO: Colombia GLO: Global RoW: Rest-of-World

The inventories for polypropylene fiber (PPF) production are listed in Table 5.8. Electricity was the only input added to the inventory of Polypropylene and granulates to produce PPF.

Table 5.8. Input and output flows of polypropylene fiber production. (Van den Heede et al., 2018; Yin et al., 2016)

Flow	Process/Category	Amount	Unit
Input			
Electricity, medium voltage	Market group for electricity, medium voltage | APOS, U - US	1.517	kWh
Polypropylene, granulate	Market for polypropylene, granulate | APOS, U - GLO	1.05	kg
Output			
Polypropylene fiber	This Study	1	kg
Waste plastic, mixture	Market group for waste plastic, mixture | APOS, U - RER	0.05	kg

GLO: Global US: United States
RER: Europe

It requires 1.515 kWh per kg of PPF, which is similar to other values reported in LCA and environmental product declarations (EPDs) of micro and macro fibers, reporting energy consumption values between 1.51 to 4.73 kWh per kg of PPF (Athena Sustainable Materials Institute, 2022; Mapei AS, 2017; Master Builders Solutions, 2023; Thrace NG, 2022).

5.4.3.2. Life Cycle Impact Assessment

Different processes of natural, treated, and synthetic fiber production were classified and compared. Figure 5.8 shows the main process contribution results in terms of the carbon footprint.

The carbon footprint of natural abaca fiber (NAF) production was 0.47 kg CO_2 eq./kg fiber (Figure 5.8. (a)), similar to the global warming potential (GWP) results of 0.4 to 1.9 kg CO_2 eq./kg of fiber analyzing flax, hemp, and jute from other studies (Duflou et al., 2012). The most significant contributing process was abaca production with 79% of GWP (Figure 5.8. (b)).

Conventional alkaline treatment increased the carbon footprint of NAF, and the GWP of treated abaca fiber – conventional treatment (TAF-CT) production was 1.48 kg CO_2 eq./kg fiber, 3 times higher than that of NAF, with the highest contributing process being sodium hydroxide production at 35%, followed by abaca production at 32%. Conventional alkaline treatment (sodium hydroxide, electricity, and other processes from the alkali treatment) represented 60% of the GWP of TAF-CT. Abaca fiber production in TAF-CT increased by 26% compared to the abaca plant harvest in NAF, and in terms of CO_2, it increased by 0.09 kg CO_2 eq./kg fiber owing to the mass reduction in the natural fiber during the alkaline treatment.

The process of reusing the sodium hydroxide solution in the treated abaca fiber – reuse treatment (TAF-RT) is 1.11 kg CO_2 eq./kg of fiber, a reduction in the carbon footprint of 0.37 kg CO_2 eq./kg fiber, or a 24.86% reduction compared with TAF-CT. The process with the greatest reduction in the GWP was NaOH production (25 %). This is mainly due to the reduction in NaOH consumption from 1.05 to 0.32 kg of NaOH/kg of fiber. The consumption of the other flows was reduced by reusing the NaOH solution; however, their influence on the final GWP did not exceed 0.52%. Polypropylene fiber (PPF) production has a carbon footprint of 3.30 kg CO_2 eq./kg fiber. The process with the highest contribution to GWP was the production of granulated polypropylene (76 %), followed by the use of electricity in synthetic fiber production (22 %). The treatment of polypropylene waste from this process accounts for 2% of the total.

When comparing the carbon footprint of PPF with that of the other fibers, PPF had the highest environmental impact. In contrast to abaca fibers, the GWP of NAF is 86% lower than that of PPF. TAF-CT has a 55% lower carbon footprint than PPF, indicating that although the treatment increases the environmental impact compared to NAF, it remains considerably lower than that of PPF. On the other hand, TAF-RT presented a 66% lower carbon footprint than PPF, highlighting the advantage of reusing NaOH solution in GWP over both PPF and TAF-CT; nevertheless, the TAF-RT GWP was still higher than that of NAF.

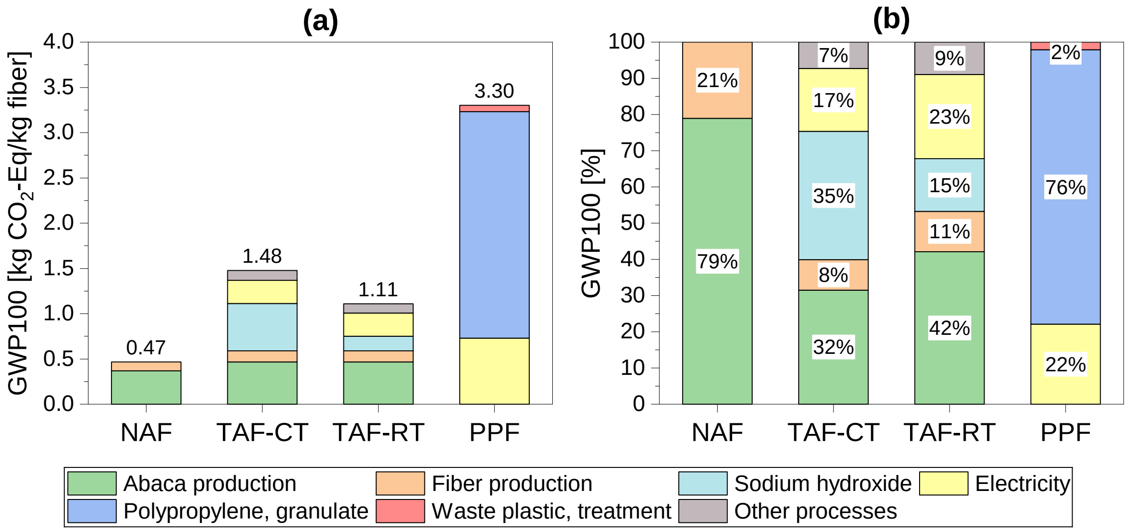

Figure 5.8. (a) Characterization results and (b) contribution analysis in Global Warming Potential (GWP) for natural abaca fiber (NAF) production, treated abaca fiber – conventional treatment (TAF-CT) production, treated abaca fiber – reuse treatment (TAF-RT) production, and polypropylene fiber (PPF) production.

However, to improve the GWP analysis, other properties, such as strengths, should be considered. When comparing the results of the carbon footprint and tensile strength (Figure 5.9), NAF has a lower tensile strength than the traditional natural fibers reviewed in other studies (flax, jute, and kenaf) (Faist Emmenegger, Bajaj, et al., 2018; Peças et al., 2018; Wernet et al., 2016); however, natural abaca fibers still have a lower carbon footprint.

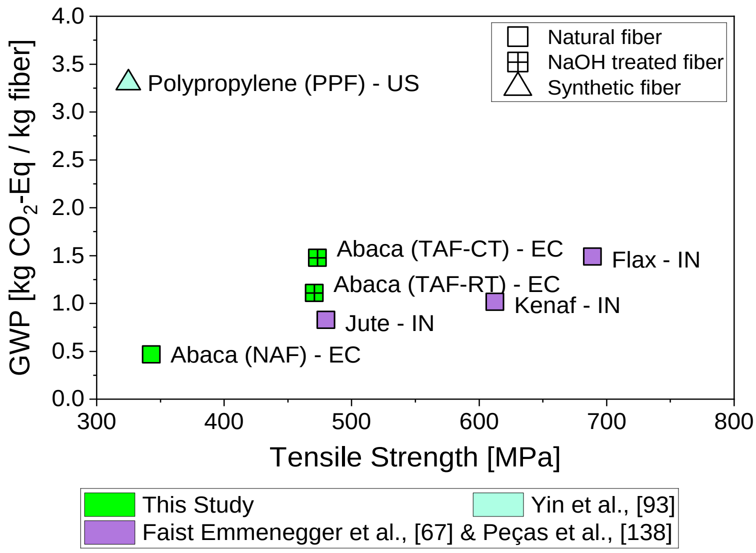

Figure 5.9. Global warming potential results of fibers in literature (Faist Emmenegger, Bajaj, et al., 2018; Peças et al., 2018; Yin et al., 2016) and this study. NAF: natural abaca fiber, TAF-CT: treated abaca fiber – conventional treatment (1 cycle), TAF-RT: treated abaca fiber – reuse treatment (reuse of NaOH solution per 8 cycles of recirculation), PPF: polypropylene fiber, EC: Ecuador, US: United States, IN: India.

With the application of alkali treatment, the tensile strength performance of abaca fibers improved while maintaining a carbon footprint similar to that of other natural fibers. In terms of GWP/tensile strength between abaca and PPF, TAF-RT has one of the highest tensile strengths with one of the lowest carbon footprints, making TAF-RT a sustainable option to be evaluated as a replacement for PPF in concrete where abaca is available. Natural fibers are competitive alternatives in terms of mechanical performance and environmental sustainability compared to synthetic alternatives such as PPF for use in concrete. PPF had the lowest tensile strength and the highest carbon footprint.

5.4.4. Limitations and recommendations for further research

The LCA results for agricultural products such as natural fibers are influenced by the use of fertilizers and pesticides (González-García et al., 2010). Although the global use of these products in abaca is negligible (Dam & Bos, 2004), 45.07% of the crops in Ecuador use chemical and organic fertilizers and pesticides. Therefore, it is recommended that the LCA calculations of these products be enhanced in future studies.

The production of abaca fibers generates substantial residual biomass (Dam & Bos, 2004). A limitation of LCA studies on agricultural products is the decomposition conditions of biomass. This study was based on the aerobic decomposition of biomass as a common condition; however, the possibility of anaerobic conditions should be analyzed in future studies.

The Ecoinvent LCI calculation tool for crop production was used to generate an inventory of the tuxy harvest of the abaca plant. One limitation of using this model is the selection of information from existing crops. In this study, banana was used as the base crop for abaca. The inventory of this study could be improved by investigating the properties of abaca cultivation in terms of carbon capture and content. However, the LCA results were not affected by capture because the methodology used in this study did not consider captured carbon in the GWP impact calculation.

The process of using and recycling NaOH solution has been applied to abaca fibers only with the aim of determining the effectiveness of the solution in treating the fiber. It is recommended that this methodology be applied to other natural fibers, such as jute, kenaf, and hemp.

This study compared the physical and environmental properties of abaca fibers, focusing on TGA, SEM, tensile strength, and LCA. It is recommended to conduct a study on the influence of these fibers and measure their cost and environmental impact when applied to cement composites, such as mortar and concrete.

5.5. Conclusions

This study investigated the impact of the recirculation and reuse of NaOH solution on the alkaline treatment of natural abaca fibers, including its effect on fiber properties and carbon footprint. The following conclusions are drawn from the experimental findings:

- The TGA analysis revealed consistent internal composition across samples T1 to T10, with similar temperatures for vaporization of absorbed water (40°C) and an average mass loss of 4%. Notably, hemicellulose, cellulose, and lignin were lost at 340°C, resulting in equivalent behavior among samples treated with recirculated NaOH solution.
- SEM observations showed uniform structures without impurities in samples T1–T7, forming elementary fibers by removing pectin, lignin, hemicellulose, and

cellulose. However, at T8, minor protrusions appeared insignificant, as the fibers remained elementary. By T9 and T10, the impurities became more visible and elementary fibers bonded, indicating the diminished effectiveness of the alkali treatment.

- Despite these variations, all the samples exhibited increased tensile strength. Up to T8, the increase was greater than 34%, demonstrating the efficacy of 3% NaOH recirculation and reuse. Beyond T8, the tensile strength improvements were slightly reduced, with increases of 29% and 7%, respectively, compared to the NAF samples. Sample T8 suffered a reduction of 2% compared to T1, which is still acceptable and in the range of comparison. However, the T9 experiments showed a decrease of 6% compared to T1, while T10 suffered the highest decrease of 21% compared to T1. At this point, the variation between all samples indicated that there was a turning point between T8 and T9. Relying on conservativity, this research concludes that T8 is the turning point, and up to this cycle of using and recycling the alkali treatment could be considered effective.
- In terms of titration, the results showed that up to T8, the NaOH concentration was relatively stable, maintaining its effectiveness in the recycling process. However, between T8 and T9, the critical turning point occurs, significantly dropping the concentration, thereby diminishing treatment efficacy. This result is related to those obtained in SEM and tensile tests, where impurities start to appear at T8 and the tensile strength starts decreasing.
- Recirculation and reuse of the 3% NaOH solution were effective up to Treatment 8, with consistent TGA results, SEM showing elementary fibers and no impurities up to T8, and nearly uniform tensile strength enhancement up to this point.
- By utilizing the Ecoinvent LCI calculation tool for crop production, key insights were gained regarding the inventory of abaca cultivation and fiber treatment. Emissions to the air, water, soil, and land associated with abaca cultivation were quantified, providing data for understanding the environmental impacts of abaca production. Additionally, a comparison of the treatment options highlighted the potential benefits of reusing the NaOH solution, presenting a substantial reduction in resource consumption and wastewater generation.
- Carbon footprint results show the environmental advantages of natural fibers, particularly abaca, over synthetic alternatives, with notable reductions in carbon footprint and higher tensile strength, especially with the proposed recirculation and reuse treatment (TAF-RT). The implementation of circular economy principles through the reuse of NaOH solution demonstrates not only environmental benefits but also resource efficiency. The reductions in NaOH consumption, wastewater generation, and overall environmental impact emphasize the potential for sustainable practices in industrial processes.

Chapter 6.
Conclusions and future works

6.1. Future research

Once the objectives of this doctoral thesis have been achieved, a series of future research directions are identified with the aim of further improving the addition of natural fiber to cement matrices. These directions provide a foundation for advancing the knowledge and application of fiber-reinforced materials, particularly in the context of sustainable construction practices.

First, the present research focused exclusively on plaster mortar, with the addition of fiber as the principal component for enhancing the material's properties. While this approach proved effective for plaster mortar applications, there is significant potential for expanding this study to other cement-based materials, such as concrete. By incorporating natural fibers into concrete, researchers can explore the material's behavior in more demanding applications, such as structural elements like slabs, where controlling shrinkage and crack formation is critical. Concrete, being a fundamental material in construction, presents unique challenges and opportunities for integrating natural fibers. Future studies could investigate the mechanical properties, durability, and performance of fiber-reinforced concrete under various loading conditions and environmental factors. For example, the inclusion of natural fibers in concrete slabs could contribute to mitigating shrinkage-induced cracking, which is a common issue in concrete structures. This expansion of research could lead to innovative solutions for both residential and commercial construction projects, improving their sustainability and resilience.

Second, the practical application of the developed fiber-reinforced mortar was explored in this study through the construction of a wall, which was subsequently plastered with the mortar and subjected to seismic load testing. While the test setup demonstrated the potential of the material for such applications, the results were inconclusive due to the over-designed nature of the wall. The absence of fissures or cracks during seismic load testing highlighted the need for a more appropriately designed test specimen that better reflects realistic conditions. Future research could revisit this experiment by constructing walls with optimized designs to allow for meaningful evaluation of the material's performance under seismic loads. Additionally, different configurations of fiber content, wall thickness, and reinforcement patterns could be tested to identify the optimal design parameters for earthquake-resistant applications. This line of research is particularly relevant in regions prone to seismic activity, where innovative materials and construction methods are needed to enhance the safety and resilience of buildings.

Beyond these initial steps, there is considerable scope for exploring other aspects of fiber-reinforced cementitious materials. For instance, studies could assess the long-term durability of the materials in various environmental conditions, such as high humidity, freeze-thaw cycles, or exposure to chemical agents. Another area of interest could be the life cycle assessment (LCA) of fiber-reinforced materials to quantify their environmental benefits compared to traditional alternatives. By examining the carbon footprint, energy consumption, and waste reduction associated with these materials, researchers can build a stronger case for their adoption in sustainable construction. Furthermore, interdisciplinary approaches that integrate structural engineering, materials science, and environmental studies could pave the way for groundbreaking advancements in the field.

In conclusion, the achievements of this doctoral thesis lay the groundwork for a multitude of future research opportunities. Expanding the focus to include other cement-based materials, optimizing seismic load testing, and exploring additional aspects of performance and sustainability will contribute to the ongoing evolution of fiber-reinforced cementitious materials. These efforts will not only improve the material's practical applications but also support the global push toward greener and more resilient construction practices.

6.2. Conclusions

According to the objectives set out on this thesis to analyze the influence of the addition of abaca fiber on the characteristics of cement agglomerates and their long term behavior and based on the research conducted whose methodology and most relevant results are documented in the articles: The effect of different treatments on abaca fibers used in cementitious composites, published in the journal *Natural Fibers*; Effect of aging process on mechanical performance of reinforced mortar with NaOH abaca fibers published in the journal *Case Studies in Construction Materials*; Characterization and life cycle assessment of alkali treated abaca fibers: the effect of reusing sodium hydroxide published in the journal *Construction and Building Materials*, the following conclusions are drawn:

- This study evaluated the effect of three different treatments on abaca fiber: hornification (HR), NaOH-3% (HS) and coating with silica fume and natural latex (LS). The results revealed that NaOH-treated fibers exhibited the best performance in flexural and compressive strength, especially when using a 0.2% dosage and a fiber length of 30mm. All treatments effectively modified the fiber by removing lignin and hemicellulose, as confirmed by TGA, XRD, FT-IR and SEM analysis. While HS treatment showed the greatest improvement in mechanical properties, LS treatment performed the poorest, likely due to minimal internal changes. HR treatment showed high initial flexural strength but eventually had lower performance than HS.
- In the second part of the research, the mechanical properties of alkali-treated abaca fiber reinforced mortar (MFTHS) and reference mortar (RM) were analyzed, after both were subjected to an accelerated wet/dry aging process (WD-1 and WD-2). Despite the aging process, the alkali treated fiber maintained its chemical composition, as shown by FT-IR and SEM analysis. The results indicated that WD-, with higher temperature and longer dipping time, was more aggressive, causing greater property deterioration compared to WD-1. However, MFTHS consistently showed better mechanical properties compared to RM, with significant improvements in flexural, compressive, and tensile strength: 20%, 28% and 26% higher, respectively. The addition of alkali treated fiber also improved shrinkage control, leading to more compact samples with better water retention.
- The third part of the research, explored the effects of recirculating and reusing a 3% NaOH solution during the alkaline treatment of natural abaca fibers, focusing on fiber properties and environmental impact. Up to Treatment 8 (T8), consistent thermogravimetric analysis (TGA) results and scanning electron microscopy (SEM) showed uniform fiber structure and significant removal of pectin, lignin, and hemicellulose, enhancing tensile strength by over 34%. However, beyond T8,

impurities emerged, tensile strength improvements diminished, and NaOH concentration dropped, marking T8 as the optimal reuse cycle. Environmental analysis using the Ecoinvent tool revealed substantial reductions in resource consumption, wastewater generation, and carbon footprint through this circular approach, showcasing the environmental and resource efficiency advantages of reusing NaOH in abaca fiber treatment. This sustainable method highlights the potential for integrating circular economy principles into industrial processes.

- Therefore, the study demonstrated that incorporating abaca fiber into cement agglomerates, combined with an alkaline treatment using recirculated NaOH solutions, significantly enhances the materials performance and sustainability. Up to the eight cycles of NaOH reuse, the alkali treatment effectively improved the tensile strength of the fibers. Additionally, for long terms, the process of W/D showed the effectiveness of treated abaca fibers incorporated to the matrix, where better mechanical behavior was achieved. Hence, it can be concluded that the addition of treated abaca fibers (alkali treatment) did improve the mechanical properties of the mortar.

Chapter 7.
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