



## Supercritical carbonation treatment on extruded fibre–cement reinforced with vegetable fibres



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### ABSTRACT

The objective of this research was to evaluate the effects of supercritical carbonation treatment for 2 h on the main hydrated phases of the cement matrix (calcium hydroxide and calcium silicate hydrate) and durability of extruded fibre–cement reinforced with bleached eucalyptus pulp and residual sisal chopped fibres. The thermal analysis, bulk density, porosity, physical characteristics and mechanical performance were evaluated before and after 200 soaking and drying cycles for following the degradation of the material under accelerated ageing conditions. The higher carbonation rate during the early stage of curing period decreased the porosity by sealing the opened pores around vegetable fibres and, consequently, led to lower water absorption and higher bulk density in the composites. The average MOR-values showed a significant increase in the case of the supercritical carbonated extruded fibre–cement in the initial age and after accelerated ageing. Besides, after 200 soaking and drying ageing cycles, the average values of energy of fracture ( $\gamma_{Wof}$ ) of the carbonated composites decrease only 28%, showing evidences of the preservation of microstructural stability and toughness of the fibre–cement composites after supercritical CO<sub>2</sub> treatment.

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### 1. Introduction

The vegetable fibre and cellulosic pulp derived from renewable resources, which are widely available in most developing countries, can be used as appropriate materials for cementitious matrix reinforcement. However, significant losses in mechanical performance in the long term have been observed in vegetable fibre–cement composites after natural or accelerated ageing (e.g. wet/dry cycling), due to the degradation mechanisms of cellulose fibres in the cementitious environment [1,2].

As strategy to mitigate long term degradation issues, part of the fibre–cement industry in the world produces fibre–cement composed of Portland cement, finely ground crystalline silica, and cellulose fibre which are autoclaved to ensure appropriate strength, durability and dimensional stability of the composites [3,4]. The autoclave is steam vented to remove air, then held at 180 °C for 6–8 h while subjected to absolute steam pressures ranging generally from about 0.7 MPa to about 1.4 MPa to give a mild cure, a

moderate cure or a severe cure [5–7]. However, this autoclave curing has exceedingly high energy demands.

Accelerated carbonation can be an alternative route to partially solve long term durability issues of cellulosic fibre–cement and also in order to make fibre–cement composites more stable since initial ages under different humidity conditions [8,9]. The interaction between carbon dioxide and hydrated Portland cement at atmospheric pressure and ambient temperature conditions is a relatively well-known phenomenon, according to diagram in the Fig. 1. The carbonation effect in the cement paste's chemical composition, porosity and permeability has been widely reported in the literature [10–13]. Accelerated carbonation curing has been identified as a technological approach which may have potential as a mitigation strategy to reduce deterioration of cellulosic fibres and to improve mechanical behaviour and volumetric stabilization of these composites [14,15]. The initial reaction on exposure to CO<sub>2</sub> appears to be an accelerated hydration of the silicates to form a C–S–H like gel and calcite. The gel has a stoichiometry similar to that found in conventional hydration in air curing [16]. The possibility of using carbon dioxide for accelerating the hardening and stabilization of products made from Portland cement is attractive. Moreover, Shao and collaborators [17] suggested the possibility of using

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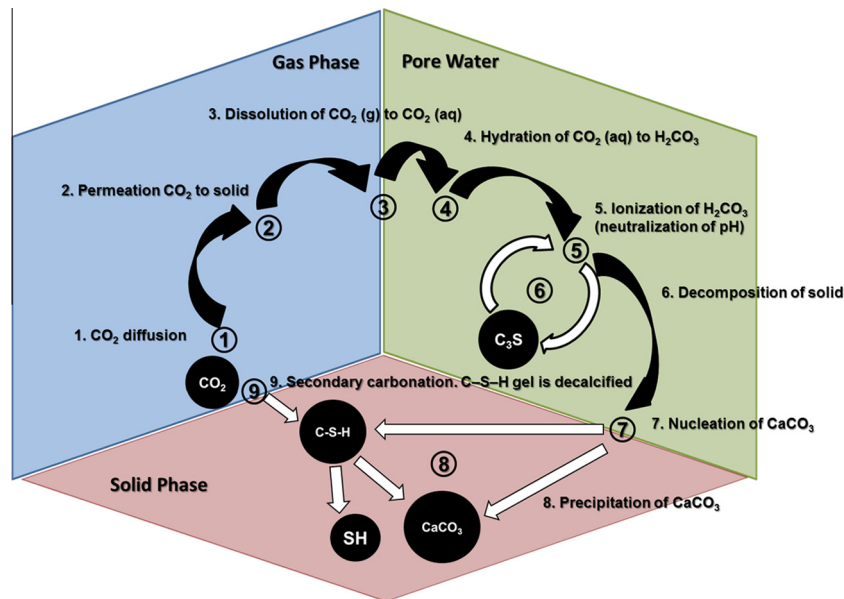


Fig. 1. Diagram with principal mechanisms for accelerated carbonation in Portland cement. Adapted from Fernández-Bertos et al. [18].

an energy efficient carbon dioxide curing to replace autoclaving in cellulose fibre reinforced cement composites production. According to these researchers, carbonation curing can save considerable amount of energy.

The development of a comprehensive model that describes the variation of composition of phases with accelerated carbonation during hydration is considered a challenge for researchers, since it must consider the kinetic and thermodynamic parameters that influence the equilibrium of each phase and the interaction thereof. Therefore, the dissolution and precipitation of chemical compounds or ionic equilibrium with carbonation process vary with the temperature, ionic strength and pH solution [11,19–22].

The use of supercritical CO<sub>2</sub> is an interesting and complex approach for the application of accelerated carbonation curing of fibre–cement composites. The effect of supercritical CO<sub>2</sub> on the structure of cements was initially investigated by the oil industry but its use as a treatment to improve the properties of cementitious composites was initiated by Jones in the late 1990s [23,24]. In the case of carbon dioxide, the fluid reaches the critical point at a pressure of 7.38 MPa and a temperature of 31.1 °C. Above these temperature and pressure, the fluid enters the supercritical state. The supercritical CO<sub>2</sub> behaves like a dense gas, acting as a solvent for water, but exhibits no surface tension, allowing penetration into very fine pores, factors by which the complete wetting of the complex pores typical for fibre–cement matrices is possible. Thus, the carbonation reactions in cementitious materials in the curing stage can be strongly accelerated by using supercritical CO<sub>2</sub> [12,13,18,25–29].

The objective of this work was to evaluate the effects of supercritical carbonation curing stage for two hours on carbonation of the main phases (calcium hydroxide and calcium silicate hydrate), the changes in bulk density, porosity, physical characteristics and mechanical performance of extruded fibre–cement reinforced with bleached eucalyptus pulp and residual sisal fibres, before and after soaking and drying accelerated ageing cycles.

## 2. Materials and methods

### 2.1. Materials and specimen preparation

The particle size distributions of the ordinary Portland cement (OPC) and ground limestone filler show that 50% of the particles

are finer than 11.8 μm and 14.5 μm, respectively. The quantitative chemical analysis was carried out by X-ray fluorescence equipment PANalytical Axios Advanced. The oxide compositions are listed in Table 1. The loss on ignition is related with the quantity of CO<sub>2</sub> in the raw materials.

For the preparation of fibre–cement was used the unrefined bleached eucalyptus cellulosic pulp produced by Fibria Celulose S.A. The pulp was analysed by a Pulpotec™ MFA-500 Morphology Fiber and Shive Analyser – MorFiTrac. The mainly parameters are the Canadian Standard Freeness (CSF) about 664 mL, as well as the length fibres of 0.83 ± 0.05 mm. For further characteristics of these eucalyptus pulp see Almeida et al. [10].

The mix design was inspired by the formulations for air cured sheets produced by the Hatschek method (changing polymer for sisal fibre) as listed in the Table 2. Brazilian ordinary Portland cement type CP V-ARI (high initial strength), correspondent to ASTM-C150, Type I [30], was chosen because of its finer particle size and higher reactivity in comparison to other blended cements available in the Brazilian market. Additionally, the CP V-ARI type of cement contains higher levels of tricalcium silicate (C<sub>3</sub>S) and dicalcium silicate (C<sub>2</sub>S) for the formation of calcium silicate hydrate (C–S–H). The water soluble polymers, hydroxypropyl methylcellulose with 86,000 average molecular weight and 5.39 cP viscosity (at 2% concentration in water at 20 °C), provided by Aditex and high

Table 1

X-ray fluorescence chemical analysis of the particulate raw material (% by mass).

Oxides	Ordinary Portland cement CP V-ARI	Limestone filler
SiO <sub>2</sub>	19.40	9.04
Al <sub>2</sub> O <sub>3</sub>	4.11	2.16
Fe <sub>2</sub> O <sub>3</sub>	2.30	1.25
MnO	–	<0.10
MgO	3.13	8.90
CaO	63.50	39.10
Na <sub>2</sub> O	0.24	0.15
K <sub>2</sub> O	1.09	0.41
TiO <sub>2</sub>	–	0.15
P <sub>2</sub> O <sub>5</sub>	–	0.16
SO <sub>3</sub>	2.97	–
Loss on ignition (1000 °C)	3.26	38.58

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