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Production of monetite-based Inorganic Phosphate Cement (M-IPC) using hydrothermal post curing (HTPC)

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ABSTRACT

A new hydrothermal post curing (HTPC) technique is developed in this study to overcome the long term evolutions such as shrinkage and cracking of Inorganic Phosphate Cement (IPC). HTPC is based on thermally induced transformation of the unstable calcium phosphates phases, e.g. brushite and amorphous calcium phosphate (ACP), into more stable phases, e.g. monetite, with prevention or minimization of the bulk shrinkage during the treatment. To achieve this effect, sufficient pore moisture is kept in IPC during the transformation stage. This way, the increase of skeletal density thus does not necessarily lead to significant global bulk shrinkage, since the contraction of the skeleton is replaced internally by an increase of the pore size. This effect can be obtained by post curing in the autoclave, when a sufficiently high temperature is used to provoke the necessary structural transformation, combined with a pressure high enough to force the pore moisture and the released bound water to stay inside the pores. After evaluation of this technique, it was noticed that the monetite percentage in the hydrothermally post cured IPC products increases from 26% to 39%, compared to the heated reference IPC. It is also verified that additional pore volume replaced the bulk shrinkage that would occur when IPC is heated without autoclave pressure. This new modified IPC end product is called monetite-based IPC (M-IPC).

1. Introduction

Calcium based phosphate cements exist for a long time but have never been used on a large scale for construction and high temperature applications. Three decades ago calcium phosphate cements were first used for the remodelling of bone defects and as coating for dental and orthopaedic implants. During this period, the first generation of bone substitute materials was proposed [1]. Rapid setting calcium phosphate cements are considered as the second generation of bone substitute materials. These cements are generally based on a combination of two or more calcium phosphate powders, reacting together in an aqueous medium to form a putty-like paste consistency [1,2]. These cements can precipitate into different end products (e.g. hydroxyapatite, calcium deficient apatite, brushite, etc.) after full conversion [3]. Chemically, the setting reaction of these cements induces high variations of pH, and their chemical reaction is highly exothermic [4–6]. A new room temperature hardening phosphate cement whose setting time can be controlled, has been developed at the Vrije Universiteit Brussel (VUB). This material is called Inorganic Phosphate Cement (IPC) and is available under the commercial brand name Vubonite [7]. Due to its non-alkaline environment during and after setting and hardening, IPC can be combined with glass fibre reinforcement, contrarily to traditional cements. Such a textile reinforced cementitious composite is an interesting material in those applications where high load-bearing capacity, good fire resistance and lightweight constructions are demanded [8]. Also, complex shapes can be produced easily without need of labour intensive shaping of the reinforcement. IPC is a two-component system, consisting of a calcium silicate powder and a phosphoric acid-based solution of metal oxides. After hardening, the material's mechanical properties are similar to those of Portland cement based materials. All components being inorganic, IPC can be used in elevated temperature applications such as moulds for shaping of composites with thermoplastic matrix or post-curing of thermosets. However, it was found [9,10] that several challenges arise when using IPC for construction and other industrial applications, in particular for high temperature applications. These challenges include shrinkage and early cracking due to ageing effects or heating. It was observed [11] that (glass fibre reinforced) IPC tends to show cracking during ageing, indicating that even at ambient conditions restrained shrinkage of the IPC matrix can lead to the introduction of internal

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