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Original Research Paper

# Characterization of various fly ashes for preparation of geopolymers with advanced applications

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ABSTRACT

Fly ashes from different power stations in Australia (Collie, Eraring, Tarong) and Mongolia (4th thermal power station, Ulaanbaatar city) have been characterized by various techniques. It was determined that the Australian fly ashes are class F while the Mongolian fly ashes are class C. Due to their chemical and mineralogical differences, the fly ashes behaved differently when alkali activated to make geopolymers. The influence of various parameters on the preparation of geopolymers have been investigated and the results are used to establish a procedure for the routine manufacture of alkaline activated products. The applicability of using fly ash for building structural elements, corrosion resistant and thermally resistant materials will be presented.



## 1. Introduction

Geopolymers are a relatively new class of inorganic materials that have potential applications as building materials, high technology tooling for aeronautics, heat resistant fibre composites and toxic waste encapsulation [1]. Alkali activated binder materials made from blast furnace slag were described by Glukhovskii in the 1960s [2]. In the late 1970s Davidovits coined the name "geopolymer" for aluminosilicates formed by polycondensation of alumina and silica rich source materials [1]. There are increasing numbers of geopolymer publications where authors report on using aluminosilicate sources such as fly ash [3], dehydroxylated kaolinite [4], perlite [5] and rice husk ash [6]. An alkali activated geopolymeric structure is formed by polycondensation of alumina and silica sources in a highly alkaline medium at ambient to slightly elevated temperatures (60–90 °C) with controlled water content. Potential applications of geopolymer products are extensive and by varying key compositional ratios, such as the Si/Al ratio, their chemical and physical properties can be adjusted to suit the desired application.

Construction and building materials are considered the main application for the geopolymers though there are many studies that have identified other potential applications. For instance, geopolymers could be used as fire resistant coatings on metal and concrete due to their fire resistant properties [7,8]. Acid resistant behaviour of geopolymers has also been reported [9]. Potential applications of geopolymers depend not only on their intrinsic structural integrity but also the chemical and mineralogical compositions of the starting materials and the extent of the geopolymerization reaction. Fly ash is a heterogeneous material with huge variability in chemical composition based on coal type and power station burning conditions.

The chemical and mineralogical composition of the source fly ash is known to strongly influence the physical properties of geopolymers and therefore their suitable applications [10]. Thus it is important to thoroughly characterize each fly ash prior to the manufacture of geopolymers or alkali activated products to ensure their correct utilization. In this paper we summarize our latest work on the preparation of fire and chemically resistant geopolymers from various Australian and Mongolian fly ashes.

## 2. Experimental

Geopolymers were made from Australian fly ashes from Collie in Western Australia, Eraring in New South Wales and Tarong in Queensland. Mongolian fly ashes (Baganuur and Shivee ovoo) from the 4th thermal power station in Ulaanbaatar were also used. Since the two boilers of the 4th thermal power station used Baganuur coal and another two boilers Shivee ovoo coal, we were able to collect two different fly ashes from this power station. The geopolymerization reaction requires the dissolution of alumina and silicate sources in an alkaline medium. However, dissolution of these sources is strongly dependent on the mineralogical

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composition of the fly ashes, with amorphous phases dissolving much faster than crystalline phases. The chemical composition of each fly ash as determined by XRF is shown in Table 1.

Australian fly ashes belong to class F fly ash while Baganuur and Shivee ovoo fly ashes are class C fly ash according to the standard [ASTM C618-12]. Mineralogical composition of the Australian fly ashes and content of the amorphous part are shown elsewhere [10,11]. Mongolian fly ashes are produced from burning lignite coal. The main crystalline phases of the Mongolian fly ashes determined by XRD (not shown here) are quartz with inclusion of a little amount of lime, anorthite and magnetite. According to their classification, based on CaO content, the Mongolian fly ashes are likely to have some self-cementing properties.

For geopolymer paste preparation from the Australian fly ashes, only the chemical composition of the amorphous part of fly ash was used. The chemical composition of the amorphous part of the fly ash was determined by subtracting the content of the crystalline component measured by quantitative XRD from the bulk chemical composition measured by XRF. Using the chemical composition of the amorphous part of the fly ash for preparation of geopolymer results in superior mechanical properties than when the bulk chemical composition is used [10]. For the Mongolian fly ashes, the bulk chemical composition was used to formulate geopolymers due to limited resources for quantitative XRD analysis. For geopolymers synthesised from the Australian fly ashes, the Si:Al ratio of the geopolymers ranged between 2.0 and 3.0, while for Mongolian fly ashes the Si:Al ranged from 2.78 to 3.9.

Phase analyses of fly ash and geopolymer compositions were determined with XRD (Bruker D-8 Advance and Simadzu MAXima_X XRD-7000) and chemical composition of fly ashes with XRF (Philips PW2404 and Rigaku Primini type). Morphology of fly ashes and microstructure of geopolymers were determined by SEM and FESEM (Evo 40 XVP and JEOL JSM-6701F). Compressive strength of prepared samples were determined with a Lloyds Universal Testing Machine with a test regime that closely complied with ASTM C39.

Geopolymer shows mostly amorphous structure. However, full reaction of fly ashes by alkaline liquids is almost impossible leaving unreacted fly ash particles alongside geopolymer binder and excess alkaline liquid. The mineralogical composition of fly ash has a considerable influence on the thermal resistance of geopolymer. Therefore, in order to improve thermal and chemical resistance of fly ash geopolymers it may be necessary to modify their microstructure and the method adopted was calcination of the geopolymer. Geopolymer samples were calcined at 600 and 1000 °C temperatures for 6 h.

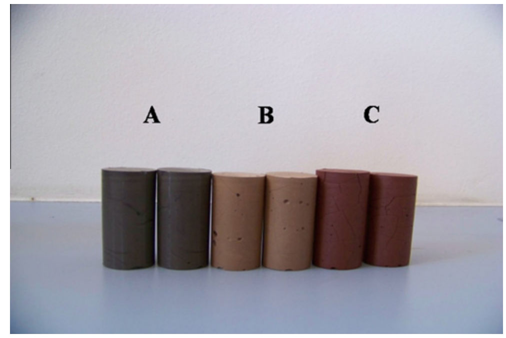


**Fig. 1.** Image of the Collie fly ash based geopolymers, as-prepared (A), after calcining at 600 °C and (B), after calcining at 1000 °C (C).

Chemical analysis of acid and alkali leached solutions was performed by ALS Chemex Pty Ltd. with an ICP-AES analyzer.

## 3. Results and discussion

### 3.1. Chemical resistance

Chemical resistance of as-prepared Collie fly ash based geopolymers was found to be good for acid but poor for alkali so an extra step of calcining the geopolymer was introduced to improve chemical resistance. Fig. 1 shows an image of Collie fly ash based geopolymers before and after calcining at 600 and 1000 °C.

Calcination at 1000 °C caused cracking of the samples with colour changing from a dark grey to red. The colour changes were caused by oxidation of iron from the fly ash and the cracking was likely due to the expansion of the quartz in the fly ash [12] making this temperature unsuitable for further experimental processing. After also trialing calcination temperatures of 800 °C and 600 °C, the lower temperature was selected as it produced minimal changes in mineralogical composition but significant improvement in chemical resistance (see following paragraphs). Quantitative XRD was used to determine that the amorphous composition of Collie fly ash based geopolymers decreased from 63.4 wt.% to 61.6 wt.%, while the content of hematite increased from 1.3 wt.% to 2.2 wt.%, quartz from 19.7 wt.% to 20.7 wt.% and mullite

**Table 1**
Chemical composition of fly ashes.

| Oxide | Collie (Australia) (wt.%) | Eraring (Australia) (wt.%) | Tarong (Australia) (wt.%) | Baganuur (Mongolia) (wt.%) | Shivee ovoo (Mongolia) (wt.%) |
|---|---|---|---|---|---|
| $SiO_2$ | 51.38 | 65.47 | 73.68 | 55.2 | 33.85 |
| $Al_2O_3$ | 26.9 | 23.0 | 22.4 | 14.15 | 12.15 |
| $Fe_2O_3$ | 13.2 | 4.03 | 0.64 | 10.55 | 9.89 |
| CaO | 1.74 | 1.59 | 0.08 | 15.0 | 30.8 |
| $K_2O$ | 0.9 | 1.68 | 0.53 | 1.31 | 0.73 |
| $TiO_2$ | 1.47 | 0.84 | 1.28 | 0.25 | 0.35 |
| MgO | 1.41 | 0.51 | 0.17 | 1.56 | 6.41 |
| $Na_2O$ | 0.41 | 0.56 | 0.09 | – | – |
| $P_2O_5$ | 1.09 | 0.27 | 0.08 | – | – |
| SrO | 0.23 | 0.05 | 0.01 | 0.25 | 0.44 |
| MnO | | | | 0.34 | 1.185 |
| $SO_3$ | | | | 1.23 | 3.65 |
| LOI (1000 °C) | 0.44 | 1.37 | 0.79 | 1.05 | 0.30 |
| Sum of aluminosilicates | 78.3 | 88.47 | 96.08 | 69.35 | 46 |
| Sum of alkali | 1.31 | 2.24 | 0.62 | 1.31 | 0.73 |
| $SiO_2/Al_2O_3$ | 1.91 | 2.85 | 3.29 | 3.9 | 2.78 |
| Si/Al (molar) | 1.62 | 2.42 | 2.79 | 3.32 | 2.37 |