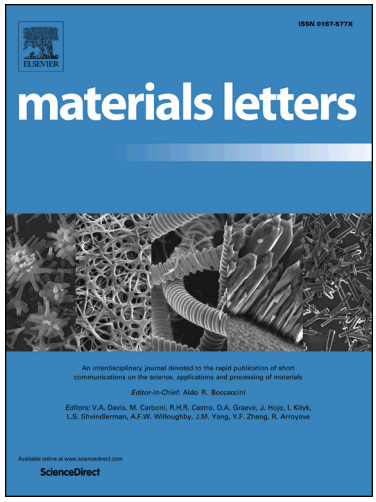

Investigation of setting reaction in magnesium potassium phosphate ceramics with time resolved infrared spectroscopy

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Investigation of setting reaction in magnesium potassium phosphate ceramics with time resolved infrared spectroscopy

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Abstract

Crucial information on reaction products in magnesium potassium phosphate ceramics, essential for material design, is lacking. Setting reaction has been followed with time resolved infrared spectroscopy, supported by 2D correlation spectroscopy. We found evidence of a first amorphous orthophosphate precursor, possibly $MgKPO_4 \cdot H_2O$, forming early, and of a second intermediate amorphous phase, more structurally similar to $MgKPO_4 \cdot 6H_2O$, forming later. Crystallization of $MgKPO_4 \cdot 6H_2O$ occurs from this last phase. Presence of amorphous $Mg(OH)_2$ and magnesium phosphates identified as precipitates from diluted suspensions, was excluded. This complex set of parallel reactions, bears analogies with reaction of zinc phosphate cements and is consistent with recent NMR results.

Keywords: Amorphous materials; Acid-base cements; FTIR.

1. Introduction

Magnesium potassium phosphate ceramics (MKPCs) are a class of acid-base cements for applications, such as concrete repair, biomaterials, radioactive waste encapsulation [1]. The most exploited MKPCs harden at room temperature through the aqueous reaction between magnesium oxide (MgO) and acid phosphate KH_2PO_4 (KDP): $MgO + KH_2PO_4 + 5H_2O = MgKPO_4 \cdot 6H_2O$ (MKP). Using multivariate statistical techniques for the analysis of *in-situ* X-ray diffraction data [2], we have described the time-evolution of an amorphous precursor, which forms at an early stage in consequence of the high supersaturation conditions attained. Investigation of this amorphous component is hampered by the impossibility of synthesizing and/or stabilizing it without altering its structure [3]. Nuclear magnetic resonance (NMR) spectroscopy, pointed to two amorphous orthophosphate hydrates, characterized by two distinct