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Effect of accelerated carbonation on the microstructure and physical properties of hybrid fiber-cement composites

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ABSTRACT

Carbonation takes place in the fiber-cement composites through the diffusion of carbon dioxide (CO_2) through the unsaturated pores of the cement matrix, and through its reaction with the hydration products of the Portland cement (mainly calcium hydroxide and CSH phases). The use of this technology in the fiber-cement production consists of an interesting procedure to prematurely decrease the alkalinity of the cement matrix, which is potentially harmful to the cellulose fiber reinforcement. It is also an initiative to CO_2 sequestration and partial replacement of petroleum-based fibers. Therefore, the objective of the present work is to show the impact of accelerated carbonation on the microstructure and physical properties of fiber-cement composites reinforced with cellulose pulp and synthetic fibers. The effectiveness of the accelerated carbonation was confirmed by thermogravimetric (TG) analysis. Accelerated carbonation increased bulk density (BD) and decreased apparent porosity (AP). The SEM micrographs show that the calcium carbonate ($CaCO_3$) formed from the carbonation reaction is precipitated in the pore structure of the matrix. The interface between the cellulose fibers and the cement matrix in the carbonated composites was improved, decreasing the typical voids around the cellulose fibers that prejudice the fiber-cement performance at long term.

1. Introduction

Fiber-cement products have been widely used in the world due to their versatility for manufacturing construction materials, such as flat boards for walls, corrugated sheets and water tanks, present in most of houses in developing regions as well as in agricultural and industrial buildings (Ikai et al., 2010).

The restrictions in the use of asbestos led to the investigation of alternative reinforcing and processing fibers, such as vegetable and synthetic 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 current market demand and about the high costs (as high as approximately 40% of the total cost of raw materials), which can make it impracticable for fiber-cement production at affordable costs. Reducing the content of synthetic fibers is an effective way to reduce production costs in air-cured fiber-cement products (Dias et al., 2010).

One of the drawbacks in the use of vegetable/cellulose fibers is their mineralization in the high alkali Portland cement matrix, with the pH around 13. Mineralization of the cellulose fibers is caused by the free ions from the dissolution of Portland cement phases that penetrated into the fiber cavity (lumen), leading to the re-precipitation of ettringite/monosulphate and calcium hydroxide into the fiber and inducing the stiffening 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 (Mohr et al., 2005, 2006). This low durability of the vegetable fibers in the cementitious matrix causes reduction of the mechanical performance of the fiber-cement composites due to the fiber mineralization and loss of adhesion between fiber and matrix (Mohr et al., 2005; Tonoli et al., 2010). Hence, the optimal situation would be to protect the cellulose fibers from mineralization with a less aggressive matrix, maintaining the fiber strength and the quality of the fiber bridging that guarantee composite ductility.

Accelerated carbonation of the cement matrix can be employed to improve the durability of the cellulose fiber-cement composites, because it reduces the alkalinity of the cement matrix, lowering the pH, and making it less aggressive to the cellulose fibers (Toledo Filho et al., 2003). Also, the consequences of carbonation on cement are the stability of the chemical hydration products and

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mechanical properties (Soroushian et al., 2012); densification of the cementitious matrix and reduction of its permeability (capillary) and porosity, which constitutes a positive process with respect to the sealing quality of the cement (Lesti et al., 2013). The carbonation is the reaction of cement hydration products with carbon dioxide (CO_2), which could represent great impact on the sustainable growth of the construction industry in the future (Silva et al., 2009).

In fiber-cement composites, the carbonation of the matrix is enhanced due to its high porosity in consequence of the fibers effect, which facilitates the penetration of CO_2 within the composite. In a previous study, Tonoli et al. (2010) 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 aging 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 performance and microstructure of hybrid (reinforced with cellulose and synthetic fibers) fiber-cement composites.

The dissemination of the use of accelerated carbonation during the initial cure fits well with the growing necessity of more sustainable technologies and raw-materials, which is associated to the consume of the CO_2 emissions eventually generated by the industrial processes, and allows partial substitution of the more expensive petroleum derivative polymer fibers used to retain the durability of the composites under aggressive climatic conditions. According to Soroushian et al. (2012), CO_2 curing of cellulose fiber-cement has also shown to enhance productivity and engineering properties (including dimensional stability) of the end product.

Carbonation can occur in use naturally by the reaction between alkaline matrix of the fiber-cement and atmospheric CO_2, but the natural reaction is very slow. Then, accelerated carbonation can complete its reaction within hours and has been proposed as an alternative technology to the natural carbonation (Lim et al., 2010). In this context, the aim of this work was to evaluate the effect of accelerated carbonation on the microstructure and physical properties 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 one in producing fiber cement components, and consists of producing fiber-cement boards by assembling thin layers (up to 1 mm) made from a suspension of Portland cement, mineral admixtures (limestone filler for example), fibers (cellulose and synthetic fibers) and water. Vacuum is applied to remove water from the layer before it is transferred to the formation cylinder (resembling a paper production process) where the layers were 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. (2010).

The average formulation is composed of around 70% of Portland cement type II-E (NBR 11578, 1991), 24% of calcium carbonate filler, 4% of eucalyptus cellulose pulp and 2% 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.

Preconditioned (water-saturated air) samples were submitted to 4 h and 10 h of accelerated carbonation in a climate chamber at around 60 °C and 90% relative humidity (RH). The CO_2 content into the climate chamber was monitored to around 15% in volume. Then the carbonated composites were taken out and maintained in water-saturated air at around 25 °C until the characterization tests. The conditions were the same for the non-carbonated specimens, however without applying CO_2. The fiber-cement samples were then cut into test specimens (160 mm × 30 mm × 4 mm) with a diamond disk cooled with water.

To check whether the accelerated carbonation took place or not, thermogravimetric (TG) analysis was performed in a NETZSCH STA 409 Model PG thermobalance. It was used the same mass (1.000 ± 0.001 g) for the TG analysis for the different samples, in order to minimize distorted results from heterogeneous mass quantity (Taylor, 1997; Hoppe Filho, 2008). The analysis was performed in nitrogen gas atmosphere with 60 mL/min flow. The heating rate was of 10 °C/min, in an alumina crucible with a volume of 3.4 mL.

2.2. Scanning electron microscopy

Scanning electron microscopy (SEM) was used with secondary electron (SE) image detector, operated at 20.0 kV accelerating voltage, for visualization of the microstructure on the fractured surface of the composites. Fractured surfaces were gold coated in a Bal-Tec SCD050 coating system before being analyzed in a Zeiss LEO Evo 40 microscope.

A back-scattered electron (BSE) image detector operating at around 15.0 kV and 20.0 kV was applied for viewing cut and polished surfaces. The BSE imaging was used to study the matrix transition zone. The preparation of specimens for BSE was accomplished with vacuum (80 kPa gauge) impregnation using epoxy resin. BSE samples were manually polished with silicon carbide abrasive paper with sequential grit sizes of 320, 600, 1200 and 2000 for 2 min each, using alcohol as lubricant. A final polishing was carried out using in turn 8–4, 4–2 and 1–0 μm diamond polishing compound during 4, 2 and 1 min each size respectively. Polished samples were carbon coated before being analyzed in a FEI Quanta 600 (FEG) microscope.

Around twenty SEM micrographs were obtained for each composite treatment and just the typical images of the microstructure observed were used in this manuscript.

2.3. Physical characterization of the fiber-cement composites

Apparent porosity (AP) and bulk density (BD) of the composites were obtained from the average of six test specimens (160 mm × 30 mm × 4 mm) for each condition, following the procedures specified by ASTM C 948-81 (2009). A quantitative estimative on the amount of CO_2 uptake during 4 and 10 h of carbonation was done from the values of dry weight evolution of the carbonated composites.

3. Results and discussion

3.1. Evaluation of the accelerated carbonation

Fig. 1 depicts the mass loss (TG) and differential weight loss (DTG) of the composites at the different carbonation conditions. During the sample preparation (milling) for TG analysis, the PVA fibers were removed from the powder sample by screening. This is the explanation for no observation of the decomposition peak between 225 and 295 °C related to PVA fibers.