



# New synthesis method for the production of coal fly ash-based foamed geopolymers



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## HIGHLIGHTS

- Foamed geopolymer was synthesised using coal fly ash, NaOH, and NaOCl at 90 °C.
- NaOCl foaming agent did not cause early foaming of the geopolymer precursor slurry.
- Varying the NaOH/FA ratio controls geopolymerisation and foaming.
- Geopolymers were obtained with porosities in the range 35–62%.

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## ABSTRACT

Foamed geopolymers were synthesised using a South African Class F coal fly ash (FA), sodium hydroxide (NaOH), and the novel foaming agent sodium hypochlorite (NaOCl) at slightly elevated temperatures (90 °C). The synthetic method has the advantage of maintaining control over foaming; the mixed and prepared cementitious slurry containing NaOCl is stable for at least 1 h at room temperature, thus, avoiding premature foaming of the slurry before moulding. In this study, the effect of NaOH/FA on the formation of foamed geopolymers was investigated at predetermined NaOCl/FA ratios (by weight). The NaOH/FA ratio was changed over the range of 0.16–0.24 at a constant NaOCl/FA composition of 0.50. Increasing NaOH/FA improved geopolymerisation up to a ratio of 0.20 and porosity up to a ratio of 0.22, after which geopolymerisation and porosity declined. These results were substantiated through XRD, SEM, FTIR, Raman analyses, mercury intrusion porosimetry, nitrogen adsorption/desorption, and helium density measurements. The formulations developed in this study may be used to fabricate materials suitable for application in construction as light weight fireproof insulation, internal walls or ceiling tiles.

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## 1. Introduction

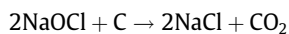
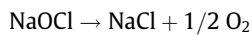
Coal-burning electric power plants generate large amounts of fly ash (FA) as a by-product of the coal combustion process. In South Africa, approximately 36.7 million tons of coal FA was produced in 2009, of which only around 5.7% was recycled, with the remaining FA is deposited in ash dumps [1]. The disposal of FA could have a negative impact on the receiving environment if the pH of the solution in contact with the ashes is not appropriately monitored [2]. Thus, significant research efforts have been expended to find novel applications for FA use other than only as an additive in cement and concrete production or in the synthesis of zeolites [3,4].

Geopolymers are a new class of materials that can be synthesised using metakaolin, natural minerals, and waste materials such as FA, slag, and red mud [5–7]. Among these feedstocks, the use of FA ash is desirable as it involves the conversion of a copious waste material into a useful product. In general, geopolymers are three-dimensional, amorphous-to-semi-crystalline aluminosilicate materials [8]. They are composed of successive SiO<sub>4</sub> and AlO<sub>4</sub> tetrahedra connected through an oxygen-bridged bonding framework, in which positive ions (Na<sup>+</sup>, K<sup>+</sup>, Li<sup>+</sup>, or Ca<sup>2+</sup>) balance the negative charge of Al<sup>3+</sup> in IV-fold coordination [9]. The dissolution of aluminosilicate materials from the surface of FA particles in a highly alkaline environment via hydrolysis is generally accepted as the first step in geopolymerisation [5], followed by oligomerisation, polymerisation, and finally, condensation of dissolved species. The newly formed geopolymeric gel binds unreacted or half-reacted FA particles together [10]. The (hardened) geopolymers prepared in this way have apparent densities of >1 g/cm<sup>3</sup> [11].

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The foamed geopolymers are lightweight materials (bulk density <1 g/cm<sup>3</sup>). They can be synthesised at high temperature or at low temperature processing conditions, the latter of which is applied at ambient or slightly-elevated temperatures and thus more cost effective and suitable for larger-scale production such as in the construction industry [12]. The low temperature processing comprises of mechanical pre-foaming, chemical foaming (hydrogen peroxide and aluminium powder as foaming agents) and silica fume in situ foaming techniques [12–15]. In an alkaline environment, hydrogen peroxide decomposes into water and oxygen, and aluminium and silicon elements in the fumed silica initiate the release of hydrogen gas, leading to a porous structure. Foaming is rapid when using the above-mentioned foaming agents, but making it difficult, if not impossible, to control reactions in larger-scale production. Conversely, the foaming agent perborate requires heat to begin foaming, allowing the foaming process to be controlled via the agency of heat [16]. In the present study, FA-based foamed geopolymers were synthesised using sodium hypochlorite (NaOCl) as the foaming agent, allowing the control of foaming via heat in this case as well [17]. The gases released during this process, although not investigated in this study, are presumably both oxygen produced by the dissociation of NaOCl and, to some extent, carbon dioxide produced by the reaction of NaOCl with the carbon present in FA according to the following reactions [18]



The FA-based foamed geopolymers prepared in this way can be used in the construction industry to make light weight fireproof insulation, internal walls or ceiling tiles [17].

The foaming agent/FA and alkali activator/FA compositions are important parameters in the synthesis of foamed geopolymers since they control both the extent of foaming and geopolymerisation. The aim of this study was to investigate the effect of NaOH/FA on the microstructure and textural properties of foamed geopolymers fabricated using a predetermined amount of NaOCl as the foaming agent at a constant Si/Al composition.

## 2. Materials and methods

### 2.1. Materials

The coal fly ash (DuraPozz) was supplied by Ash Resources (Pty Ltd) from the Lethabo power station in South Africa. The FA had a mean particle size of 25 µm; typically, 90% of the product's particles passed through a 45 µm sieve according to the supplier. NaOCl (Kimex, South Africa, 12%, density 1.173 g/L) and sodium hydroxide (Merck, 98%) were the other reagents used during experiments.

### 2.2. Synthesis of foamed geopolymers

A series of samples were prepared in two sets. In the first set, NaOCl/FA was varied (0.40, 0.50 and 0.60) while keeping NaOH/FA constant (0.20) (by weight). The results of nitrogen adsorption and desorption tests done on these samples revealed that the sample with NaOCl/FA = 0.50 had the highest specific and mesoporous surface area compared to the other samples. Thus, a second set of experiments were

**Table 1**  
Initial compositional ratios of the foamed geopolymer samples (by weight).

Sample no	NaOCl/FA = 0.50	
	NaOH/FA	H <sub>2</sub> O/FA
1	0.16	0.44
2	0.18	0.44
3	0.20	0.44
4	0.22	0.44
5	0.24	0.44

done by varying NaOH/FA (from 0.16 to 0.24 with 0.02 increments) while keeping NaOCl/FA constant (0.50) (wt) (Table 1). Those samples were named according to only their initial NaOH/FA ratios in the later sections of the paper.

During synthesis, firstly, the NaOCl solution was added to FA and stirred for 10 min using a flat blade impeller to obtain a homogeneous slurry. Thereafter the temperature of the slurry was kept below 40 °C whilst adding small portions of NaOH pellets until all the predetermined amount of NaOH dissolved. The cementitious slurry was stirred for an additional half hour. 150 g portions of the main slurry, having entrapped gas, such as air and probably carbon dioxide, were poured into individual polypropylene containers with lids, each container was sealed with a plastic bag [19]. The purpose of sealing was to ensure minimal loss of water or moisture during the subsequent hydrothermal treatment. The sealing also limited the absorption of atmospheric water vapour and carbon dioxide by the NaOH solution in the slurry. Temperature was gradually increased during four days of hydrothermal treatment to prevent instant expansion of the geopolymer paste and to maximize dissolution and polycondensation reactions. The sealed samples were placed in an oven and aged at 30 °C for a day. The aged samples were heated for three days: the oven temperature was held at 60 °C during the first day and 90 °C during the second and third day (Fig. 1). After completion of hydrothermal treatment, each foamed geopolymer sample was unwrapped, cured further for a day at 30 °C and afterwards kept in its sealed container. Fig. 2 shows the FA used in the synthesis and the foamed geopolymer with NaOH/FA = 0.20 produced from this FA.

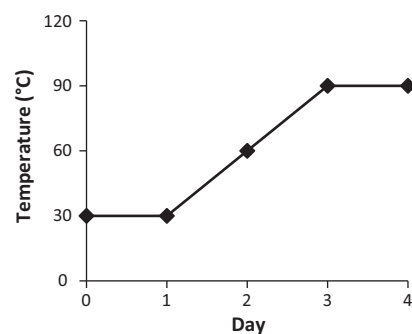
### 2.3. Characterisation

Elemental composition of FA was determined by XRF spectroscopy on a PANalyticalAxios Wavelength Dispersive spectrometer. The loss on ignition (LOI) test was done at 950 °C. The XRD patterns of the fly ash and the powder geopolymers samples were recorded on a Bruker powder diffractometer (D8 Advance) equipped with a theta-theta goniometer setting which includes a Cu target X-ray tube (CuKα1 line at λ = 1.5406 Å) and PSD Vantec-1 detector. Data evaluation was done using the EVA software from Bruker based on the International Centre for Diffraction Data (ICDD) PDF database 1998. Small samples of the geopolymer blocks were carbon coated and their surfaces investigated by SEM/EDS analysis. Micrographs were recorded using a Zeiss Auriga field-emission scanning electron microscope. The elemental analysis was done using an Oxford Aztec EDS attached to the SEM. Attenuated total reflectance Fourier transform infrared (ATR-FTIR) absorption spectra were recorded in the range 4000–450 cm<sup>-1</sup> using a Perkin-Elmer-Spectrum100 FTIR spectrometer, equipped with the Universal ATR top plate and diamond crystal. Spectra were recorded at a spectral resolution of 4 cm<sup>-1</sup> and a scan speed of 0.2 cm/s and data were normalised according to the Spectrum software from Perkin-Elmer. Raman spectra were acquired using a Jobin-Yvon LabRAM HR Raman spectrometer with the 514.5 nm line from a Lexel argon ion laser as excitation source.

Before mercury intrusion porosimetry (MIP) analysis, the geopolymer monoliths (1 \* 1 \* 2 cm<sup>3</sup>) were dried in an oven at 95 °C for 24 h. The Auto pore II 9220 Micromeritics mercury intrusion porosimeter used during measurements can work at low (to 200 kPa) and high pressures (from 414 MPa to 200 kPa), respectively. The Washburn equation relates the pressure measured during the test to the pore radius as follows by assuming that all the pores are cylindrical and thus pore openings are circular in cross section, which makes it possible to derive the model equation

$$P = -2\gamma \cos(\theta)/R$$

where  $P$  (psi) is the applied pressure,  $\gamma$  is the surface tension of mercury (485 dyne/cm),  $\theta$  is the advancing/receding contact angle (130°) and  $R$  is the capillary radius (Å) [20,21]. Nitrogen adsorption and desorption analyses of the foamed geopolymers were performed using an automatic Quantachrome gas sorption analyzer (instrument model ASIQC0V102-2). Small pieces of geopolymer, about 0.6 \* 0.6 \* 0.6 cm in size, were degassed for 14 h under a nitrogen atmosphere at a temperature of 100 °C to remove air from the pores. The samples were then cooled under a nitrogen



**Fig. 1.** Curing regime of foamed geopolymers.

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