RESEARCH ARTICLE



Impact of accelerated aging cycles on the performance of extruded cement-based composites reinforced with non-bleached eucalyptus fibers

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Abstract

Thermo-mechanical pulping produces well-individualized fibers compared to wood particles and less fragile fibers compared to Kraft pulping, besides presenting higher volume, higher yield, and lower production cost, which can be an exciting alternative for the fiber-cement industries. This study evaluated the impact of soak and dry-aging cycles on the performance of extruded composites reinforced with non-bleached eucalyptus fibers. The cement matrix comprised cement (70%) and limestone (30%). Composites were reinforced with 1 to 5% of eucalyptus fiber by cement mass and tested on the 28th day of cure at 99% relative humidity and after 400 accelerated aging cycles. The water absorption and apparent porosity gradually increased with the reinforcement level. Composites with 4 and 5% fibers showed the highest toughness (0.21 and 0.23 kJ/ m², respectively). The aging by 400 soak-dry cycles reduced the composites' water absorption and apparent porosity. The modulus of elasticity (MOE), rupture (MOR), and toughness increased, except for toughness for composites reinforced with 1 and 5% fibers, explained by the cementitious matrix's continuous hydration, fiber mineralization, and natural carbonation. In general, eucalyptus thermo-mechanical fibers were suitable for producing cementitious composites. Cementitious composites with 3% fibers presented a higher MOR, MOE, low water absorption, and apparent porosity after 400 accelerated aging cycles. In addition, the composites with 4% fibers also presented remarkable improvements in these properties. The aging cycles did not result in composites with less resistance, a positive fact for their application as tiles and materials for external use in civil construction.

Keywords Thermo-mechanical process · Vegetal fibers · Cement composites · Extrusion process

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Introduction

The construction industry has been looking toward the sustainability of the available products and services. The prohibition of asbestos fibers (standard reinforcements of cement composite) in several countries because of the health dangers associated with its inhalation has stimulated countless kinds of research in the last decades on substitute lignocellulosic sources. Potential feedstock fibers are wood (Zhao et al. 2013), sisal (Melo Filho et al. 2013), hemp (Sedan et al. 2008), coconut (Andiç-Çakir et al. 2014), jute (Fonseca et al. 2021), the cellulosic pulp (Tonoli et al. 2013), unbleached Kraft pulp waste (Silva et al. 2015), Eucalyptus fibers (Silva et al. 2019), and bamboo (Ardanuy et al. 2011). The advantages of using lignocellulosic fibers are the high specific resistance and modulus, natural and renewable traits, low cost, and low environmental impact for growth and extraction (Tonoli et al. 2011).

Eucalyptus fibers from wood thermo-mechanical pulping are produced on a large scale and high production yield, with no harmful chemicals to the environment and lower waste generation (Magniont et al. 2012). These fibers are already used to produce fiber panels, indicating their potential to develop new non-structural materials. The thermo-mechanical pulp, in comparison with chemical pulp, has significant advantages, such as high yield (>90%), larger volume, good printability, and lower affinity with water (Tian et al. 2014). Another critical aspect of these pulps is their integrity. Most fibers break in the middle lamella, containing many polysaccharides, lignin, and pectin (Liu et al. 2016). Therefore, in thermo-mechanical pulping, hydrophobic wood substances keep on the fiber surface (Liao et al. 2021), which can provide a barrier to the alkalis of the cementitious matrix, increasing the durability of these fibers in the composite. These fibers are already used to produce fiberboards, paper, and packaging, among other uses, indicating their potential for developing new eco-friendly non-structural materials.

The extrusion involves forming fiber-cement composites by forcing the mixture of cement and fibers through the die, adjustable to form materials of various configurations (Teixeira et al. 2012). This continuous process requires a lower investment cost than other well-established methods, such as the Hatschek; hence, it may provide materials with great potential for low-cost commercial applications. The extrusion is an advantageous and versatile economical mass-production method capable of producing flat, structural, and hollow shapes. This process allows the use of many waste materials, including lignocellulosic fibers, by successfully incorporating them into the matrix to reinforce fiber-cement composites (Almeida et al. 2013).

However, the possible low compatibility between plant fibers and the cement matrix raises several drawbacks. The vegetal fibers absorb large quantities of water because of their hydrophilic nature (Silva et al. 2019). Consequently, the flowing ability of the cement mixture decreases. These dimensional variations will likely create cracks within the fiber cement at both the macroscopic and microscopic scales, decreasing the composite durability (Al-Mohamadawi et al. 2016). In addition, the dissolution of extractives of the fiber surface in the cement mixture disturbs the cement hydration and causes some delay in the composite cure (Sellami et al. 2013).

The choice of thermo-mechanical eucalyptus fibers is based on their low production costs and lower environmental aggression for obtainment, as it avoids the bleaching stage, which is the most polluting part of the pulping process. Additionally, there is a lack of information on their suitability to produce extruded fiber-cement composites. The accelerated aging test simulates natural aging by exposing cement materials to continuous soak and dry cycles, or conditions varying into a series of heating-freezing and humidity-freezing cycles (Gehlot and Shrivastava 2024). Therefore, it provides information about the future changes promoted by the mineralogical phase composition on the transition zone between the matrix and cellulose fibers and the resulting microstructure of the fiber-cement composites. Understanding fiber degradation and inhibition is essential to establish new strategies for cement composite production by predicting the evolution of their microstructure, properties, and overall performance when they age in service. Both the performance and the useful life of the constructions have become an increasing concern to civil construction companies and consumers (Martins et al. 2018).

This study aimed to evaluate the impact of soak and dryaging cycles on the performance of extruded composites reinforced with eucalyptus fibers derived from thermomechanical pulping, a non-bleached process. By exposing the composites to aging cycles and assessing their impacts, better uses and applications can be established safely and without the risk of severe damage to the final structure, such as roofs and facades, wall panels, tubes and pipes, industrial coatings, and insulating boards.

Material and methods

Materials

The wood fibers were extracted from *Eucalyptus* spp. (around 7-year-old) by a thermo-mechanical pulping process and purchased from an MDF industrial unit of Eucatex S.A (Itu — SP, Brazil). A paraffin emulsion ($\sim 0.8\%$ by mass) was applied in the industrial process of wood chip defibration to decrease the wear of the equipment and improve the dimensional stability of the fibers. A matrix with a mix

design of 1:0.5:0.4 (binder:filler:water/binder) by weight was prepared. The binder used was Brazilian ordinary Portland cement CPV ARI (calcium, aluminum and iron silicates, calcium sulfate, and carbonate filler), with high initial resistance, provided by CSN Cimentos (Arcos — MG, Brazil), according to the standard NBR16697 (ABNT 2018), correspondent to the type III C150M standard (ASTM 2021). The filler was limestone (CaCO₃: 98.0 to 100.0%; MgCO₃: Max 0.50%; Bulk density: 0.37 to 0.47 g cm⁻³) provided by Quimisul (Joinville — SC, Brazil). Hydroxypropylmethylcellulose (HPMC) was supplied by Aditex Ltda. (Brazil), and it features a viscosity between 60,000 and 70,000 cP and is a self-compacting additive for concrete polyether carboxylic (commercially named ADVA 175) furnished by Grace-BR Ltda. It had a density of 1.10 g cm⁻³ and a pH of ~ 3.40.

Characterization of the fibers

The basic density of the fibers was determined as described in the NBR 11941 (ABNT 2003) standard with some adjustments in the obtainment of volume (water displacement of fibers). The NBR 14853 (ABNT 2010a) was used to determine total extractives; the NBR 7989 (ABNT 2010b) was used to determine the amount of lignin, and the NBR 13999 (ABNT 2017) was used to determine the amount of ash, while holocellulose was determined by weight difference. An Olympus BX51 (Japan) optical light microscope was used to measure the length and average diameter of the fiber bundles. The light microscopy revealed individualized and mostly long fibers, besides some short fragments (Fig. 1). The average dimensions of the anatomical structures are typical of individualized fibers.

Hydration test

Vegetal-based materials can attenuate or even inhibit the temperature rise during the hydration process of Portland 35791

cement. A preliminary hydration test was conducted using the same method described in Okino et al. (2004) to assess the compatibility of eucalyptus fibers derived from the thermomechanical process with the cement matrix. Distilled water (90.5 ml) was added to a mixture of cement (200 g) and wood (15 g oven-dry basis) in a polyethylene bag for 3 min. The cement–wood–water mixture was placed in a wide-mouth insulated flask with a thermocouple wire and then covered with styrofoam. The time to attain maximum temperature was considered to be the required setting time of the mixture. The Portland cement used was CPV ARI, the same used to produce the fibercement composites.

Extrusion of the composites

The cementitious matrix consisted of cement CPV ARI according to the standard NBR16697 (ABNT 2018) and ground agricultural limestone. The main chemical and physical characteristics of this cement and ground limestone are presented in Almeida et al. 2013. The mixture for extrusion was prepared by adding 1% (by mass) of rheology modifiers: Hydroxypropylmethylcellulose (HPMC) and polyether carboxylic (commercially named ADVA 175). The final water/ cement ratio was around 0.40. The dry powders (cement, filler, and HPMC) were mixed for 10 min and the thermomechanical fibers were gradually added to homogenize the mixture. The ADVA was diluted in water in a ratio of around 0.1 and the solution was added to the dry mixture under stirring for 5 min. The resulting compound was inserted into the extruder to obtain the composites with $20 \times 30 \times 200$ mm (Fig. 2). The composites were produced on a laboratory scale by extrusion in a VERDES 051 model helical extruder. The mix-design formulations of the composites are described in Table 1. Fourteen replicates were produced for each treatment and the curing day of the composites was recorded.



Fig. 1 Typical light microscopy image of the eucalyptus fibers

Fig. 2 Production process of fiber-cement composites; numbers 1 to 5 demonstrate step by step, from preparing the materials to curing the composites and accelerated aging cycles



 Table 1
 Experimental design of the extruded fiber-cement composites

Treatments	Cement	Limestone	Fibers	
	(% by mass)			
Control	70.0	30.0	0	
FC_1%	69.3	29.7	1	
FC_2%	68.6	29.4	2	
FC_3%	67.9	29.1	3	
FC_4%	67.2	28.8	4	
FC_5%	66.5	28.5	5	

Accelerated aging cycles

The accelerated aging test aims to simulate natural aging with exposure to soak and dry cycles (Almeida et al. 2013). Fiber-cement specimens were successively immersed in for 170 min in water at 20 ± 5 °C and, after 10 min, they were heated for 170 min to 60 ± 5 °C in a ventilated oven. Ten minutes of interval at room temperature was applied before the subsequent extrusion. Cycles of 400 soak and dry were executed to check the impact of weathering on the physical and mechanical performance of seven specimens by treatment.

Characterization of the composites and data analysis

Apparent porosity (AP), bulk density (BD), and water absorption (WA) of the fiber-cement composites were determined following the procedures described in the C948-81 (ASTM 2016) standard. The mechanical test was performed using the universal testing machine (Time-Shijin, WDW-20E model) equipped with a 20 kN load cell. Four-point bending was performed (lower spam equal to 150 mm). The properties limit of proportionality (LOP), modulus of elasticity (MOE), rupture (MOR), and toughness (TO) were evaluated and are described in detail in ABNT (2021). Data analysis was performed using a randomized design.

Scanning electron microscopy (SEM)

On the 28th day of cure and after 400 accelerated aging cycles, micrographs of the fracture regions were performed after a static bending strength test. The microstructural properties of the cement matrix and the fiber/matrix interface were evaluated by scanning electron microscopy (SEM) with a secondary electronic image detector (SEI), operated with an acceleration voltage of 20.0 kV. The fractured surfaces were metallized with a gold bath before being analyzed using a microscope Zeiss LEO Evo 40.

Results and discussion

Hydration test

The pure CPV ARI cement achieved hydration temperatures of 80 °C, whereas the cement mixed with fibers reached a maximum of 36 °C (Fig. 3). The composites with 4% and 5% fibers only reached the initial setting after 14 and 22 days of curing, respectively, in a saturated environment (relative humidity > 95%). The decrease in maximum temperature can



 Table 2
 Chemical components

 of the non-bleached eucalyptus
 fibers

Components	Values (%)	
Ash	0.5 ^(±0.1) *	
Total extractives	10.9 ^(±0.3)	
Total lignin	$23.0^{(\pm 0.1)}$	
Holocellulose	65.6 ^(±0.3)	

^{*}Standard deviation

result from a lower cement solidification value or a certain mass of lignocellulosic material that does not contribute to the heat generation. The amount and types of extractives of the fibers (Table 2) in contact with the water of the cement matrix are potential inhibitors of the hydration and composites curing (Sellami et al. 2013).

The poor compatibility of plant fibers and cement is mainly due to the effect of dissolved extractives of plant fibers on cement hydration, besides the presence of a large number of hydroxyl groups in the cell walls with high water absorption, resulting in a negative effect on the physical and mechanical properties of the composites (Pereira et al. 2019). Some plant fibers contain pectin, known for its calcium ion chelating capacity, and fix calcium ions of the cementitious matrix on their surface (Le Troedec et al. 2008), an effect known as "egg carton" (Grant et al. 1973). As calcium is essential to form calcium silicate hydrate (C-S-H), the main hydration product of cement, its absence delays the hardening of the composites (Sawsen et al. 2015). The inhibition index found for the mixture of fibers and matrix was 34.52%, which is considered moderate inhibition according to the classification proposed by Okino et al. (2004). The authors above stated that inhibition index values between 10 and 50 indicate moderate inhibition. Pereira et al. (2019) found an inhibition index of 1.93% for cement with coconut fibers (extractives content 3.38%), causing a low influence on the inhibition of the cement in the solidification process. In general, there is an inverse relationship between the extractive content of the lignocellulosic material and the hydration temperature of the cement + fiber (Lopes et al. 2005). In addition, during the middle and late periods of the hydration reaction, saccharides, lignin, and other substances contained in extractives will form an adsorbed layer on the surfaces of cement particles, which has a particular binding effect on cement. This affects the hydration process, reduces the hydration rate, and causes the temperature peak of the paste with lignocellulosic material to be lower than that of the pure cement paste (Jiang et al. 2015).

Therefore, the eucalyptus fibers probably delayed the stiffening of the composites reinforced because of hydration and inhibition problems of the cementitious matrix. According to the literature, the hydration test was carried out with a fixed fiber content (Okino et al. 2004). Still, a gradual increase in the influence/inhibition of eucalyptus fibers in the cement matrix may occur as the fiber content in the composite increases. The calorimetry test assesses the compatibility between the Portland cement and eucalyptus fibers by evaluating the difference between the maximum temperature reached by the neat cement paste and fibercontaining paste over curing.

Physical properties of the composites

On the 28th day of cure, fiber pull-out could be observed in the fracture region of the composite, indicating a weak fiber/matrix interaction. After accelerated aging, the fibers were ruptured after the composite fracture, which indicates a higher fiber/matrix interaction, and partial degradation of these fibers due to the cementitious matrix alkalinity (Fig. 4).

After 28 days of curing, it is noted that the properties of water absorption and apparent porosity tend to reduce until the reinforcement percentage of 3% (Fig. 5). For reinforcement percentages of 4 and 5%, there was a significant increase in the values found for the properties mentioned. The bulk density of the composites tended to decrease, mainly for the 4 and 5% of fiber reinforcements. The decrease in density occurred because the fibers' density was significantly lower than the cement matrix (Silva et al.





Fig. 5 A Water absorption, **B** apparent porosity, and **C** bulk density of eucalyptus fiber-reinforced composites on the 28th day of cure and after 400 accelerated aging cycles

2015). The average basic density of eucalyptus fibers was 0.186 g cm^3 , providing lower density to the composites with higher fiber amounts. Additionally, greater porosity of the composite contributes to lower density values, and greater cell wall availability results in a greater capacity for water absorption.

After 400 soak-dry aging cycles, the composite water absorption and apparent porosity reduced significantly until the reinforcement level of 3%. With reinforcement levels of 4 and 5%, the water absorption and apparent porosity values were superior to the others. The bulk density of the fiber-cement composites tended to increase, mainly for the contents of 4 and 5% of fiber reinforcement, after 400 cycles. Aging cycles cause mineralization of the fiber and result in friable composites due to the filling of voids by cement hydrates. This process also favored the reduction of porosity and water absorption, increasing the bulk density of the fibers used as reinforcement favoring the densification of the fiber-cement composites. Composite porosity is a crucial aspect to be considered during the manufacturing process and in the final performance of the material. It can significantly impact the composite's physical, mechanical, and thermal properties. Silva et al. (2015) report that the presence of pores can reduce the mechanical strength of the material and compromise its fatigue resistance and durability. Additionally, pores can serve as stress concentration points, which can lead to premature failure under load. Therefore, minimizing porosity during the manufacturing process may involve optimizing processing parameters such as temperature, pressure, and material feed rate and using appropriate curing techniques.

Extractives also influenced the physical properties, explaining notable differences between the 4% and 5% reinforced composites. More fibers in the cement matrix increase the contact area between the fiber and the matrix, accentuating the inhibition effect and delaying the initial hydration of the cement. This effect contributes to an increase in the number of permeable pores and, consequently, the water absorption in the fiber cement. The composites reinforced with 4 and 5% of fibers were the most affected by the 400 cycles, probably due to their highly porous structure. An increased amount of voids favors the carbonation and hydration of the available free lime through continuous exposition to water and natural carbonation of the cement matrix (Roma et al. 2008). Previous studies also reported the same patterns for composites with increasing fiber levels (Wei and Meyer 2015). Overall, composites with fiber content lower than 3% presented physical properties similar to control composites, as also observed for composites from previous works using cellulose Kraft pulp as reinforcement (Silva et al. 2015).

Mechanical properties of the composites

Figure 6 shows the typical static stress vs. deflection curves of the control sample and fiber-reinforced composites on the 28th day of cure and 400 soak and dry-aging cycles.

Fiber-cement boards containing 1% fiber did not show reinforcing characteristics. In this case, the stress/deflection curve obtained a similar behavior to the cement board without fibers as reinforcement at the time of rupture. Moreover, the 5%-reinforced composites had the lowest mechanical strength. After accelerated aging, despite the evidenced gain in mechanical properties (see Fig. 6B) attributed to the improved cement hydration, the fibers lost their reinforcing characteristics due to their degradation and mineralization. Comparative studies of aging processes, under real conditions, and of short-term accelerated aging processes, under simulated conditions, are very useful for the assessment of durability (Bhering et al. 2023). Overall, the mechanical properties increased with fiber content after the soak and dry cycles. Filomeno et al. (2023) presented an increase in strength of 4 MPa in fiber cement making use of accelerated carbonation at 1 day for curing, after 200 cycles of accelerated aging. This tendency indicates that the cementitious matrix continues hydrating with contact with water during soaking. Then, hydration products are formed around the fibers, improving their connection with the matrix and mechanical performance. However, toughness decreased in composites containing 1 and 5% of fibers, probably because

Fig. 6 A Typical stress-deflection curves of the fiberreinforced composites on the 28th day of cure. **B** Typical stress-deflection curves of the fiber-reinforced composites after 400 accelerated aging cycles. **C** Deflection as a function of fiber content in the composite samples on the 28th day. **D** Deflection as a function of fiber content in the composite samples at 400 cycles



of the fiber-free spaces in the 1%-reinforced composites and fiber agglomerates in the 5%-reinforced composites with inadequate fiber dispersion. After 400 aging cycles, the deposition of re-precipitated hydration products (e.g., calcium hydroxide, ettringite, monocarbonate) in the transition zone between the fibers' surfaces and matrix, which to some extent may decrease the energy absorption during the fiber pull-out (Melo Filho et al. 2013), reduced deflection (Fig. 6D). Furthermore, migrating cement hydration products with aging to the fiber lumen and cavities may cause fiber mineralization (Roma et al. 2008). This re-precipitation turns the fiber stiffer, as observed for 5%-reinforced composites (Fig. 7). The increase in the fiber content led to lower values of the limit of proportionality, MOR, and MOE cured after 28 days (Fig. 8). In addition, the composites with fiber content above 3% presented higher toughness compared to the non-reinforced.

Fibers with high lignin content and other non-cellulosic compounds are more sensitive to alkaline environments and may influence the durability of the composite (Tolêdo Filho et al. 2003). Thermo-mechanical pulping results in a pulp chemical composition similar to wood since lignin is preserved (Silva et al. 2019). As previously reported, the extractives present in the fibers probably caused some curing and hydration delay (see Fig. 3), harming the mechanical





Fig. 8 A MOE, **B** limit of proportionality, **C** MOR, and **D** toughness of eucalyptus fiber-reinforced composites on the 28th day of cure and 400 accelerated aging cycles

properties of the composites. This fact highlights the fundamental need to remove secondary components, that is, extractives from the raw material that inhibit cement hydration reactions in the cement-fiber interaction. On the other hand, Thielemans et al. (2002) studied composites with hemp fibers treated with lignin and found improved tensile and flexural properties.

Excessive amounts of fibers may characteristically balllike agglomerate, causing the "balling effect" (Chakraborty et al. 2013). Moreover, incorporating fiber works as a kind of "bridge" for cement hydration in lower time. The increase in fiber content increases the number of "bridges," reducing cement hydration products, which reduces flexural strength (Xie et al. 2015), as observed in the composites containing 4 and 5% of fibers. Using small amounts of fibers may lead to an uneven distribution of the reinforcements in the matrix. Regions with no fibers are starting points for crack propagation (Khorami and Ganjian 2011), harming the toughness of the composites with 1 and 2% of fibers. From 3% on, the increased fiber level gradually increased the composites' toughness, with an optimum transference of the stress from the matrix to the fibers. Then, the fiber pull-out may occur at the interface, allowing its pullout from the matrix (see Fig. 7A), generating considerable frictional energy, and increasing the toughness (Coutts 2005). Applying 3% of eucalyptus thermo-mechanical fibers generally led to physical-mechanical performance similar to results reported by other studies that used cellulose Kraft pulp as reinforcement (Mohr et al. 2005).

After 400 soak and dry cycles, the 3%-reinforced composites presented a significant gain in mechanical properties, indicating good resistance and durability. At the same time, other works found in the literature showed losses in the mechanical properties after 400 cycles of accelerated aging (Santos et al. 2015). However, from Fig. 7B and C, it is possible to verify that the fibers underwent a process of degradation and mineralization, losing the reinforcement characteristic. Fiber degradation was compensated by continuous hydration of the cementitious matrix, resulting in a gain in mechanical properties of the fiber-cement composites after accelerated aging.

Conclusions

In this study, the impact of soak and dry-aging cycles on the performance of extruded composites reinforced with eucalyptus fibers derived from thermo-mechanical pulping was evaluated. The following conclusions are drawn from this experimental study:

• Eucalyptus fibers derived from the non-bleached process can promote moderate inhibition of the cementitious matrix in cement composites. As the fiber content increases, the hydration of the anhydrous cement is affected, causing an increase in porosity from 23 to 40% and in water absorption from 12 to 25%, considering 28 days.

- The MOR of the composites reinforced with 3 and 4%, and the MOE for the composites reinforced with 2 and 3% increased after 400 cycles of aging. The physical– mechanical properties of fiber-cement reinforced with 3% fiber are similar to fiber-cement reinforced with bleached Kraft pulp.
- Fiber pull-out was observed after 28 days of cure, indicating a weak fiber/matrix interaction. The accelerated aging provided a higher fiber/matrix interaction and partial degradation of the fibers due to the alkalinity of the cementitious matrix. These fibers become interesting for application in fiber-cement due to the fiber-cement physical-mechanical properties and mainly because the production process does not involve chemical steps, ensuring lower waste generation and lower final cost.
- On the other hand, the extractives delay the hydration of cement; thus, depending on the percentage of reinforcement used, the gain in mechanical properties becomes gradual and slower than the fiber cement reinforced with Kraft pulp.
- Evaluating the properties as a whole shows that the optimum level of fibers from the thermomechanical pulping process is between 3 and 3.5% of reinforcement. In future works, it would be worthwhile to evaluate different types of bleaching in this pulp to understand the lignin role in the degradation process of these fibers, as well as the behavior of the composite.

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Declarations

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