



Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Mineralogical and microstructural changes promoted by accelerated carbonation and ageing cycles of hybrid fiber–cement composites



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HIGHLIGHTS

- Interface between cellulose fibers and cement matrix in the carbonated composites was improved.
- Changes in cement phase composition with carbonation led to decrease of voids around the cellulose fibers.
- Initiative to CO₂ sequestration and partial replacement of petroleum-based fibers in fiber–cement.

ARTICLE INFO

Article history:

Received 21 February 2014

Received in revised form 7 May 2014

Accepted 29 June 2014

Keywords:

Vegetable fibers
Cellulose fiber
Fiber/matrix bond
Cure

ABSTRACT

Carbonation takes place in the fiber–cement composites by the diffusion of carbon dioxide (CO₂) through the unsaturated pores in the cement matrix, and through its reaction with the hydration products of the Portland cement (mainly calcium hydroxide – Ca(OH)₂ and calcium silicate hydrate – C–S–H phases). The use of this technology during the fiber–cement production consists of an interesting procedure to prematurely decrease the alkalinity of the cement matrix, which is potentially harmful to those reinforcing fibers that are vulnerable to the alkali attack. It is also an initiative to CO₂ sequestration and partial replacement of petroleum-based fibers as is the case of cellulose pulps. Therefore, the objective of the present work is to show the effect of accelerated carbonation and ageing on the mineralogical composition and microstructure of fiber–cement composites reinforced with both cellulose pulp and synthetic fibers. The effectiveness of the accelerated carbonation was confirmed by X-ray diffraction (XRD) and thermogravimetric (TG) analysis. Accelerated carbonation increased the content of calcium carbonate (CaCO₃) and consumed the Ca(OH)₂, C–S–H, monosulfate (AFm), ettringite (Aft) and monocarboaluminate (Mc) phases. The SEM micrographs showed that absence of AFm and Aft needles around the cellulose fibers in the carbonated composites, confirmed by the absence of the peaks that represents these phases in the XRD spectra. The CaCO₃ formed from the carbonation reaction is precipitated in the pore structure of the matrix also acting as a binder and refining the pore size distribution. The interface between the cellulose fibers and the cement matrix in the carbonated composites was improved, decreasing the typical transition zone around the cellulose fibers that prejudice the fiber–cement performance at long term.

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1. Introduction

The studies of new technologies for fiber–cement products led to the investigation of alternative reinforcing and processing fibers, such as cellulose pulp or macro fibers and synthetic polymeric

fibers. Vegetable fibers are widely available in most developing countries and present several interesting characteristics, such as low density, renewable character, biodegradability and availability at low cost and in a wide variety of morphologies and aspect ratios. On the other hand, synthetic fibers are developed from fossil fuels and there is a concern by the fiber–cement industries about the availability of these fibers to the market demand in the near future and about their high costs, which can make it impracticable for

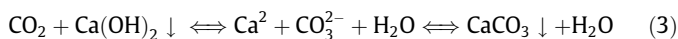
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fiber–cement production at affordable costs. Reducing the content of synthetic fibers is an effective way to reduce fabrication costs in air-cured fiber–cement products [1]. Fiber–cement materials have been widely used in the world due to their versatility for manufacturing construction elements, such as flat boards for walls, corrugated sheets and tubes, present in most of houses in developing regions as well as in agricultural and industrial buildings [2].

One of the drawbacks in the use of cellulose fibers is their mineralization and degradation of their constituents in the high alkalinity of the Portland cement matrix, with the pH of about 13. Mineralization of the cellulose fibers is caused by the free ions from the dissolution of Portland cement phases that penetrate into the fiber cavity (lumen), leading to the re-precipitation of ettringite/monosulfate and calcium hydroxide into the fiber. This re-precipitation of solids into the fiber voids induces the embrittlement of the cellulose fibers. Secondary (re-precipitated) ettringite formation has been previously suggested as one of the degradation mechanisms of pulp fiber into cement matrix [3,4]. This low durability of the cellulose fibers in the cement matrix causes the reduction of the mechanical performance of the fiber–cement composites due to the fiber mineralization and loss of adhesion between fiber and matrix [3,5,6]. Hence, the optimal situation would be to protect the cellulose fibers from mineralization and chemical attack with a less aggressive matrix (e.g. lower alkalinity), maintaining the fiber strength and the quality of the fiber bridging that guarantee the composite ductility during its lifetime [7].

Early age carbonation had been successfully used in partial replacement of steam in concrete-pipe production to improve durability [8] and can result in a number of other benefits in the pre-cast concrete production, including the improvement of the initial strength, dimensional stability, cement savings and less product waste [9–11]. This accelerated carbonation of the cement matrix can be applied to improve the durability of the cellulose fiber–cement composites, because it reduces the alkalinity of the cement matrix and reduces the pH of solution, resulting in a less aggressive solution to the cellulose fibers [12]. The carbonation is the reaction of cement hydration products with carbon dioxide (CO₂), which could also represent great impact on the sustainable growth of the construction industry in the future [13]. The carbonation is an acid–base neutralization reaction of the alkaline nature of cement. The CO₂ first dissolves in the pore solution and then reacts with the OH[−], forming CO₃^{2−} (Eq. (1)). Consequently, the pH of the pore solution decreases. As the solution is saturated in calcium ions, due to the C₃S hydration, (pH of 12.5, Eq. (2)), the precipitation of CaCO₃ starts (Eq. (3)), according to the following reactions [14]:



Additionally, the consequences of carbonation are the stability of the mechanical behavior [11], densification of the cement matrix and reduction of its permeability (capillary) and porosity, which constitutes a positive process with respect to the pores sealing of the cement [15].

In the fiber–cement composites, especially those produced by the Hatschek process (wet method), the carbonation of the matrix is enhanced due to its high capillary porosity as a consequence of the presence of significant volume of fibers and also due to the vacuum drainage in the fabrication process, which facilitates the penetration of CO₂ within the composite. In a previous study, Tonoli et al. [5] evaluated the effect of accelerated carbonation, applied

after 28 days of cure, on the durability of cellulose fiber–cement composites. They concluded that this procedure improved the initial mechanical strength, decreased the calcium hydroxide content and led to a denser matrix, but the embrittlement of the composites after severe ageing was not avoided. That study showed the necessity to anticipate the fast carbonation for initial periods of cure, which would not allow time for the alkali to attack the cellulose fibers during the cement hydration. Therefore, the present work contributes to the widespread use of accelerated carbonation as a way to mitigate cellulose degradation into the fiber–cement composites. However, there is still a lack of information about the changes promoted by the accelerated carbonation on the mineralogical phase composition and microstructure of hybrid (reinforced with cellulose and synthetic fibers) fiber–cement composites submitted to ageing cycles.

The carbonation can occur in use naturally by the reaction between alkaline matrix of the fiber–cement and atmospheric CO₂, but the natural reaction is very slow. Then, the accelerated carbonation can complete its reaction within hours and has been proposed as an alternative technology to the natural carbonation [16,17]. In this context, the aim of this work is to evaluate the effect of accelerated carbonation in the early stages and ageing on the mineralogical composition and microstructure of fiber–cement composites with hybrid reinforcement.

2. Material and methods

2.1. Sampling and accelerated carbonation

The fiber–cement samples with nominal thickness of 4 mm were obtained from the Hatschek process. The Hatschek process is the most employed in the fiber cement industry worldwide and consists of producing fiber–cement boards by assembling thin layers (up to 1 mm) made of sub-layers (approximately 0.3 each) that are formed in rotary sieves and progressively accumulated in the filtering felt from a suspension of Portland cement, mineral additions (limestone filler for example), cellulose and synthetic fibers and water. Vacuum is applied to remove the excess water from the layer before it is transferred to the formation cylinder (resembling a paper production process) where the layers are stacked. Then, the green fiber–cement sheet is cut, shaped (e.g. corrugating) and cured. More detailed description of the Hatschek process can be found in Dias et al. [1].

The average formulation is composed of about 70 wt% of Portland cement type II-E (NBR 11578:1 standard), 24 wt% of calcium carbonate filler, 4 wt% of eucalyptus cellulose pulp and 2 wt% of polyvinyl alcohol (PVA) fibers. The calcium carbonate filler (limestone) is normally used in the industry for partial substitution of Portland cement in order to reduce costs concerning the production of fiber–cement.

The preconditioned (water-saturated air) samples were submitted to 10 h of accelerated carbonation in the climate chamber at temperature of 60 °C and relative humidity (RH) of 90%. The CO₂ content in the climate chamber was monitored to about 15 vol%. Then the carbonated composites were taken out and maintained in water-saturated air at temperature of 25 °C until the characterization tests. The conditions were the same for the non-carbonated specimens, however without applying CO₂. The fiber–cement samples were cut into test specimens (160 mm × 30 mm × 4 mm) with a diamond disk cooled with water.

2.2. Soak and dry accelerated ageing cycles

The soak and dry accelerated ageing cycles involved comparative analysis of the mineralogical composition and microstructure of the composites, before and after this test. Composites were successively immersed into water at 20 ± 5 °C for 170 min and, after resting for 10 min, they were heated to a temperature of 70 ± 5 °C for 170 min in a ventilated oven. An interval of 10 min (at room temperature) is usual prior to the beginning of the next cycle, as recommended by EN 494:1994 standard [18]. Each soak and dry set represents one cycle, which was repeated 200 times (i.e. 200 ageing cycles) continuously over 24 h, as used in Tonoli et al. [19]. This method simulates natural ageing in severe conditions, although additional studies are needed to determine the most relevant accelerated conditions corresponding to natural weathering, in order to predict more precisely the long-term behavior of the composites produced.

2.3. X-ray diffraction (XRD)

The diffractometer Bruker D8 Advance model consisted of a high voltage generator 3 kW X-ray tube with copper anode (Cu K α radiation of 1.54 Å), usually operating at 40 kV and 50 mA, LYNXEYE detector, carrying an anti-scatter slit of 3 mm

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