INFLUENCE OF THE INITIAL MOISTURE CONTENT ON THE CARBONATION DEGREE OF FIBER-CEMENT COMPOSITES

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ABSTRACT

The objective of this work is to show the effect of the initial moisture content of fiber-cement composites on their carbonation degree (CD) after their exposure to CO_2 atmosphere. Fiber-cement composites were produced by Hatschek process and subjected to carbonation at early age for 10 h of exposure to 15% of CO_2. Part of the composites was previously subjected to different oven-drying times (20, 40, 60 and 120 min). The CD was determined using thermogravimetric measurements. As expected, the higher the time of drying, the higher was the moisture remover from the composites. Accelerated carbonation caused the decrease of Aft, C-S-H, AFm, monocarbonates and $Ca(OH)_2$; and increased the $CaCO_3$ content. The longest time of drying (120 min) led to the lower content of $Ca(OH)_2$ and the higher content of $CaCO_3$. Fiber-cement composites not dried and those dried for 40 min presented 19 and 21% of moisture respectively and led to CD of 34 and 48%, respectively; while those fiber-cements dried for 120 min (14% of moisture) presented a CD of 55%. Higher CDs led to the significant decrease in water absorption and apparent porosity of the composites, and caused an increase in bulk density of the fiber-cement composites. Therefore, the control of the initial moisture and free pores of the fiber-cement composites seems to be a key strategy to improve the efficiency of the accelerated carbonation of the fiber-cement composites.

KEYWORDS:

Porosity; degree of carbonation; carbonation at early ages; accelerated carbonation.

INTRODUCTION

Fiber-cement products have been extensively used in constructions worldwide due to their versatility for manufacturing construction elements, such as flat boards for walls, sidings, corrugated sheets and tubes, that are present in residential, agricultural and industrial buildings (Ikai et al., 2010; Ardanuy et al., 2015; Hakamy et al., 2015).

The accelerated carbonation of the cement matrix has been reported as a strategy to improve the durability of the cellulose fiber-cement composites, because it reduces the alkalinity of the cement matrix, decreasing the pH for values lower than 9, and making the environment less aggressive to the cellulose fibers (Toledo Filho et al.,

2003; Pizzol et al., 2014). Furthermore, the consequences of carbonation are the stability of the mechanical behavior (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 pores sealing of the cementitious material (Lesti et al., 2013).

In a previous study, Tonoli et al. (2010a) showed the advantages of anticipating the fast carbonation for initial periods of curing, which would not allow time for the alkali to attack the cellulose fibers during the cement hydration. Therefore, in this initial condition, water plays an important role in the carbonation rate, as carbonation does not occur without water which acts as a catalyser in the carbonation process. However, water saturation can fill the pores and prevent CO_2 gas penetration into the fiber-cement capillary network. In fact, carbonation reaches the highest efficiency when samples have low free water content and have a reasonable pore volume to optimize the carbonation process (Kottititum et al., 2018). A better understanding of the changes promoted by different initial moistures of the fiber-cement on the efficiency of their carbonation may permit new strategies for predicting the evolution of microstructural and macroscopic properties, and ultimately contribute to engineering of the overall performance of the fiber-cement composites.

The objective of this work is to show the effect of the initial moisture content of fiber-cement composites on their carbonation degree (CD) after their exposition to high CO_2 concentration at atmospheric pressure.

EXPERIMENTAL

Materials

A blend of virgin and recycled cellulose pulp with Schopper Riegler (SR) value varying from 50-80 was used. Polypropylene (PP) fibers (Brazil) used were ~10 mm long, presenting real density between 0.9 to 1.0 g/cm3 and dtex of ~1.12.

The approximate composition by mass of the blended Portland-Limestone cement (CPII-F, Brazil) used was: ~60.2% CaO, ~16.8% SiO2, ~4.2 Al2O3, ~2.4 F2O3. Additionally the average density was ~2.65 g/cm3 and the specific surface area was ~5200 cm2/g, with average particle diameter of ~18.4 µm. Calcium carbonate filler (Brazil) presented average density of ~2.75 g/cm3 and specific surface area of ~12500 cm2/g, while particle diameter of ~21.2 µm.

Fiber-cement production and drying

The composition by mass of the fiber-cement composites was (dry ingredients): ~95% cementitious matrix and ~5% of fibrous reinforcement (cellulose pulp + PP fibers).

Green fiber-cement sheets were obtained from an industrial Hatsheck process, with the initial moisture of around 27% by mass. Then, the sheets were packed with polyethylene films for an initial curing at saturated condition at 25ºC for two days. The resulting sheets were then exposed to drying at ~25ºC and relative humidity (RH) of ~55% for 20, 40, 60 and 120 min. Reference samples were maintained packed as in the initial condition.

Accelerated carbonation

After each condition of drying, the fiber-cement sheets were subjected to carbonation at early age for 10 h of exposition to 15% (v/v) of CO_2 at atmospheric pressure, at 60ºC and 90% relative humidity (RH).

After 10 h of carbonation, a supplementary cure was applied leaving the fiber-cement sheets in sealed plastic bags for 8 days at 25ºC. The moisture of the fiber-cement sheets in this stage after carbonation was around 7%.

Thermogravimetry of the fiber-cement composites

Samples of the different treatments were immersed in isopropyl alcohol for 1 h and oven-dried at 70ºC for 24 h and kept in sealed plastic bags, in order to interrupt the cement hydration. To check the carbonation extension, thermogravimetric (TGA) analysis was recorded using a Netzsch equipment with STA 409 simultaneous analysis system using a dynamic nitrogen stream (flow rate = 60 mL/min) at a heating rate of 10°C/min. The same mass (around 1 g) was used for all samples, in order to reduce inaccurate results from heterogeneous mass

quantities (Taylor, 1997). The results of thermogravimetry were also used for determination of the carbonation degree (CD) reported in the next section.

Determination of the carbonation degree (CD)

The carbonation degree (CD) was determined using Eq. (1), as presented by Matsushita et al. (2000).

$$CD\ (\%) = (C - C_0) / (C_{max} - C_0) * 100 \tag{1}$$

Where C is the CO_2 amount in the carbonated sample, C_0 the percentage of CO_2 in the non-carbonated sample, and C_{max} is the theoretical amount of CO_2 necessary to react with the total amount of CaO in the Portland cement and originating $CaCO_3$. The percentage of CO_2 absorbed may be calculated by the thermal decomposition of the carbonates (mainly $CaCO_3$) obtained by thermogravimetry. The temperature of $CaCO_3$ decomposition occurs between 550 and 1000ºC (Kim et al., 2013; Goodbrake et al., 1979) and the mass loss in this temperature range is the amount of CO_2 in the sample. The theoretical maximum capacity of CO_2 absorption (C_{max}) was determined as presented in Eq. (2) (Huntzinger et al., 2009).

$$C_{max}\ (\%) = 0.785\ (CaO - 0.56 * Ca(CO_3) - 0.7 * SO_3) + 1.091 * MgO + 0.71 * Na_2O + 0.468 * K_2O \tag{2}$$

Where it was assumed that all CaO, MgO, Na_2O and K_2O formed in the cementitious matrix reacted with the CO_2, forming carbonates (Steinour, 1959; Huntzinger et al., 2009). This equation also excludes the unreactive calcium carbonate present in the CaO account, which is a conservative estimative of the maximum capacity of CO_2 absorption of the cement used.

Physical characterization

Apparent void volume (AVV), water absorption (WA) and bulk density (BD) of the composites were calculated from the average of six test specimens (165 mm x 40 mm x 5 mm) for each condition, following the procedures specified at ASTM C 948-81 (2009).
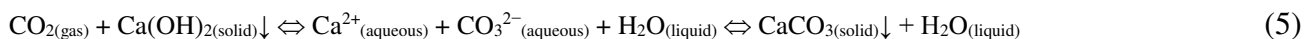
RESULTS AND DISCUSSION

Moisture Content and porosity of the fiber-cement for each drying condition
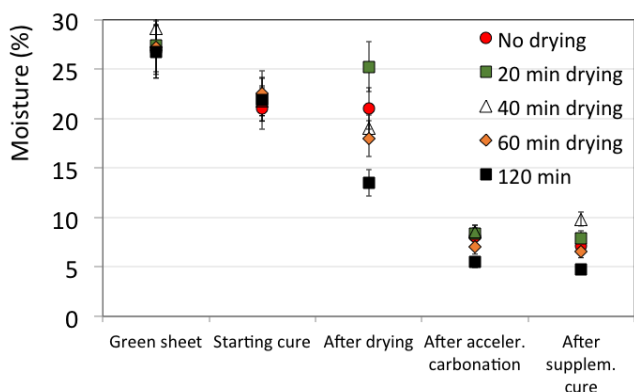
Figure 1 shows the moisture content of the fiber-cement composites after each curing step. The green fiber-cement sheets had around 27% of moisture. After the initial curing in laboratory (2 days at 25ºC, at around 100% RH), the fiber-cement composites presented moisture of around 22%, that is, the moisture immediately before the submission to the different drying periods. The higher drying time (120 min) decreased the moisture content of the fiber-cements to approximately 14% before the application to accelerated carbonation. As expected, the higher the drying time, the higher the withdrawal of moisture from the pores in the composites and the lower their green density before carbonation. In addition, the higher the drying time, the higher the porosity and the percentage of pores not filled with /moisture. In general, fiber-cement composites are produced by the Hatschek process, which gives to them high initial porosity in consequence of significant volume of fibers and due to the applied vacuum drainage in the fabrication process. However, in the initial period of curing, great part of the pores is filled with water and can adversely affect the rate of carbonation of the composite. Then, removing part of this moisture from the fiber-cement pores may facilitate the penetration of CO_2 within the composite.

The accelerated carbonation decreased drastically the moisture content of the fiber-cement composites, even with 90% of RH in the environmental chamber. This happened because the carbonation reaction required water for dissolution of CO_2 into the pores. CO_2 first diffuses into the empty pores, dissolves in the pore solution and then reacts with the OH^-, forming CO_3^{2-} (Eq. 3). Simultaneously, the dissolution of $Ca(OH)_2$ also occurs, forming Ca^{2+} and OH^- (Eq. 4). The carbonation is an acid-base neutralization reaction of the alkaline nature of cement (Frías & Goñi, 2013). As the solution is saturated in calcium ions, due to the C_3S and C_2S hydration (pH

of ~12.5), the precipitation of calcium carbonate (CaCO$_3$) polymorphs (calcite, vaterite or aragonite) starts, concomitantly with the release of water (Eq. 5) and the pH decrease in the pore solution. Therefore, carbonation decreases the internal moisture of the composites because the carbonation reaction releases water to the environment while forming CaCO$_3$ in their microstructure. After carbonation, the moisture content of the fiber-cements reduced to around 8-9% for shorter drying times (0, 20 e 40 min), while for samples carbonated after 60 and 120 min of drying, the moisture decreased to 6-7%.

$$CO_{2(gas)} + 2OH^-_{(aqueous)} \Leftrightarrow CO_3^{2-}{}_{(aqueous)} + H_2O_{(liquid)} \qquad (3)$$

$$Ca(OH)_{2(solid)}\downarrow \Leftrightarrow Ca^{2+}_{(aqueous)} + 2OH^-_{(aqueous)} \qquad (4)$$

$$CO_{2(gas)} + Ca(OH)_{2(solid)}\downarrow \Leftrightarrow Ca^{2+}_{(aqueous)} + CO_3^{2-}{}_{(aqueous)} + H_2O_{(liquid)} \Leftrightarrow CaCO_{3(solid)}\downarrow + H_2O_{(liquid)} \qquad (5)$$

After the supplementary curing, the moisture content of the fiber-cements continued to decrease, due to the residual cement hydrations and remaining carbonation reactions due to the carbonated water in the microstructure of the composites, releasing water from the precipitation of the CaCO$_3$. The moisture content of the fiber-cement with longer drying time (120 min) was lower than 5% after supplementary cure. This result suggests the necessity of inclusion of additional moisture after carbonation as a way to proportionate an efficient hydration.

Figure 1. Average and standard deviation values of moisture content of the fiber-cement composites just after the production (green sheet), after the starting cure of 2 days in laboratory, after the different drying times (20, 40, 60 and 120 min), after accelerated carbonation, and after the supplementary cure.
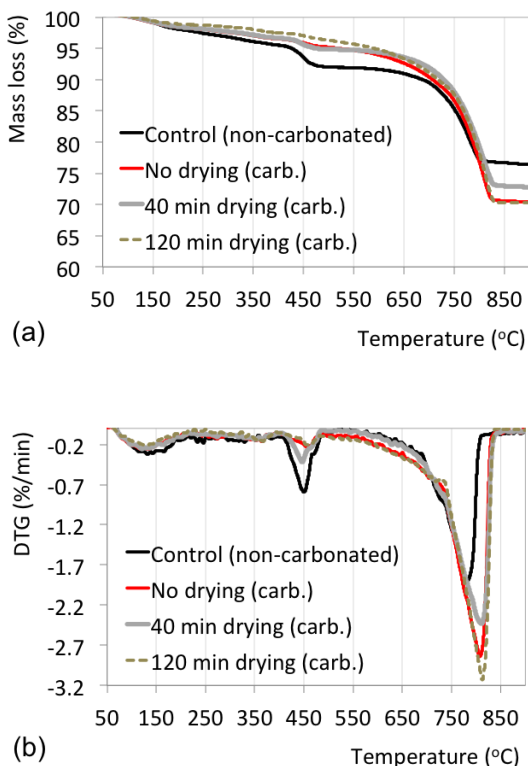
Thermogravimetry and carbonation degree (CD)

Figure 2 depicts the thermogravimetry (TG) and differential weight loss (DTG) of the composites at the different carbonation conditions. Endothermic events can be identified in the three main zones of decomposition: 50-250°C (hydrated phases: C-S-H, AFm, Mc and Aft) (Ylmén e Jaglid, 2013), 400-450°C (Ca(OH)$_2$ or portlandite – P) and 500-850°C (less stable and stable carbonates). The changes in the weight loss between 90 and 200°C correspond to the interaction of C-S-H, ettringite (C$_3$AS$_3$H$_{32}$ – AFt), monosulfoaluminate (C$_3$ASH$_{12}$ – AFm), monocarboaluminate (C$_4$AcH$_{11}$ – Mc) with CO$_2$ as also observed by Almeida et al. (2010, 2013) and Frías & Goñi (2013) adopting different curing conditions and cement matrices. These results indicate that the carbonation rate occurred in a high extension, reducing the weight loss in the range between 50-250°C of the carbonated composites. The C-S-H is progressively decalcified, being converted to CaCO$_3$ and S-H (silica hydrates). Dissolution of Ca(OH)$_2$ generally takes place more readily, while decalcification of C-S-H occurs when Ca(OH)$_2$ is inaccessible or locally reduced (Tonoli et al., 2016). Nevertheless, when the porosity in the

fiber-cement is sufficiently high to permit constant CO_2 diffusion, the $Ca(OH)_2$ is further reduced and the calcium from C-S-H also reacts with carbon dioxide (Fernández-Bertos e al., 2004).

The temperature range of between 295 and 370ºC corresponding to the degradation of the cellulose fibers, and a higher mass loss was observed for fiber-cement carbonated after drying for 120 min. Figure 2 also shows the changes in the endothermic peaks between 450 and 500ºC related to the decomposition of the $Ca(OH)_2$. The mass loss in this range decreased as the carbonation took place. Smaller mass loss at this temperature range was observed for the carbonated samples. Finally, the accelerated carbonation increased the mass loss at the range between 500 and 850ºC, which are associated to the carbonates. Carbonation increasedthe mass loss between 500 and 750ºC related to the thermal decomposition of poorly crystalized $CaCO_3$ polymorphs and at the range between 750 and 850°C related to decomposition of the well-crystalized $CaCO_3$, as also reported by Rostami et al. (2011). According to Thiery et al. (2007), the well-crystallized calcite decomposes between 780-990°C, and the presence of vaterite and aragonite causes the carbonates to decompose at lower temperatures (680-780°C), while decomposition between 550-680°C is probably associated with amorphous calcium carbonate. The decomposition ranging between 500 and 850ºC for non-carbonated (control) composites is related to the carbonate content in the as-received cement and calcium carbonate content from limestone filler, the supplementary material applied in the fiber-cement formulation, in order to reduce production costs and increase dimensional stability of the final product. Additionally, the accelerated carbonation increased the temperature of maximum decomposition of the carbonates (from around 770ºC to 810ºC).

CD and absorbed CO_2 increased with the decrease of initial moisture of the fiber-cements from different drying conditions (Table 1). Lin et al. (2015) reported a higher increase of CD for samples with moisture between 5 and 15%, and showed that moistures of around 25% are too high for the diffusion of CO_2 into the microstructure. Fiber-cements not dried and samples dried for 40 min had moisture contents of around 19-21% before exposition to CO_2, and resulted in CD between 34 and 48%, while samples dried for 120 min reached a moisture content of 14% and resulted in the CD of 55%. The results of CO_2 absorbed indicate that for a corrugated sheet of 5 mm thickness and with mass of 18 kg, the absorption of CO_2 will probably reach around 1.6 kg, with the formation of around 3.6 kg of $CaCO_3$.

Figure 2. Typical thermogravimetry curves – TG (a); derivatives – DTG (b); of the samples before carbonation and carbonated in different conditions of drying (no drying, and dried for 40 and 120 min).

Table 1 – Content of absorbed CO₂ before and after normalization and carbonation degree (CD) determined with the CaCO₃ content formed by accelerated carbonation after different times of drying (no drying, and dried for 40 and 120 min).

	CO_2 absorbed (%)	CD (%)
Control (non-carbonated)	0	0
No drying (carbonated)	9.1	48
40 min drying (carbonated)	6.4	34
120 min drying (carbonated)	10.4	55

Physical properties

A decrease in water absorption (WA) and apparent porosity (AP) with the increase in initial drying rate was observed as a consequence of the significant increase of bulk density (BD) with the increased carbonation. The 20 min of drying was not enough to significantly ($p < 0.05$) change the physical properties of the carbonated samples in relation to the undried samples. The carbonation of $Ca(OH)_2$ is followed by an increase in the volume of solids (Johannesson et al., 2001). Silva et al. (2009) showed the reduction of 15% in porosity and approximately 6.5% increase in the bulk density of the composites submitted to accelerated carbonation for 14 days. $CaCO_3$ is precipitated in the pore structure of the composite matrix, filling up the voids and thereby blocking the free intake of water due to the pore size refinement (Hyvert et al., 2010). Then, these pores become almost closed instead of the initially predominant capillary pores, and consequently less permeable to silicates, so decreasing the diffusion of Si and S ions during the curing periods.

The $CaCO_3$ polymorphs produced by accelerated carbonation were precipitated in the pore structure of the carbonated matrix, filling the voids and thereby blocking the intake of free water and other hydration products as opposed to the initially predominant capillary pores. These closed pores decrease permeability and diffusion of Si, S and Al ions during the curing and secondary hydration. Consequently, less AFt phase is formed around the fibers because of lower diffusion of S- and Al-rich phases. In carbonated composites, the greater adhesion between the cement and the cellulose fiber in conjunction with the intensive lumens filled with the carbonated products may lead one to expect the embrittlement of the composite. However, the fiber cell wall in carbonated composites would be less attacked by the alkaline ions, resulting in lower mineralization of the fiber cell wall. Tonoli et al. (2016) showed that S is widely distributed in the carbonated matrix, while Al-rich phases are concentrated in the C_3A (tricalcium aluminate) or C_4AF (tetracalcium aluminoferrite) anhydrous grains, and have not been solubilized and re-precipitated around the cellulose fibers. They reported therefore that less Aft, Afm and Mc phases are formed in the transition zone around the fibers because of the lower diffusion of S- and Al-rich phases. These S-rich phases are responsible for peeling off cellulose chains, degrading its structure at long term. Furthermore, it is known that the carbonates formation leads to the decrease of the alkalinity of the matrix (Pu et al., 2012), and therefore providing a less aggressive environment to the cellulose fibers. The chemical stability of the carbonated hydration products is also an important advantage (Soroushian et al., 2012) for the macroscopic performance of the material.

CONCLUSIONS

Accelerated carbonation is presented here as an interesting strategy to prematurely decrease the alkalinity of the cement matrix by consumption of ions Ca^{2+} and OH^- in the pore solution. Accelerated carbonation caused the decrease of Aft, C-S-H, Afm, monocarbonates and $Ca(OH)_2$; and increased the $CaCO_3$ content. The greater time of drying (120 min) led to a lower content of $Ca(OH)_2$ and the higher content of $CaCO_3$. Fiber-cement composites without drying and with 40 min of drying presented 19 and 21% of moisture respectively and led to carbonation degree (CD) of 48 and 34% respectively; while those fiber-cements dried for 120 min (14% of moisture) presented a CD of 55%. Higher CDs led to the significant decrease of water absorption and apparent porosity of the composites, while causing an increase in bulk density of the fiber-cement composites. Therefore, the control of the initial moisture and free pores of the fiber-cement composites seems to be an important strategy

to improve the efficiency of the accelerated carbonation of fiber-cement composites. $CaCO_3$ polymorphs filling the voids and the pore structure of the carbonated matrix resulted in the densification of the matrix, making the matrix more chemically stable with lower diffusion of ions from curing and secondary hydration. It improves the contact between fibers and cement matrix thus preventing the movement of water due to the decrease in the pore size network and diffusion of degradation products (e.g. Ca and S ions) into the composite.

ACKNOWLEDGMENTS

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