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Utilization of vegetal fibers for production of reinforced cementitious materials

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Abstract

Vegetal fibers produced from agroindustrial resources have been used as reinforcement in cementitious materials in the macro, micro and nanometric scales. The cellulosic pulp, besides being used as the reinforcing element, is also the processing fiber that is responsible for the filtration system in the Hatschek method. On the other hand, the nanofibrillated cellulose has the advantage of having good mechanical performance and high specific surface, which contributes to improve the adhesion between fiber and matrix. In hybrid reinforcement, with both micro and nanofibers, the cellulose performs bonding elements with the matrix and acts as stress transfer bridges in the micro and nano-cracking network, with corresponding strengthening and toughening of the cementitious composite. Some strategies are studied to mitigate the degradation of the vegetal fibers used in cost-effective and non-conventional fiber cement, as well as to reach sustainable fiber cement production. As a practical example, accelerated carbonation curing at early age is a developing technology to increase the durability of composite materials: it decreases porosity, promotes a higher density in the interface, generating a good fiber–matrix adhesion and a better mechanical behavior. Thus, vegetal fibers are potentially applicable to produce high mechanical performance and sustainable cementitious materials for use in civil construction.

Keywords: Sustainable fiber cement; Carbonation; Macrofibers; Pulp; Nanofibrillated cellulose

1 Introduction

The construction industry is a major driver of the global economy both in industrialized and in developing regions. As a consequence, this sector is associated with several environmental problems [1,2].

The construction industry, in general, and the Portland cement value chain in particular, are material intensive. From environmental point of view, civil construction consumes more than 50% of the world's raw materials [3]. Cement-based materials waste is an obvious and almost unexplored option. Combined with natural aggregates, it consumes a growing share materials used by modern society, a fraction that surpasses 1/3 of the total materials [4]. The Portland cement industry also releases about 6-8% of the anthropogenic CO_2 [1,5]. In order to comply with the Paris agreement on climate change [6], the cement value chain has to innovate, going beyond the usual clinker substitution, by blast furnace slag or traditional pozzolans, and energy efficiency. This will probably imply in adapt to local constraints, such as availability of raw, preferable waste, materials and different demands from each market.

Silica-fume, metakaolin and fly ash are the most used pozzolanic additions in combination with ordinary Portland cement and carbonate filler [7]. The particle size distribution of these materials is a very important parameter for processing many cement products, as powder packing can be tailored by selecting of raw materials with suitable sizes and fractions. In the design of the bulk cementitious matrix of fiber-cement, various raw materials must be properly combined and adjusted, with different particle size range. Besides, some basic characteristics of the interfacial transition zone (ITZ) between particles in Portland cement paste with mineral additions and between cement paste and short lignocellulose fibers must be studied, evaluated and correlated to mechanical properties of these composites. The pozzolanic additions can react with the free portlandite available in the ITZ to form extra calcium silicate hydrates (CSH) and help with denser and less aggressive interface with the vegetal fibers [8].

Pozzolans can be produced by the use of biomass for energy co-generation, especially if firing conditions are controlled. For example, sugar cane bagasse ash and rice husk ash are reported elsewhere to perform pozzolanic activity.

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Approximately 140 billion metric tons of biomass are produced every year in the world from agriculture. For each ton of processed sugarcane, there are 270 kg of bagasse [5,9-12]. In India, for instance, it is reported that about 600 Mt/year of rice husk have been generated from agricultural sources alone. If fired during industrial processing, for average losses on ignition of 7%, 42 Mt/year of ashes could be produced in one year; approximately 15% of India's cement production [10].

Another option for mitigation strategy is dematerialization. The use of small volume fraction of fibers is known to allow the production of thin sheets that can be used in roofing, partition walls and even as slabs, increasing the efficiency of the resources used and therefore, reducing materials intensity of this industry. In developing countries, like Brazil, Hatschek produced lightweight fiber cement components are one of the most popular solutions for roofing, a market conquered by the corrugated sheets of asbestos-reinforced or synthetic fiber-reinforced products (Fig. 1).

Hatschek was the first method of fiber cement manufacturing, and it was invented by Ludwig Hatschek in the 1890s. This process begins from slurry mixture with cellulose pulp, reinforcing fibers, Portland cement and mineral additions in water. This fed into a papermaking like machine in which a cylindrical sieve or sieves rotate through the slurry. The solids are deposited on the sieve, which on each rotation transfers the layer of solids onto a continuous belt. The layers are built up to the desired thickness and then removed and, if necessary, compressed by stack pressing [13].

Nowadays, almost all asbestos fiber is replaced by plastic counterparts such as polypropylene (PP) and polyvinyl-alcohol (PVA), which are alkali resistant [14]. However, the synthetic fibers are non-renewable, expensive and their production consumes a significant amount of energy and petrochemical raw materials [15]. The asbestos free solution is more expensive than the asbestos reinforced composites, especially due to the increase of the cost of raw materials.

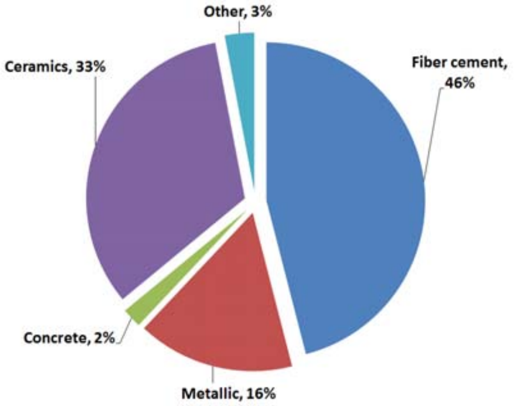

Figure 1. Various types of materials used for popular solutions of roofing in Brazil (Based on [16]).

In many countries, the transition from asbestos reinforced to asbestos free fiber cement (due to health concerns) implied in a reduction of market share of this lightweight fiber cement-based roofing, which had been replaced by roofing technologies frequently heavier and costly, a factor that has social and environmental implications. The use of vegetal fibers to produce low-cost, sustainable fiber cement composites have been discussed since 1988 [17-20] and can be a local option in many regions of the world.

In this paper, we will discuss the potential of bio-based fibers to promote a resource-efficient and low-carbon cement based value chain.

2 The potential of bio-based fibers

In recent years, an increasing attention has been given to the use of cellulose fibers as alternatives to replace plastic fibers in air-cured composites [7,15,16,19]. However, air cured fiber cement reinforced exclusively with cellulose fibers presents some reduction in the mechanical performance. It is due to low durability of the vegetal fibers in a highly alkaline environment, such as the cementitious matrix. Currently, plastic fibers are the main reinforcement of fiber cement composites, because these fibers are less sensitive to alkali attack and possess good mechanical properties, resulting in composites with better performance in the long-term. Cellulose fibers are used only as process fibers in the Hatschek production of air cured plastic fiber reinforced components [7].

The commercial use of vegetal fiber is still limited. These natural fibers are a functionally graded material that has a hierarchical structure in the macro, micro and nanometric scales with interest as reinforcement in cementitious materials [21,22]. Motivations for their use include the decreased raw materials costs and a potential contribution to reducing environmental impacts of fiber cement sheets, because they are renewable, may require lower amounts of energy to produce and, being cheap, may help lightweight fiber cement to remain in the market. Additionally, vegetal fibers can be locally produced, generating local social and economic benefits. Vegetal fiber from different origins has been studied as a reinforcement of cement-based matrices, such as banana [23], sisal [24-26], hemp [27], green coconut [28,29], sugarcane bagasse [30], curauá [31,32], bamboo pulp [22,33,34], pine and eucalyptus pulps [15,35-37].

Nevertheless, the reduction of vegetal fiber durability is caused mainly by the alkaline (pH ~12-13) environment of the cement matrix and gradually filling of the inner cores of the vegetal fibers with the cement hydration products, leading to the embrittlement of the fibers, and reducing their mechanical performance [38-40]. Some strategies to mitigate the degradation of the vegetal fibers used as reinforcement in cost-effective and non-conventional fiber cement, as well as to reach a more sustainable fiber cement production are indicated in Fig. 2, such as functionally graded materials [41,42], reduction of the consumption of Portland cement [1], utilization of different binders [5,37,43,44], mineral additions from agroindustrial wastes [9-12] carbonation curing [45,46] and nanofibrillation of the lignocellulosic fiber [21].

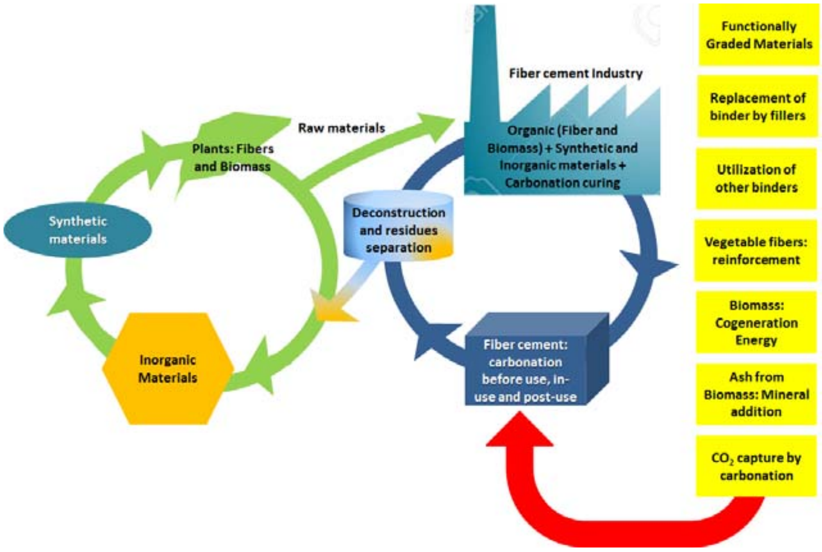

Figure 2. Schematic diagram showing a concept process consisting of different approaches to improve the sustainability of the fiber cement industry.

In order to offer durability and resilience of the fiber cement reinforced with vegetal fiber, contribute to infrastructure sustainability, extended service life and expand the range of functional uses of vegetal fibers in construction applications, it is necessary to adopt systemic research using the concept of integrated structures and materials design as represented in Fig. 3 [32,47]. Thus, material properties serve as a natural interface between structural engineering and materials engineering.

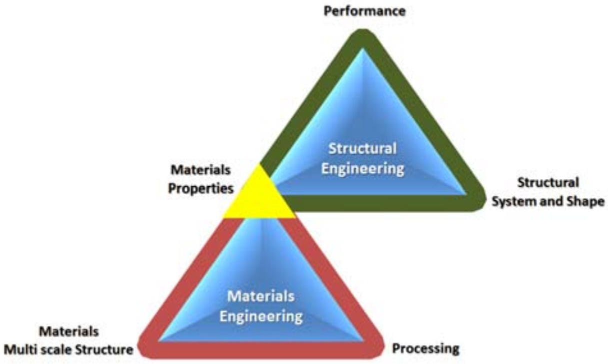

Figure 3. Schematic diagram of Integrated Structures and Materials Design (Based on [47]).

Thus, to design cementitious composites reinforced with vegetal fibers to meet the concept of integrated structures and materials can be considered a complex and systematic task. For example, the project design must consider the interrelationship between interfacial transition zone, fibers, matrix and processing method as depicted in the schematic illustration showed in Fig. 4.

These approaches may result in efficient gains, cost reduction and contribution to sustainable development with regard to environmental aspects. Therefore, vegetal fibers have attracted great interest because of their regional availability and high specific tensile strength (associated to their low density). However, their effective consolidation as raw material in the civil construction industry involves a great technical and scientific effort.

3 Study on the potential of two lignocellulosic material

3.1 Cellulosic pulp

In addition to the macrofibers, the microfibers obtained through chemical and mechanical pulping are also used as reinforcement of cementitious materials. In such composites, the cellulosic pulps are used to promote packaging with the particles of the matrix. In the industrial production of fiber cement (Hatschek process), the cellulosic pulp is used to form a network for retention of the cement particles during the water drainage stage [7,15].

The advantage of the use of pulp as reinforcement in comparison to macrofibers is the greater surface roughness of the fiber, which increases the adhesion capacity between the fiber and the matrix. Another advantage is that the short length of these fibers facilitates their distribution and homogenization in the matrix, improving the fiber-matrix bond and, consequently, the reinforcing efficiency [48-50]. In addition, the pulp can originate from wood (pine and eucalyptus, e.g.) and other non-wood lignocellulosic fibers.

Wood pulps (pine and eucalyptus) were used in the range of 4-10% by mass, as reinforcement of cementitious matrices [35-37,46,51,52]. Non-wood fibers are also commonly used as reinforcement of cementitious composites, such as sisal [39,53-56], banana [23,53-55], bamboo [22,33,34], fique [35], cotton [57] and agricultural waste fibers [30].

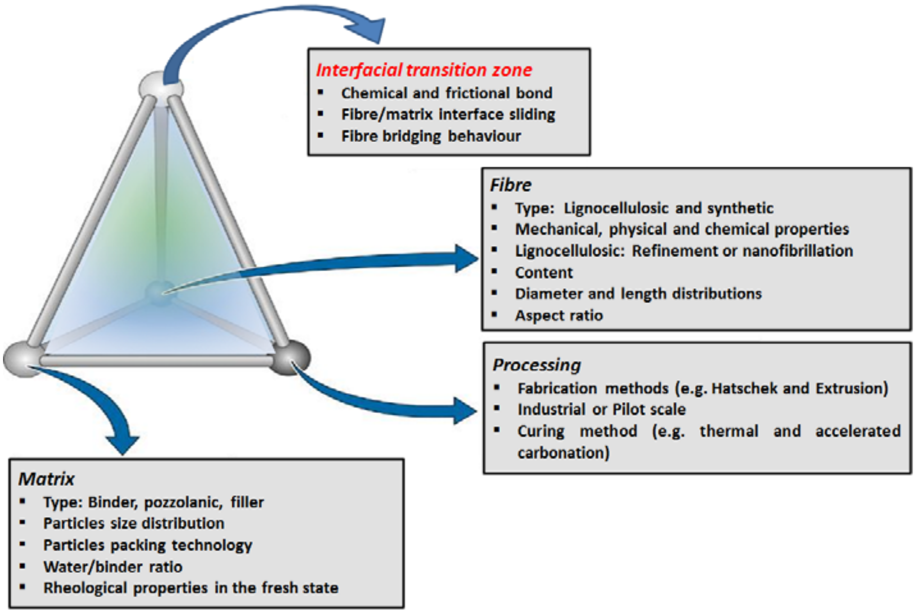

Figure 4. The tetrahedral interrelation of the main constituents of the cement based composite design. Adapted from [8].

Khorami and Ganjian [30] compared the flexural performance of cementitious composites reinforced with contents of 2% and 4% by mass of the sugarcane bagasse, wheat wastes and eucalyptus pulp. In that study, the content of 2% of fibers promotes no significant changes in flexural performance of the composites in comparison to composites without fibers; however, the content of 4% of pulp improved significantly the flexural strength of the composites. Khorami and Ganjian [58] evaluated the effect of different residual Kraft pulp contents (1-14%) on the flexural strength of cementitious composites. The authors concluded that the optimum reinforcement content was 8% by mass, where the modulus of rupture value was 250% higher than composites with 1% fiber.

Correia et al. [22] used bamboo pulp, produced by the organosolv pulping process, in different contents (6%, 8%, 10% and 12% by mass) as reinforcement of cementitious composites, with matrix composed by Ordinary Portland cement type CP V-ARI (75% by mass) correspondent to ASTM-C150 Type I, and metakaolin (25% by mass), and the mechanical results from bending test are presented in Fig. 5. The fiber cement is a component of the civil construction, which is submitted usually to the flexural effort. The content of 8% of bamboo pulp was sufficient to ensure the capacity of reinforcement before and after the crack propagation. The mechanical strength of the composites reinforced with 10% and 12% of pulp was lower due to the greater porosity caused by the higher pulp content. However, the toughness in the post-fissured condition was significantly higher for the higher amounts of pulp.

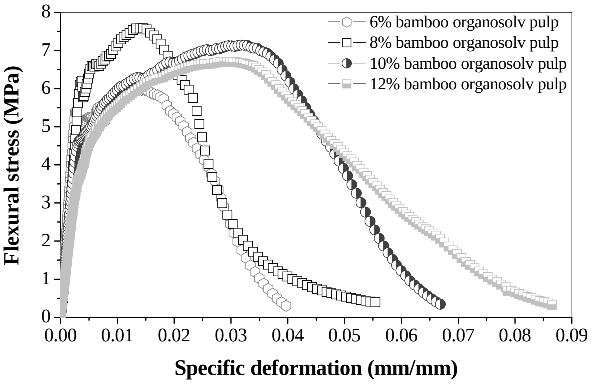

Figure 5. Typical stress - strain curves of the composites containing 6, 8, 10 and 12% bamboo organosolv pulp during flexural tests after 8 days of thermal curing. Adapted from [22].

3.2 Nanofibers as reinforcement of cement-based composites

Nanofibrillated cellulose consists of a bundle of stretched cellulose chain molecules with long, flexible and entangled cellulose nanofibers with dimensions of approximately 1-100 nm and they are composed of alternating crystalline and amorphous regions [59-61]. They are produced from the isolation of cell wall of the lignocellulosic materials using shear forces without chemicals. Fig. 6a and Fig. 6b, provided by Correia et al. [21], presents a visual result of the transformation of the cellulose pulp prior to nanofibrillated cellulose after 5 cycles of nanofibrillation by the grinding process.

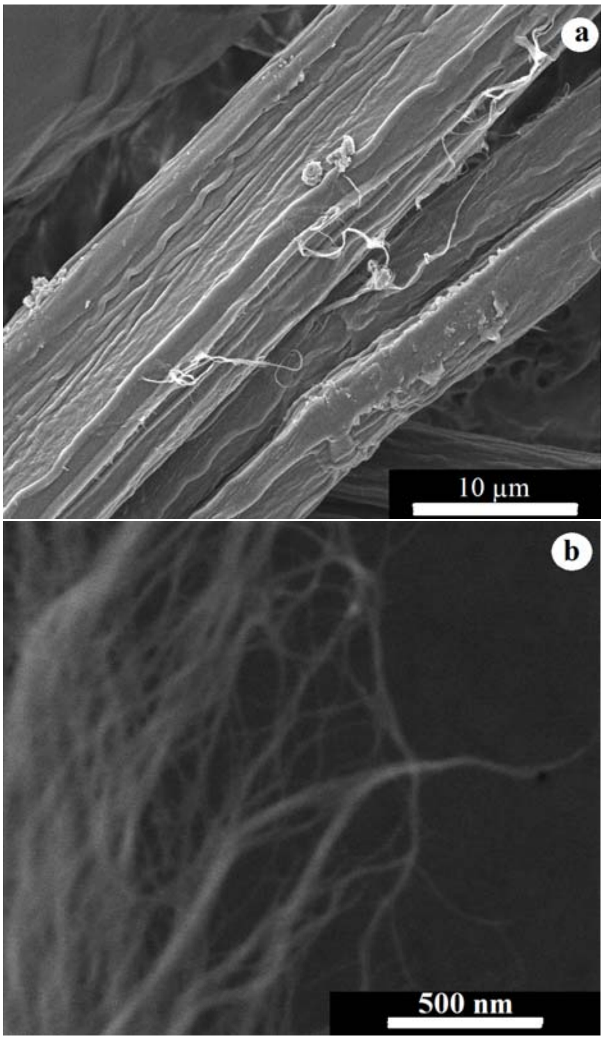

Figure 6. Unbleached bamboo pulp (a) and nanofibrillated cellulose from bamboo pulp after 5 nanofibrillation cycles. Adapted from [21].

Vegetal fibers in nanoscale have also been used as reinforcement of cementitious composites with the objective of reinforcing materials. Nanofibrillated cellulose has the advantage of high modulus of elasticity (82.3 GPa) [10] and high specific surface area (50-70 m^2/g) [62], in comparison to cellulosic pulp, which contributes to improve the adhesion between fiber and matrix. In addition, the nanofibers act as stress transfer bridges in the nano-cracks during loading [63-65]. According to Han et al. [66], the nanofibers can delay the beginning of the crack progression at the nanoscale and therefore, higher loads are required to start cracking the composite, which indicates the increase of the mechanical strength of the cementitious matrix.

Most of the work already published focuses on the use of carbon nanofibers and nanotubes as reinforcement due to the mechanical properties of these nanofibers, such as high modulus of elasticity (1TPa for nanotubes), high tensile strength (50-200 GPa) and 280% increase in the rupture strength [64,67]. When using conventional mixing methods, there is a strong tendency of agglomeration of the nanofibers, which increases the water demand for adequate rheological behavior, which increases the variability of the composites properties. However, if the production process succeeds to adequately disperse fibers and keep the water demand in control, besides the mechanical performance improvement, they can reduce the electrical resistivity and contribute to the increase of the durability of cementitious composites due to the reduction of water permeability [68-70]. However, the use of carbon nanofibers and nanotubes increases the cost of the final composite.

Thus, the nanofibers from vegetal resource are an alternative as reinforcement of cementitious materials at the nanoscale, since these nanofibers have as advantages, abundance worldwide, it is a renewable source, they have low density and high mechanical resistance. The cellulosic nanofibers, in general, have low modulus of elasticity in comparison to the fibers of carbon, glass and steel, and consequently, the cellulosic nanofibers promote a greater toughening of the brittle matrices [21,71,72].

The literature presents works on cementitious composites reinforced with vegetal nanofibers obtained by different methods, such as, acid hydrolysis and mechanical nanofibrillation. Cellulose nanocrystals are produced by acid hydrolysis, and are added to a cement matrix to improve the physical and mechanical properties. The work developed by Cao et al. [73-75] was the first one published in high impact journals on the use of cellulose nanocrystals in cementitious materials [76].

The results presented by Cao et al. [73,75] show that in some cements cellulose nanocrystals contributed to the improvement in the cement hydration and of microstructure of matrix, and consequently, the addition of cellulose nanocrystals increased the flexural strength of composites. The water diffusion rate in the high-density hydrated calcium silicate increased with the addition of cellulose nanocrystals and the flexural strength of the composites increased 20-30% with the addition of 0.2% by volume of nanocrystals [73-75].

Thomson et al. [77] produced hybrid composites by slurry vacuum dewatering followed by pressing process – a system that overcomes the difficulties associated to the demand of high amounts of mixing water for dispersing nanofibers and other nanoparticles – reinforced with 8% of cellulosic pulp and the contents of 0.5, 1.0 and 2.0% of cellulose nanocrystals. The mechanical results showed increased modulus of rupture with up to 0.5% of nanocrystals.

The cellulose nanofibers obtained by the mechanical process (nanofibrillation) appear to have advantages over cellulose nanocrystals.

Nanofibrillated cellulose has higher specific surface area and higher aspect ratio than cellulose nanocrystals, which increases the interaction between fibers and matrix, promoting the higher mechanical resistance of the composites reinforced with nanofibrillated cellulose [21,72,78].

One pioneering work about the use of nanofibrillated cellulose as a reinforcement of cementitious materials was carried out by Ardanuy et al. [78,79]. In these studies, the authors used 3.3% (by mass) of nanofibrillated cellulose from sisal as reinforcement of mortar in comparison to mortar reinforced with the same content of cellulosic pulp. The mortar reinforced with nanofibers had an increase of 26.4% in the tensile strength under flexural load and a corresponding increase of 41.5% in the modulus of elasticity, compared to mortar reinforced uniquely with pulp.

Onuaguluchi et al. [80] produced cement pastes by incorporating of lower contents of nanofibrillated cellulose originated from bleached pine pulp. The authors used the contents of 0.05%, 0.1%, 0.2% and 0.4% of nanofibrillated cellulose. The results showed that the reinforced pastes with 0.1% of nanofibers had an increase of 106% in flexural strength and of 184% in energy toughness, compared with pastes without nanofibers with the same water/cement ratio of 0.5. According to the authors, the improvement of these properties is attributed to the characteristics of high specific surface area of nanofibers, which increases the interface between fiber and matrix, and the high hydrophilicity of the cellulose, which promotes better adhesion with the cement. However, the pastes with contents above 0.1% did not have a good mechanical behavior due to the agglomeration of the nanofibers.

The efficiency of the nanofibers as reinforcement in the cement-based composites has been studied by the Research Nucleus on Materials for Biosystems, at University of São Paulo, Brazil. Cement composites reinforced with nanofibrillated cellulose were produced from bamboo organosolv pulp, and were compared to the composites with 8% of bamboo organosolv pulp and the hybrid composites with 8% total cellulose but different proportions between pulp (7%, 6% and 5%) and nanofibrillated (1%, 2% and 3%). All samples were produced by vacuum dewatering process followed by compression and the matrices were composed by Ordinary Portland cement type CP V-ARI (75% by mass) correspondent to ASTM-C150 Type I, and by the ground limestone filler (25% by mass) [13,53].

The stress-strain curves presented in Fig. 7 demonstrate that composites with nanofibrillated cellulose have higher flexural strength than those reinforced by conventional cellulose pulp. However, the composites reinforced with only nanofibrillated cellulose showed a fragile behavior, with much lower toughness energy than those hybrid composites reinforced, which combined cellulose pulp and nanofibrillated cellulose. Hybrid composites presented much better performance than the reference composites reinforced with pulp only. In hybrid reinforcement, with micro and nanofibers, nanofibrillated cellulose seems to bond strongly with the matrix and acts as stress transfer bridges in the micro and nano-cracking, a mechanism that can explain the measured strengthening of the composite. However, the conventional pulps increase the tenacity due to a well-known pullout process, despite reducing the flexural strength, probably because they act as large defects in the matrix.

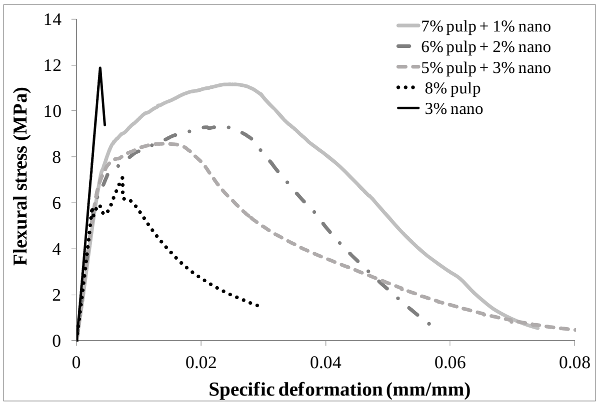

Figure 7. Typical stress-strain curves of the composites with 3% of nanofibrillated cellulose, the hybrid composites with different content of pulp and nanofibrillated cellulose and composites with 8% of pulp at 8 days.

The values of the modulus of rupture of the hybrid composites and of the composite reinforced with just 3% of nanofibrillated cellulose are significantly higher in comparison to the composite without nanofibers (8% of pulp). The modulus of rupture is directly associated with the existence of the defects in the matrix and at the interfacial transition zone (ITZ) between the fiber and the matrix, and with the performance of the fibers used as reinforcement. The ITZ can be defined as the three-dimensional boundary between the fiber and the matrix. In this region, the interactions can occur through various mechanisms. The mechanisms are described as: (i) micromechanical interlocking, (ii) permanent or induced dipole interactions, (iii) chemical bonding, (iv) chain entanglement/fibrillation, and (v) adhesion by embrittlement/mineralization of the fiber [8]. The nanofibrillated cellulose promoted the improvement of the stress transfer throughout the bulk of the composites when they were subjected to loading. Nanofibrillated cellulose also contributed to the increase of the physical and chemical adhesion, friction and mechanical anchorage with the matrix, induced by its high specific surface area. Besides, it has a better mechanical anchorage in the cementitious matrix without the effect of the drying shrinkage of fibers [8].

The existence of branches of the fibrillated nanocellulose increases the anchoring capacity of the nanofibrillated cellulose with pulp and matrix. The ability to form stress transfer bridges between the nanofibrillated cellulose and the matrix can be illustrated in Fig. 8.

Therefore, these results show that the use of nanofibrillated cellulose is effective for increasing the mechanical performance of cementitious materials. However, there is no consensus regarding the optimum nano-reinforcement content used to improve the properties of the cement-based materials.

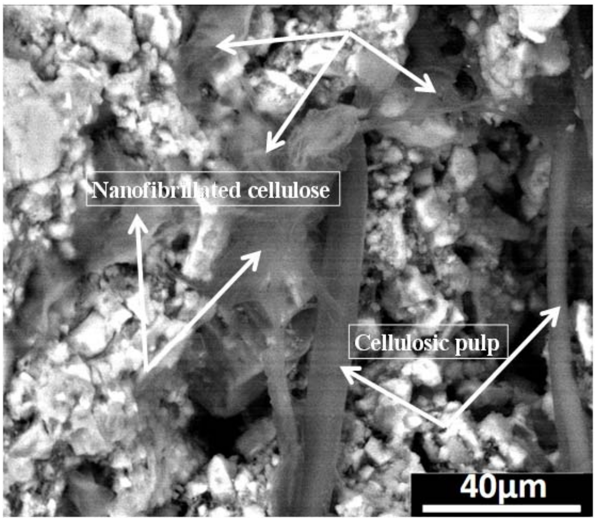

Figure 8. Micrograph of hybrid composite showing the adhesion capacity of nanofibrillated cellulose with the pulp and cement matrix.

4 Accelerated carbonation curing for cement-based composites

Despite the high performance of cellulose as reinforcement of cement matrices, it has been reported in the literature that vegetal fibers have low durability in an alkaline matrix, such as cement [19]. A way to overcome the alkaline degradation of the fibers is neutralization by accelerated carbonation curing, which also reduces total porosity improving mechanical resistance of the matrix [21,36] and densifies the fiber/matrix interface, which may reduce the fracture energy of the composite. Carbonation is also a carbon capture and storage process of cement based materials for which kinetics is accelerated in porous thin-sections such those of fiber cement composites, and therefore helps CO_2 mitigation.

Santos et al. [81] evaluated the effect of accelerated carbonation in supercritical condition on extruded fiber cement reinforced with bleached eucalyptus pulp and residual sisal fibers after 3 days of initial curing and after 200 cycles of accelerated aging (soak and dry cycles). According to results presented in Fig. 9, after accelerated aging the average values of the energy of fracture of the carbonated and non-carbonated composites reduced 28% and 56% respectively. This result shows evidence of the improved conservation of microstructural stability and of the toughness of cementitious composites reinforced with vegetal fibers after curing by accelerated carbonation in supercritical conditions.

The results achieved by Urrea-Ceferino et al. [36] show that accelerated carbonation curing reduces matrix alkalinity and mitigates the cellulose degradation in cementitious composites.

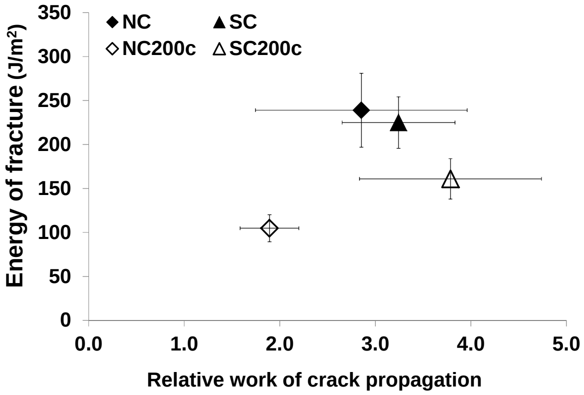

Figure 9. Energy of fracture versus relative work of crack propagation of extruded cellulosic fiber cement non-carbonated (NC) and submitted to supercritical CO_2 (SC), before and after exposure to 200 soaking and drying cycles (200c) (Based on [81]).

Table 1 presents the results of the study developed by Correia et al. [82]. The authors studied the effect of hybrid reinforcement (bamboo nanofibrillated cellulose + bamboo pulp) in the performance of carbonated cement composites produced by the extrusion process in comparison to carbonated composites reinforced with only bamboo pulp, at 28 days and after 200 cycles of immersion and drying. The results indicate that the nanofibers present a higher contribution to delay the onset of crack propagation and its potential to act as stress transfer bridges in the nanocracking, mainly after 200 cycles of immersion and drying. The results of flexural stress (MOR) and energy of fracture shows that after accelerated aging the nanofibrillated cellulose preserved partially its ability to form bonds, once the fibers have remained undamaged [82].

Table 1. Mechanical performance of hybrid composites and composites reinforced with pulp at 28 days and after 200 cycles of immersion and drying [83].

Composites		MOR (MPa)*	Energy of Fracture (J/m^2)*
28 days	8% pulp + 1% NC	19.9a	421.8a
	9% pulp	14.8b	394.7a
Aged (200 cycles)	8% pulp + 1% NC	20.1a	382.2a
	9% pulp	17.8b	379.1a

*Average values followed by the same letters do not differ significantly by the Tukey test ($p < 0.05$).

Thus, the potential of the use of nanofibrillated cellulose as a reinforcing element of cementitious materials to increase the mechanical performance of these materials was assessed. Additionally, the use of accelerated carbonation curing optimize the effect of nanofibrillated cellulose, reducing pore size and porosity, densifying the nanofiber / matrix interface and increasing the durability of the nanofibers in the alkaline environment.

5 Final remarks

Biomass has been a source of materials for long time. For the cement-based industry, biomass can produce, among other raw materials, pozzolanic ashes and fibers. The abundance of the vegetal fibers in all regions of the world, their physical and mechanical properties, make of these materials an interesting option for reinforcement for inorganic matrices.

The nanofibrillated fibers have further advantages over cellulose pulp microfibers, and other vegetal fibers for the use as reinforcement, including their higher specific surface area, which favors the adhesion with the matrix. They have also lower lignin content, which reduces alkaline susceptibility. Cellulose nanocrystals have also a potential in cement, including because they may interfere with cement hydration kinetics. As with all nanoparticles, dispersion and water demand for good rheological behavior are a major challenge. Vacuum dewatering production process, which is used worldwide by the fiber cement industry for more than one century - seems to be the easier way to avoid this complication, but other routes can also be feasible with adequate technology.

Durability of these fibers in alkaline Portland cements is a challenge. Accelerated carbonation cure reduces alkalinity of the matrix and mitigates the cellulose degradation in cementitious composites, meanwhile captures CO_2. Additionally, accelerated carbonation curing favors porosity refining and increases densification of the fiber/matrix interface, thus improving the mechanical performance of cementitious composites reinforced with vegetal fibers.

Finally, it is possible to state that cement based composites reinforced with vegetal fibers are technically possible to be produced and used in civil construction in regions where they are abundant, but their effective consolidation as raw material still involves a great technical and scientific challenge.

Acknowledgments

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