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A comparison between different foaming methods for the synthesis of light weight geopolymers

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Abstract

Foaming to reduce the density of geopolymeric materials is increasingly being reported in the literature as it has been shown to be effective in improving their insulating properties. However, there is no consistency in foaming methods and as such this study was performed to compare methods in order to better understand their effect on the properties of geopolymers. A surfactant and two chemical foaming agents (hydrogen peroxide and aluminium powder) were added to a fly ash based geopolymer matrix. Surfactant was also combined with each of the chemical foaming agents in order to stabilise the foam in the geopolymer matrix and to reduce coarse pores. The physical, mechanical and microstructural properties of the low density geopolymers are presented and the effects of the foaming agents' characteristics on the hardened product is discussed, as well as the relative merits of the different procedures to synthesise the foamed geopolymer. It was found that homogeneous microstructures with small pores can be obtained by adding surfactant and hydrogen peroxide. The combination of hydrogen peroxide (0.1 wt%) and surfactant (1.0 wt\%) produced geopolymer foams with density and compressive strength values of 0.94 g/cm³ and 4.6 MPa, respectively. $© 2014 Elsevier Ltd and Techna Group S.r.l. All rights reserved.$

Keywords: B. Porosity; Foaming agent; Surfactant; Geopolymers

1. Introduction

Ordinary Portland cement, the most common cementitious building material, is responsible for a significant amount of global $CO₂$ emissions due to the decomposition of limestone and the combustion of fossil fuels during production. Geopolymer and other similar binders such as alkali activated materials (AAMs) have attracted a lot of attention $[1-3]$ $[1-3]$ $[1-3]$ as suitable alternatives due to their significantly lower emissions during production [\[4\]](#page--1-0). An additional benefit of the use of geopolymer compared with OPC concrete is based on the possibility of using high-volume industrial waste in highperformance concretes, with a significant reduction in $CO₂$ emissions [\[5\].](#page--1-0) Geopolymer is a class of three-dimensional

alumino-silicate materials [\[6\].](#page--1-0) Geopolymers are based on aluminosilicate units such as sialate $[-Si-O-AI-O]$, sialate $\begin{bmatrix} -\text{Si}-\text{O}-\text{Al}-\text{O}-\text{Si}-\text{O} \end{bmatrix}$ or sialate disiloxo $[-Si-O-Al-O-Si-O-Si-O]$ [\[6\].](#page--1-0) The polymerised materials contain tetrahedrally coordinated Al and Si, with charge balance of the Al tetrahedra being achieved by the presence of Na⁺ or K⁺ ions [\[7\].](#page--1-0)

The weight of concrete represents a large proportion of dead load on a structure; the use of geopolymers of lower density is beneficial in terms of reduced structural load-bearing with further benefits of acoustic and thermal insulation [\[8](#page--1-0)–[10\]](#page--1-0). However, mechanical strength relates strongly with density and low density geopolymers can exhibit unacceptably low strength [\[11\]](#page--1-0). Sufficient mechanical strengths can be achieved with the controlled addition of foaming agents in order to achieve an optimum density and pore structure.

Different foaming agents can be used to synthesise low density geopolymers. Surfactants are liquid admixtures that can be used to produce light weight materials by entraining air

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during mixing. Another option is to use chemical products mixed into the geopolymer slurry which react with the alkali to generate gas which produces a foamed microstructure in the hardened material [\[12\]](#page--1-0). The addition of metals, such as zinc or aluminium, to the geopolymer paste generates hydrogen gas [\[13\]](#page--1-0). Metallic aluminium powder is commonly used and is very reactive in alkaline environments. Aluminate Al_2O^- and $H₂$ gas are liberated according to Eq. (1) [\[12\]](#page--1-0):

$$
8\text{Al} + 2\text{OH}^- + 2\text{H}_2\text{O} \rightarrow 4\text{Al}_2\text{O}^- + 3\text{H}_2 \tag{1}
$$

The reaction of metallic silicon present as an impurity in silicon carbides or silica fume also generates hydrogen gas when exposed to alkali [\[14,15\].](#page--1-0) Another class of chemical foaming agents is peroxides such as hydrogen peroxide and organic peroxides which react to evolve oxygen gas [\[13\]](#page--1-0). Bubbles of O_2 are trapped within the paste, expanding and increasing the volume. Hydrogen peroxide is thermodynamically unstable and can be easily decomposed to water and oxygen gas according to Eqs. (2) and (3) [\[16\]:](#page--1-0)

$$
H_2O_2 + OH^- \rightarrow HO_2^- + H_2O;
$$
 (2)

$$
HO_2^- + H_2O_2 \to H_2O + O_2 + OH^-. \tag{3}
$$

The synthesis of low density geopolymers using hydrogen peroxide is influenced by the optimisation of the kinetics of peroxide decomposition with production of oxygen and the increase in viscosity of the geopolymer paste [\[17\]](#page--1-0).

The synthesis of low density geopolymers represents a challenge and an optimised procedure should be investigated for each different foaming technique: small pore size and uniform pore distribution needs to be achieved and pore collapse should be avoided.

This paper presents a study on different foaming techniques to synthesise low density geopolymers. Physical and mechanical properties as well as microstructural analysis of geopolymer samples are reported. Varying concentrations of three foaming agents (surfactant, aluminium powder and hydrogen peroxide) were investigated in order to assess their influence on the final properties of low density geopolymers. Samples made with a combination of surfactant and chemical foaming agents were also studied in order to achieve a more homogeneous distribution of small pores.

2. Materials and methods

2.1. Materials

Fly ash was sourced from the Eraring power station in New South Wales, Australia. Sodium aluminate solution was used as the alkali activator. Solutions were prepared by dissolving sodium hydroxide pellets from Univar Pty Ltd. and sodium aluminate powder supplied by Sigma in deionised water. The solution was allowed to dissolve overnight at 70° C and then used for synthesising geopolymers after 24 h. Aluminium powder with a particle size of 50 μm and a purity of 99.5%

(product code AL006020, Goodfellow, U.K.) and hydrogen peroxide solution with 30% w/w supplied by Rowe Scientific were used as chemical foaming agents. Sika \mathbb{B} Lightcrete 02 was used as the surfactant for foaming and is reported by the manufacturer to contain 40 wt% solution of fatty acid, amide and sodium salt of $C_{14}-C_{16}$ sulphonic acid in water.

2.2. Geopolymer synthesis

Geopolymers were synthesised with targeted compositional ratios of Si: $Al = 2.0$, Na: $Al = 1.1$ and a water content of 21 wt %. Control samples were made by mixing the fly ash with the activating solution for 10 min.

Different procedures were used to add the foaming agents: the chemical foaming agents were added to the geopolymer slurry after the 10 min mixing period and mixed for a further 20 s at high RPM. Aluminium powder was added in concentration from 0.01 wt% to 0.05 wt% and the concentration of hydrogen peroxide was from 0.1 wt% to 0.4 wt%.

The surfactant (from 1.0 wt% to 5.0 wt%) was added to the geopolymer slurry after the initial 10 min mixing and mixed for a further 2 min with a whisk attachment at high RPM.

The concentration ranges are different for each foaming agent. The maximum concentration used was fixed at a level below the starting point of pore collapse phenomena that was determined for the geopolymer matrix used in this study.

Geopolymer mixed with surfactant and chemical foaming agents were synthesised with 1.0 wt% of surfactant that was added after 5 min of mixing and the chemical foaming agents added after 10 min. Immediately after mixing, samples were poured into cylindrical moulds (50 mm diameter, 100 mm height), sealed and cured at 70° C for 24 h.

2.3. Characterizations

The density of the samples was measured by dividing the dry mass by the volume. Cylindrical samples (50 mm diameter, 100 mm high) were used for density measurements. All reported results are an average of 4 different measurements.

Water absorption was calculated as the per cent increase in weight of the specimens after exposure to water at ambient temperature until their complete saturation and/or sample weight does not vary more than 0.1%.

Pore size distribution measurements were carried out on all specimens by a mercury intrusion porosimeter (MIP, Carlo Erba 2000) equipped with a macropore unit (Model 120, Fison Instruments). Samples for porosimetry were cut by diamond saw to approximately 1 cm^3 , dried under vacuum and kept under a P_2O_5 atmosphere in a vacuum dry box until testing. The MIP measurements were carried out using a contact angle of 141.3° , a Hg surface tension of 480 dyne/cm and a pressure ranging from 0 to 200 MPa. Applying the Washburn [\[18\]](#page--1-0) equation to calculate the pore dimension intruded by mercury at each pressure step, the pore radius ranging between 0.0035 μm and 33 μm can be detected. The suitability of MIP for pore size and pore size distribution is frequently debated [\[19](#page--1-0)–[21\]](#page--1-0), however its use in cement based materials is

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