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## Correlation of microstructural and mechanical properties of geopolymers produced from fly ash and slag at room temperature

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### HIGHLIGHTS

- High strength geopolymers were developed for curing at room temperature.
- Substitution of fly ash with GGBS by more than 50% leads to increase in flexural strength only.
- The freeze-drying is a desirable method for pre-conditioning of geopolymers for microstructural analysis.
- Good correlation is observed between the microstructure and strength development of geopolymers.

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### ABSTRACT

High strength fly ash (FA)-based geopolymer pastes containing slag were characterized by means of X-ray diffraction (XRD), thermogravimetry (TGA), and environmental scanning electron microscopy (ESEM). Various ratios of FA were replaced with ground granulated blast furnace slag (GGBS) to enhance the early strength development of the geopolymers cured at room temperature. Compressive and flexural strength tests were conducted to determine the mechanical properties, and mercury intrusion porosimetry (MIP) measurements were carried out to investigate the microstructure of the hardened geopolymers. A new pre-conditioning drying method for MIP investigations was applied and its effects on the GPs' microstructure were evaluated. The comparison of results showed that freeze-drying by using liquid nitrogen could be a great alternative to the conventional drying methods for geopolymers. The compressive and flexural strengths of geopolymers containing slag reached up to around 100 MPa and 10 MPa, respectively. The decreasing porosity of the geopolymers correlated well with the obtained increasing values of the compressive strength. However, neither of them noticeably improved when over 50% of FA was replaced by GGBS, while a further increase in the flexural strength was observed.

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### 1. Introduction

Geopolymers (GPs) are promising materials for high-temperature applications, fire- and heat-resistant coatings, and are also an alternative binder to ordinary Portland cement [1–4]. The terms “geopolymer” and “alkali-activated” material have been widely discussed by many researchers. Whether “geopolymer” is the subset of “alkali-activated” or it is a separated material class, has been the subject of a wide and yet still ongoing discussions

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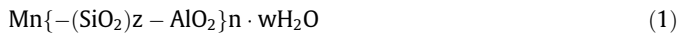
[5]. GPs are usually synthesized by mixing an aluminosilicate reactive material such as FA with highly alkaline solutions e.g. potassium hydroxide (KOH), sodium hydroxide (NaOH), potassium silicate or sodium silicate [6,7].

The network formation in GPs is based on chemical reactions between an alkaline hydroxide or silicate solution and aluminosilicate solid materials, transferring amorphous to three dimensional polymeric structures consisting of Si–O–Al bonds [1,2,7–10]. This process offers the possibility to transfer abundant alumino-silicate by-products, such as FA into a composite with high mechanical strength (e.g. 60 MPa or over) as well as great acid and fire resistance [6,11].

The process itself comprises the following steps: (i) dissolution of the aluminosilicate solids in the highly alkaline aqueous

solution, (ii) formation of oligomeric precursors consisting of Si–O–Si/Si–O–Al, (iii) polycondensation of the oligomers to build up a three-dimensional aluminosilicate network and, finally, (iv) hardening by cross-linking of the whole system into a polymeric structure through bonding of the undissolved solid particles into an established network [12–17].

The empirical formula of the poly-sialates is given by Eq. (1):



where M is a cation such as  $\text{K}^+$ ,  $\text{Na}^+$  or  $\text{Ca}^{2+}$ ; n and z are numbers of repeating units. Other cations such as  $\text{Li}^+$ ,  $\text{Ba}^{2+}$ ,  $\text{NH}_4^+$  and  $\text{H}_3\text{O}^+$  may also be present in the built network [11,18–21].

In civil engineering, the main reason for using geopolymers is to produce non-cementitious composites in order to reduce the carbon dioxide ( $\text{CO}_2$ ) emissions associated with the clinker production process. It has recently been revealed that the concentration and the type of the alkaline activator not only affect the strength of geopolymer material [1,22], but also significantly influence the environmental impacts [23–25]. Therefore, choosing a waste material such as FA with appropriate chemical or mineral characteristics is an important factor in order to reduce the required amounts of alkali activator, and as a result, the  $\text{CO}_2$  emissions. For instance, FA/GGBS based geopolymer concrete requires less sodium silicate solution compared to metakaolin to be activated [23]. Habert et al. [23] in 2011 suggested that the composition of alkaline solutions, namely, sodium silicate, and the Si-to-Al ratio of by-products are the key factors for controlling the environmental impacts of geopolymers. Pasupathy et al. [26] demonstrated the importance of the performance based strategy to develop standard specifications for geopolymer concrete by examining how the additional  $\text{Na}_2\text{SiO}_3$  activator could enhance the carbonation ratio. It was observed that two geopolymer concretes, with a similar mix design and compressive strength, could have different carbonation behavior.

Some investigations have been undertaken on the development of high strength GPs cured at room temperature. Nugteren et al. [27] presented a high compressive strength (100 MPa) geopolymer by reduction of the size fraction of the FA, but at the cost of worse workability. Another commonly used method of improving the mechanical properties of GPs, particularly for those with FA due to their slow strength development, is using elevated curing temperatures (over 40 °C) [3]. However, the consideration of energy consumed during such curing process reverts the concerning issue in terms of  $\text{CO}_2$  emissions [24]. In order to eliminate the need for elevated curing temperatures [28], FA was replaced by GGBS in this investigation.

It is considered that the substitution of FA with GGBS may change the GP microstructure such as porosity [29]. For cement-based materials a strong correlation between the densification of microstructure and mechanical or durability properties has been widely reported [30–34]. However, such relationships for GPs are less reported. A weak relationship between compressive strength and the porosity of geopolymers has been reported [19,35]. However, there is a concern that such findings may be compromised by an inappropriate pre-conditioning drying method applied prior to the porosity measurement tests. For instance, the conventional oven drying method may change the microstructure of GPs, in particular that of cured at the room temperature [36]. This arises the necessity to examine the effects of the pre-conditioning drying methods on the microstructure of GPs.

This paper is aimed at correlating the microstructural development and mechanical performance of high strength FA-based GP pastes containing different amounts of slag cured at room temperature. One of the key steps to achieve this goal is to adopt a novel pre-conditioning method, i.e. freeze drying using liquid nitrogen, to stop reactions and to remove free water from the samples, while

preserving the GPs' microstructure. The study started with a full characterization of the raw materials with regard to their physical and mineralogical properties. It was then followed by a series of microstructural analysis and mechanical property tests. Results are presented and discussed to display the correlation between the microstructural and mechanical development of GPs.

## 2. Experimental program

### 2.1. Materials

Steamant<sup>®</sup> H-4 FA obtained from Steag Power Minerals GmbH, Germany, and the ground granulated blast-furnace slag (GGBS) from Opterra GmbH, Germany were used in this investigation. The alkaline activators were sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) composed of (27.25%  $\text{SiO}_2$ , 8.15%  $\text{Na}_2\text{O}$  and 64.60%  $\text{H}_2\text{O}$  by mass) supplied by Carl Jäger Tonindustriebedarf GmbH, Germany, and sodium hydroxide (NaOH) pellets (99 wt%) supplied by Grüssing GmbH Analytika, Germany. Silica modulus (Ms) i.e.  $\text{SiO}_2/\text{Na}_2\text{O}$  of the sodium silicate solution is 3.34. An appropriate amount of sodium hydroxide pellet was mixed 24 h prior to GPs production with distilled water to achieve the desired NaOH molar ratio of 10 M.

### 2.2. Sample preparation

Firstly, FA and GGBS were dry mixed by hand for 5 min. using a spoon. Secondly, the slurry containing Na-silicate and NaOH (10 M) was added and mixed for another 3 min. Afterwards the fresh samples were cast in 10 mm × 10 mm × 60 mm molds and cured in plastic bags at room temperature (23 °C) until the testing date. The mix proportions are listed in Table 1. FA was replaced with GGBS at 30, 40, 50, 60, 70 and 100% by mass ratios, which caused an increase in Si-to-Al ratio. For the majority of the mixtures (up to 60% of GGBS) the Si-to- $\text{Na}_2\text{O}$  ratio was kept constant. However, for the mixtures with 70% and 100% slag the low workability required a higher addition of the liquid component.

### 2.3. Characterization and test methods

Thermogravimetric Analysis (TGA), X-Ray Diffraction (XRD), Environmental Scanning Electron Microscope-Energy Dispersive X-Ray Spectroscopy (ESEM-EDX) were applied to investigate the raw materials as well as the synthesized GPs. Additionally, mechanical testing and mercury intrusion porosity (MIP) measurements were performed on hardened GPs.

Most microstructure inspection requires a sample pre-conditioning by drying, which may change both the morphology and mechanical properties of geopolymers drastically [29]. In this investigation, a freeze-drying method was applied. Samples were exposed to liquid nitrogen to remove free water and consequently to stop the reaction. Subsequently, samples were stored in a vacuum desiccator until further analysis. To evaluate the effect of nitrogen drying on the GPs' microstructure, the results obtained for the freeze-dried samples were compared to those dried by acetone and to un-dried samples. The testing procedure was adapted from a study performed by Ismail et al. [36].

#### 2.3.1. Laser diffraction particle size analysis

The particle size distribution of the raw materials was measured by using a LS 13-320 from Beckman Coulter. 0.5 g of each material was mixed gently in a magnetic mixer in isopropanol before the test.

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