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Synthesis, hydration and sintering of calcium aluminate nanopowder for advanced applications

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ABSTRACT

Seven batches of calcium aluminate powder containing different ratios of alumina and calcium oxide were chemically prepared by thermal decomposition method. The produced powder was investigated in terms of phase composition and morphology by XRD pattern and scanning electron microscope (SEM). The hydration and sintering of the prepared powder were also studied. Phase composition, microstructure and mechanical properties of the hydrated and sintered bodies were tested. The results revealed that the batches containing high CaO/Al_2O_3 ratios i.e., batches 1, 2 and 3 composed mainly of $CaO.Al_2O_3$ (CA) and $12CaO.7Al_2O_3$ ($C_{12}A_7$) phases while batches containing low CaO/Al_2O_3 ratios i.e., batches 4, 5, 6 and 7 composed of CA and CA_2 ($CaO.2Al_2O_3$) phases. The amount of these phases affected the properties of hydrated as well as the sintered bodies. The hydration of batches No. 1, 2 and 3 achieved higher strength than batches containing low CaO/Al_2O_3 ratios due to the presence of CA and $C_{12}A_7$ as a major component, since they react rapidly with water. The presence of $C_{12}A_7$ phase in the batches containing higher CaO/Al_2O_3 ratios decreased the apparent porosity and consequently increased the mechanical properties of the sintered bodies. The obtained materials can be applied for different advanced applications. The bioactivity of the obtained materials will be studied and published as a second part of this article.

1. Introduction

The term calcium aluminate cements (CAC) covers range of inorganic binders characterized by the presence of monocalcium aluminate CA ($CaO.Al_2O_3$) as their main constituent. Calcium aluminate cement is also called aluminous cement or high alumina cement. The chemical composition of calcium aluminate cement may vary over wide range of Al_2O_3 contents ranging between about 40% and 80%. Unlike Portland cement, it does not contain tricalcium silicate, but may contain limited amounts of dicalcium silicate. Compared with Portland cement, the annual production of calcium aluminate cements is very small. They are also more expensive. Calcium aluminate cements have several unique properties where the performance of Portland cement is insufficient. These properties include: rapid strength development even at low temperature, high temperature resistance refractory performance, and resistance to wide range of chemically aggressive conditions [1,2]. In recent years new applications for calcium aluminates have emerged in optical, bio and structural ceramics. Some amorphous calcium aluminate compositions are photosensitive and hence are potential candidates for optical information storage devices [3–6]. They also have very desirable infrared transmission properties for optical fiber applications [7,8]. Conventionally, calcium aluminate cement is produced by fusing limestone as a source of calcium oxide (CaO) and

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bauxite as a source of aluminium oxide (Al_2O_3) at high temperatures up to 1400 °C. The oxide composition of the blend may vary over a wide range depending on the type of calcium aluminate cement to be produced. The produced material is then ground to fine powder. CA is the main constituent of all types of calcium aluminate cement in addition to undesirable amounts of $CaAl_4O_7$, $Ca_{12}Al_{14}O_{33}$ in addition to the starting reactants. Wet chemical synthesis methods can be applied for the preparation of ceramic powders with special characteristics, such as high sinterability, high surface area, well-defined and controlled chemical compositions and homogeneous distribution of the elements. Alternative low temperature techniques such as sol–gel, polymeric precursor processes and combustion synthesis have been applied for the synthesis of calcium aluminate compounds, instead of solid-state synthesis [9–13]. The present work aims at the preparation and investigation of calcium aluminate nanopowder for advanced applications such as biomedical applications. Studying the hydration, phase composition and sintering of such kind of nanopowder was also the goal of this work.

2. Experimental

Hydrated aluminium nitrate $Al(NO_3)_3.9H_2O$ and hydrated calcium nitrate $Ca(NO_3)_2.4H_2O$ were used as starting materials for the preparation of monocalcium aluminate with different CaO/Al_2O_3 ratios. The batches were designed according to Table 1.

Aluminium nitrate and calcium nitrate masses were weighed and mixed with about 50 ml of water. The solution was heated on a sand bath up to 300 °C. After evaporation of water during continuous heating, brown fumes of nitrogen oxides were detected. The samples were left until all fumes disappeared and the mixture solidified again. A tough white solid was formed at the end of the heating process. The produced solids were calcined at 500 °C and 950 °C for 1 hour to study the effect of calcination temperature on the phase formation. The phase composition of the fired powders were determinate by XRD using Philips model Bruker D_8 Advance, Germany, with Cu Kα target and secondary monochromator, V = 40 kv, A = 40 mA. Ni filter. A SEM, Philips XL.30, Netherlands, was used to study the morphology and particle size of the powder calcined at 500 °C and 950 °C. Also Infrared analysis (IR) was used to identify the products.

Table 1
Molar ratios of CaO/Al_2O_3 for cement batches.

Batch No.	CaO/Al_2O_3 molar ratios
B1	1.05:1
B2	1.10:1
B3	1:1
B4	1:1.05
B5	1:1.10
B6	1:1.15
B7	1:1.20

The calcined powders prepared at 950 °C have been mixed with an adequate amount of water to form cement paste then casted in a cubic steel mould with dimensions of 1 × 1 × 1 cm using a vibrating table at frequency 50 Hz and 4 min vibrating time and kept under 100% relative humid conditions for 30 days. They were then removed from water, dried and tested for cold crushing strength [14].

The produced calcium aluminate powders were pressed at 50 MPa and sintered in an electrical furnace between 1350 °C and 1550 °C. Densification parameters in terms of bulk density and apparent porosity were determined by Archimeds methods. XRD and SEM were also used for

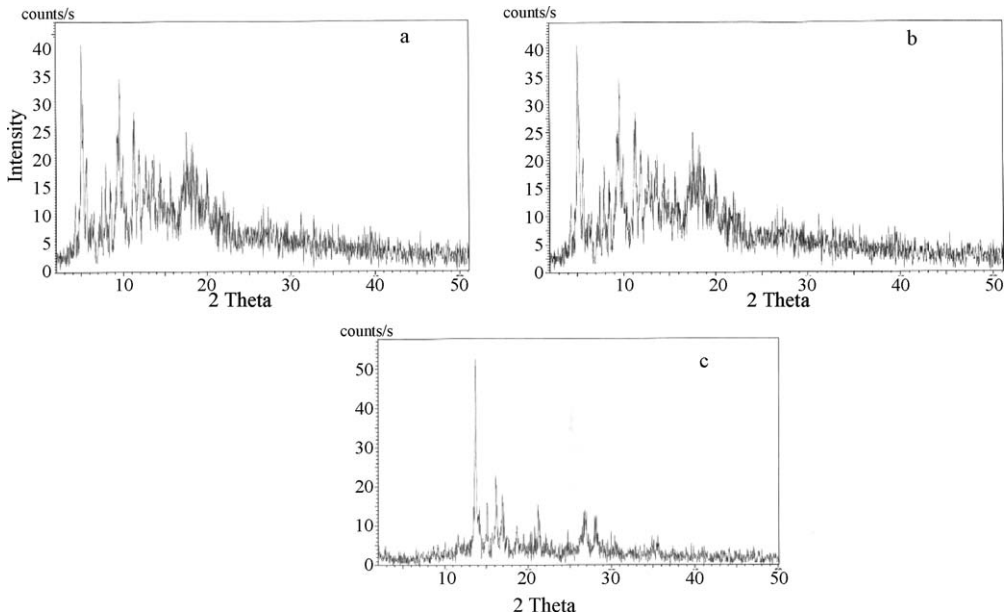

Fig. 1. XRD patterns of as-prepared calcium aluminate powder (a), calcined at 500 °C (b) and 950 °C (c), respectively.