NOVEL GEPOLYMER FOAMS BY GEL-CASTING

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INTRODUCTION

Ceramic foams are known to exhibit high permeability, high specific surface area, good insulating characteristics, high refractoriness, chemical resistance, well-developed surface pores and three dimensional network pore structure. Those characteristics enable ceramic foams to be widely used in industrial fields as filters for contaminated hot gas purification, or solid/liquid separation process, catalyst support and thermal insulators [1]. There are several ways to produce highly porous ceramics, and the main processing routes are: the replica technique, the sacrificial template method, and the direct-foaming technique [2]. Belonging to the latter category, the gelcasting process enables to achieve foamed ceramic with porosity levels up to 90% from ceramic slurries. This process consists in vigorously stirring a ceramic suspension containing water-soluble monomers and a surfactant [3]. Wet foams are thermodynamically unstable systems in which processes like drainage of the liquid phase and gas bubble coarsening lead to foam degradation and final destruction. Diffusion of gas occurs between bubbles of different sizes and consequently different concentrations of gas due to the difference in Laplace pressure between them (Ostwald ripening) leading of foam degradation governed by the reduction of the Gibbs free energy of the system [4]. To avoid this, surfactants are used as surface-active agent for the stabilization of wet foams. These long-chain amphiphilic molecules adsorb at the gas bubble surfaces with their hydrophilic tail in contact with the aqueous phase. The foaming ability of a surfactant is related to its effectiveness to lower the interfacial energy or the surface tension at the gas–liquid interface. Surfactants are classified according to the hydrophilic group as anionic, cationic, non-ionic, and amphoteric. With respect to a slurry based on geopolymer precursors, which contains several ions in solution (K^+, Al^{3+}, Fe^{3+}, SiO_4^{2-}), a non-ionic surfactant has a more pronounced effect since it possesses hydrophilic groups with no electric charges. After wet foam formation (typically by stirring at high velocity), the suspension is rapidly gelled by means of the polymerization of the monomers introduced as binders, giving rigid a ceramic foam that is then sintered at high temperature [4,5]. In the case of the geopolymers, the reaction of geopolymerization itself, rather than the use of organic monomers, gels the structure retaining the high porosity produced in the wet state. No further heat treatment is required in the case of geopolymer, if the temperature of application is not too high. The aim of this work is to investigate the applicability of the gelcasting process to geopolymer with the specific aim of producing highly porous components possessing open, interconnected porosity.

MATERIALS AND METHODS

Experiments were carried out using as precursors metakaolin obtained from the calcination of *Minasolo* kaolin at 750°C for 6 hours in a muffle, fly ash class F, and as alkaline activators potassium hydroxide KOH pellets (85% of purity) and potassium silicate (Si/K = 1.66, density 1.39 g/l, viscosity 800 cP). Considering the high content of iron oxide (Fe_2O_3) present in the fly ash used (10.2 wt.%), 30 wt% of fly ash was used as a maximum addition wit respect to metakaolin, because the iron element tends to replace the aluminium in the process of connecting the tetrahedral $SiO_4^{2-}$and it usually affects detrimentally the mechanical strength [6]. In order to decrease the viscosity of the suspension, polyacrylic acid (Dolapix CE-64) was used as deflocculant and Tween 80 and Triton X-100 were used as non-ionic surfactants. Slurries with solid content ranging from 61 up to 71 wt% were prepared. Figure 1 show in a schematic diagram, the process used to produce porous geopolymer components.

All samples were prepared using either Tween 80 or Triton X-100 surfactants, in order to evaluate the effect of the specific type of surfactant on the characteristics of the geopolymer foams obtained maintaining similar all the other processing parameters (stirring velocity, solid content, stirring time, surfactant content).

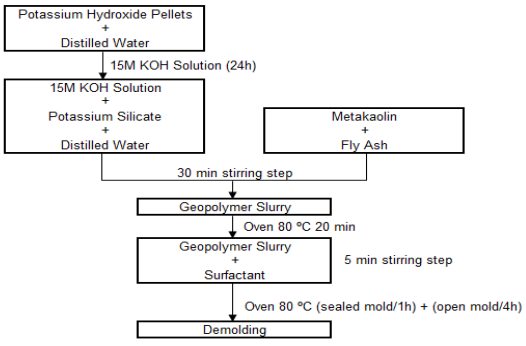

Figure 1. Schematic diagram for the production of geopolymer foams production using an "adapted" gelcasting process

RESULTS AND DISCUSSION

Characterization

Density measurement

The bulk density ρ of a geopolymer foam is given by the mass of a parallelepiped of foam divided by its apparent volume. The theoretical density ρ_0, of the pore-free solid material, was measured with an Accupyc 1330 (Micrometrics) helium pycnometer with a 3.5 cm^3 cell. So, an estimative of the pore volume fraction X_P could be determined using the relation $X_P = 100 * (1-\rho/\rho_0)$ [7].

Microstructure analysis

The morphology of the samples was studied using a Nikon Coolpix 990 coupled in a Wild Heerbrugg Optical Microscope Type 376788. The pore size distribution of a sample was evaluated from pictures using the Axio Vision 4.8.2 LE image software. Measurement values obtained by image analysis were converted to 3D values in order to determine the effective cell-size through the stereological equation $D_{sphere} = D_{circle}/0.785$ [8]. As a starting parameter, the effect of the solid content in the slurry on porosity was considered so that, by setting this parameter to a specific value, the influence of other processing variables such as surfactant amount, mixing speed and time on the total porosity and pore size and distribution, could be investigated.

Effect of solid content in the slurry on total porosity

To evaluate the effect of solid content in the slurry on the total porosity, a surfactant content of 2 wt% and a mixing speed of 1500 rpm were fixed. Figure 2 shows that, as expected, the increase in solid content led to a reduction in porosity of geopolymer foamed structures obtained by this processing route

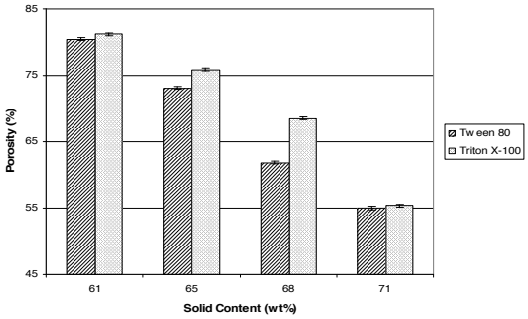

Figure 2. Effect of solid content in the slurry on total porosity (a surfactant content = 2 wt%; mixing speed = 1500 rpm).

Effect of mixing speed and surfactant content on total porosity

With the aim of evaluate the effect the rotation speed and of the surfactant content on total porosity, two amounts of surfactant were used. Figure 3.A (left) is for samples containing 2 wt% of surfactant (Tween 80) and Figure 3.B (right) shows how porosity changed with the addition of 4 wt% of surfactant. For both surfactant contents, the total porosity produced was measured for three different speed rotation, as 800, 1500 and 2000 rpm. After these preliminary experiments, all studies were conducted on samples with 68 wt% solid content. Fig. 3 also shows the morphology of samples produced using either 2 (left) or 4 (right) wt% of Tween 80 surfactant (mixing speed = 1500 rpm). Highly porous foams possessing fully interconnected pores were produced by this method.

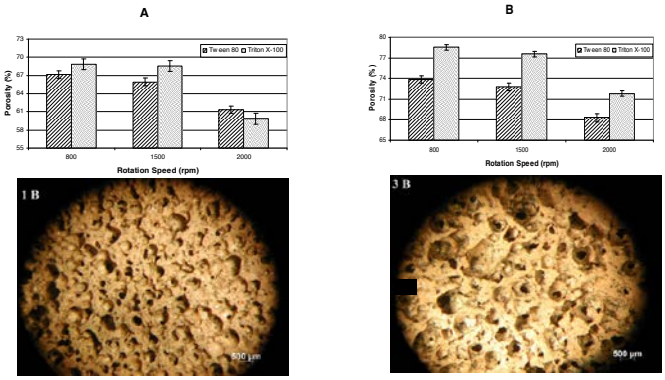

Figure 3. Effect on the total porosity of the mixing speed and the surfactant content in the slurry (68 wt% solid content): with 2 wt% Tween 80 surfactant (left/A) and with 4 wt% Tween 80 surfactant (right/B). Morphology of the foams (1B, 2 wt%; 3B = 4 wt%)

CONCLUSIONS AND PERSPECTIVES

The results of this initial research on the adaptation of the gel-casting process in order to obtaining highly porous geopolymer foams showed its technical feasibility. It is also a clear need for further study, that predicts the choice of additives to be used (type and properties, adding conditions, etc.) in order to optimize the process in order to tailor the characteristics of the desired component. More characterization of the foam properties (mechanical resistance, permeability, microstructure, etc.) will also be undertaken. On the basis of this research, we will then evaluate several possible applications of the foams obtained by this new processing route and adjust the process conditions for the development of porous ceramics/geopolymer structures suited for different applications.

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