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Influence of water and aluminium powder content on the properties of waste-containing geopolymer foams

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ABSTRACT

The production of low thermal conductivity geopolymers has attracted increasing attention over the past years due to their low cost and green processing technology. In this work, light and porous geopolymers were produced by the proper association of aluminium content and solid-to-liquid ratio. This allowed the production of very low thermal conductivity geopolymers (78.6 mW/m K) exhibiting homogeneous pore size distribution which suggests their use in thermal insulating applications. Moreover this study also reduced the existing knowledge gap concerning the fresh-state characterization of foamed geopolymer slurries. The influence of aluminium powder, curing temperature and solid-to-liquid ratio on the calorimetric response of biomass fly ash-containing geopolymer slurries was evaluated. The calorimetric response of geopolymer slurries shows that the time needed to reach the maximum temperature decreases when the aluminium powder rises, thus shortening the open time before in situ application. It was also found that the geopolymerization rate is governed by the curing temperature.

1. Introduction

Despite the concerns regarding the global energy consumption a distressing increase of 48% is projected from 2012 to 2040 [1]. Within this period the energy-related CO₂ emissions will also rise by 24% [1]. Buildings are the largest energy-consuming sector accounting for 30–40% of the world energy consumption [2], whose energy demand is expected to grow nearly 58% by 2050 [3]. To reverse this unsustainable scenario policy frameworks regarding the energy performance of buildings have been adopted [4]. This sector is considered to be one of the largest sources of cost effective energy saving and CO₂ reduction potential [5]. Indeed, space heating and cooling are the main energy end-uses in buildings and these can be highly reduced by improving the energy efficiency of building envelopes [6]. An attractive option is the incorporation of thermal insulating materials which significantly reduce the energy consumption [7] and increase buildings energy efficiency.

Lightweight geopolymers emerge as an alternative to conventional low thermal conductivity materials due to performance and environmental advantages [8,9]. Commonly foaming agents (e.g. hydrogen peroxide, silica fume, metallic powders) are added to the geopolymer slurry to generate gas bubbles and produce porous geopolymers [10–12]. Low thermal conductivity geopolymers (up to 107 mW/m K)

have been recently reported by the authors, but using hydrogen peroxide as blowing agent [8] generating large size pores and an uneven pore size distribution. However in this work, a better control of pore size and its distribution was observed by the proper association of aluminium (Al) powder (foaming agent) and solid-to-liquid ratio. The latter is crucial to allow the industrial application of this innovative low thermal conductivity material. Moreover this investigation promotes a greater insight concerning the influence of the foaming agent on the fresh-state properties of lightweight geopolymers, reducing the existing knowledge gap. This evaluation is of the utmost importance if the technology is to become widespread.

The geopolymerization reaction is known to be exothermic [13] and their exothermicity can be used to forecast the final geopolymer properties (after hardening). However, the number of studies considering the geopolymers calorimetric characterization is rather limited [14]. Still Ravikumar et al. [15] evaluated the influence of the starting precursors (slag and fly ash), while Puligilla et al. [16] studied the role of calcium on the reaction rate of fly ash-slag geopolymers. Here the calorimetric response of foamed geopolymer slurries was evaluated and the maximum temperature and the time required to reach it were associated with the polymerization extension and setting time, respectively. This study provides valuable insights on the influence of foaming agents in the geopolymers setting time, which is vital for the

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production, transport and storage of geopolymeric mixtures.

In this study, very low thermal conductivity biomass fly ash (FA)-containing geopolymers were produced exhibiting high total porosity (up to 87%) and homogeneous pore size distribution. The effect of the Al content and solid-to-liquid ratio on the geopolymers fresh-state (temperature evolution) and hardened-state properties (compressive strength, thermal conductivity and porosity) was evaluated.

The recycling of biomass FA is still insipient and a common practice for FA management includes the landfill disposal. In this context the incorporation of this unexplored waste in geopolymer production, as proposed here, may reduce/prevent wastes landfilling. In addition, due to its lower activator requirement, compared to benchmark metakaolin (MK), the FA-based geopolymers can be produced at lower cost [17]. Besides the environmental advantage, the reduction in wastes landfilling is also important from an economic view point. In fact, the landfill tax has been increasing over the past years, corresponding to an average of 80 €/ton in the EU27 [18]. Therefore the proposed approach will also lessen the economic burden associated with the wastes disposal.

2. Experimental conditions

2.1. Materials

Two aluminosilicate sources were used: benchmark MK (Argical™ M1200S; Univar®) and biomass FA coming from a co-generation plant [8].

Sodium silicate (water = 62.1 wt%; SiO₂/Na₂O = 3.15; Quimialmel) and 10 M sodium hydroxide (ACS reagent, 97%; Sigma Aldrich) were used as activators. The sodium hydroxide molarity was selected considering previous works [8,19].

A commercial Al powder (Expandit BE 1101, Grimm Metallpulver GmbH) was used as a foaming agent, while a commercial surfactant (Hotapur OSB, Clariant) was employed to stabilize the foam.

2.2. Geopolymers preparation

Aiming to evaluate the influence of the Al content and the solid-to-liquid (S/L) ratio on the slurries calorimetric response and the geopolymer properties after hardening eight compositions were prepared (see Table 1). Previous investigations have shown that rising the water content in the compositions (i.e. using lower S/L ratios) increases the geopolymers' water absorption, while decreasing their apparent density [20]. Accordingly the compositions were specifically designed to ensure the production of highly porous geopolymers, by using low S/L ratios (0.55 and 0.61). The S/L ratios were selected considering both preliminary tests and recent investigations by the authors [8,20].

The solid components (MK and FA) were mixed in a plastic bag, and then added to the previously homogenized alkaline solution (sodium silicate, sodium hydroxide and water) and mixed for 10 min. Afterwards Al powder and surfactant were added to the blend and

mixed for 2 min. The slurry was then poured to plastic moulds, sealed to prevent moisture release and cured for 1 day at 40 °C and 65% relative humidity. Subsequently the specimens were demoulded and left curing at room temperature for the remaining period (28th days). It should be highlighted that the foam formation takes place immediately after adding the Al powder to the geopolymeric slurry, and then continues for a few minutes during curing at 40 °C.

2.3. Materials characterization

The aluminosilicate precursors and the geopolymer samples were studied by X-ray powder diffraction (XRD) to assess their mineralogical composition using a Rigaku Geigerflex D/max-Series instrument (Cu K α radiation, 10–80°, 0.02° 2 θ step-scan and 10 s/step), and phase identification by PANalytical X'Pert HighScore Plus software.

Archimedes' principle was used to measure the specimen's water absorption, whereas their bulk density was determined by measuring samples mass and volume.

The BET specific surface areas of the aluminosilicate precursors were measured by N₂ adsorption using a 5-point BET method (Micromeritics Gemini 2380), while their chemical composition was determined by X-ray fluorescence (Philips X'Pert PRO MPD spectrometer). Particle size distribution was evaluated by laser diffraction (Coulter LS230 analyzer).

The true density of the un-foamed geopolymers (3.27 g/cm³) was determined by the helium pycnometer technique (Multipycnometer, Quantachrome). Then geopolymers' total porosity was calculated [8].

The geopolymers' compressive strength (d = 36 mm; length = 60 mm) was measured at the 28th day using a Universal Testing Machine (Shimadzu model AG-25 TA; 0.5 mm/min). Thermal conductivity was measured at room temperature on cubic samples (64,000 mm³) by using a heat flow meter apparatus according to ASTM C518-04. In these tests (compressive strength and thermal conductivity) three specimens of each formulation were tested.

The specimens' microstructure was evaluated by using scanning electron microscopy (SEM - Hitachi S4100 equipped with EDS - Rontec), whereas their morphology was investigated using optical analysis (Leica EZ4HD microscope).

The calorimetric response of the geopolymer slurries upon curing was measured by using a quasi-adiabatic calorimeter, which was placed inside a climatic chamber setting the temperature to 25 °C and 40 °C.

3. Results and discussion

3.1. Raw materials characterization

The XRD patterns of the two aluminosilicate precursors are shown in Fig. 1. As depicted, benchmark MK is considerably more amorphous than FA. Nevertheless, some crystalline phases, namely quartz, anatase and muscovite, were also observed for this commercial aluminosilicate. The biomass FA contains large amounts of crystalline phases, such as

Table 1
Geopolymer preparation: mixture composition and solid-to-liquid ratio.

Sample name	Mixture proportion (g)							S/L	Study objectives
	FA	MK	Sodium silicate	NaOH	H ₂ O	Al	Surfactant		
F1	12.61	25.22	33.63	22.07	6.46	0.02	0.05	0.61	Influence of water and Al content
F2						0.04			
F3						0.06			
F4						0.08			
F5	12.61	25.22	33.63	22.07	12.93	0.02	0.05	0.55	
F6						0.04			
F7						0.06			
F8						0.08			

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