



Formation of one-part-mixing geopolymers and geopolymer ceramics from geopolymer powder



Liew Yun-Ming^{a,*}, Heah Cheng-Yong^a, Long-yuan Li^b, Nur Ain Jaya^a, Mohd Mustafa Al Bakri Abdullah^{a,c}, Tan Soo Jin^c, Kamarudin Hussin^{a,c}

^a Center of Excellence Geopolymer and Green Technology (CEGeoGTech), School of Materials Engineering, Universiti Malaysia Perlis (UniMAP), P.O. Box 77, D/A Pejabat Pos Besar, Kangar, Perlis 01000, Malaysia

^b School of Engineering, Plymouth University, Plymouth PL4 8AA, UK

^c Faculty of Engineering Technology, Universiti Malaysia Perlis (UniMAP), P.O. Box 77, D/A Pejabat Pos Besar, Kangar, Perlis 01000, Malaysia

HIGHLIGHTS

- Geopolymer powder could produce one-part-mixing geopolymers by mixing with water.
- Continual geopolymer matrix formation was revealed in one-part-mixing geopolymer.
- Geopolymer powder could be used to produce high strength ceramics.
- Powder metallurgy and sintering could reduce the risk of excessive cracking.

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ABSTRACT

Geopolymer powder prepared through pre-curing and pulverization showed great potential to produce one-part-mixing geopolymers as well as high flexural strength geopolymer ceramics. The one-part-mixing geopolymers were prepared by mixing geopolymer powder with water while the geopolymer ceramics were prepared by powder metallurgy and sintering. The one-part-mixing geopolymers achieved a compressive strength of 10 MPa after 28 days with formation of geopolymer precipitates in conjunction with zeolite phases. Despite the lower strength, they remained stable and did not disintegrate when immersed in water. Besides, the geopolymer ceramics exhibited high flexural strength (90 MPa) after sintering at 1200 °C as result of nepheline formation.

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1. Introduction

Geopolymers have become a potential alternative binder to ordinary Portland cement (OPC) in some applications due to its sustainability criteria of lower emission of greenhouse gases and low energy consumption [1]. Geopolymers are inorganic polymeric materials with three-dimensional Si—O—Al frameworks synthesized from aluminosilicates which dissolved in alkaline medium [2]. Apart from having excellent early and long-term mechanical strength, geopolymers also have low shrinkage value, low density, good immobilization of heavy metal and good fire and chemical resistance [3,4]. This accounts for the suitability of geopolymers for applications as construction materials in building, refractory and wastewater treatment industries [5–8].

Geopolymers are formed through geopolymerization reaction involving the dissolution of aluminosilicate sources in highly alkaline medium releasing Si and Al species, diffusion, exchange and oligomerization between the dissolved species in aluminosilicates and alkaline solution with formation of small coagulated structures, gelation and lastly hardening to form hard solid [9–11]. The ordinary formation process of geopolymers was a two-part-mixing process, which involves preparation of alkali activator 1 day prior to mixing and the mixing of solid aluminosilicates with the prepared alkali activator. Due to the importance and growing demands of geopolymer technology, researchers are finding ways to produce geopolymers from a ready-mix precursor that can directly mix with water just like OPC does, which is termed as the one-part-mixing geopolymer system [12]. There are few reasons whereby the one-part-mixing geopolymer system is essential. This is to avoid handling of a large quantity of highly corrosive solution as in the two-part-mixing geopolymer mix. Besides, the

* Corresponding author.

E-mail address: yun86_liew@yahoo.com.my (L. Yun-Ming).

use of highly corrosive solution requires large storage as well as restricts the transportation of the solution for on-site applications. Hence, large-scale applications are limited.

Early study on one-part-mixing geopolymer mixture involved the calcination of aluminosilicates with solid alkali hydroxides or carbonates. Clinker was formed, pulverized into powder (one-part-mixing geopolymer precursor) and finally added with water to form one-part-mixing geopolymers. The calcination process involved transformation into Al-rich phase with hydroxides incorporated in the aluminosilicates matrix and formation of reactive functional groups. In particular, Kolousek et al. [13] calcined low-quality kaolin together with alkali hydroxides at 550 °C for 4 h, followed by pulverization and mixing water to produce one-part-mixing geopolymers. However, the one-part-mixing geopolymers exhibited extremely low compressive strength (<1 MPa) after 7 days. Subsequently, Feng et al. [14] prepared one-part-mixing geopolymer precursor through the calcination of a mixture of albite, sodium hydroxide (NaOH) and sodium carbonate (Na_2CO_3). The produced one-part-mixing geopolymers had excellent compressive strength of >40 MPa after 28 days. The final products contained amorphous geopolymer phases and crystalline zeolite phases. A similar study had been carried out by Ke et al. [15] using red mud and NaOH pellets. The one-part-mixing geopolymers attained a maximum compressive strength of 10 MPa after 28 days. These studies required the necessity of high-temperature calcination in order to obtain the one-part-mixing geopolymer precursor. Another method of producing one-part-mixing geopolymers with no requirement of calcination was proposed by Hajimohammadi et al. [16]. One-part-mixing geopolymers were formed by direct mixing geothermal silica, solid sodium aluminate with water. Nevertheless, their study focused on the microstructural observation and the compressive strength of the one-part-mixing geopolymers was not discussed. Formation of geopolymer matrix and typical characteristic diffuse halo of geopolymer could be observed through SEM and XRD analyses, respectively. In addition, Nematollahi et al. [17] formed one-part-mixing geopolymers constituting low calcium fly ash, slag, hydrated lime with solid sodium silicate (Na_2SiO_3) and NaOH. The comparison of geopolymers formed through one-part and two-part mixing processes has found that the one-part-mixing geopolymers made from fly ash have moderate to high compressive strength of over 37 MPa after 28 days. The compressive strength was comparable to the two-part-mixing geopolymers (42.5 MPa after 28 days).

In the present study, the aluminosilicates and alkali activator were mixed to produce geopolymer paste which was pre-cured and then pulverized into geopolymer powder. The geopolymer powder was mixed with water directly to form one-part-mixing geopolymers. The adopted preparation method differed from those aforementioned above in order to study any possible formation methods of one-part-mixing geopolymers. The previous works have investigated the effect of NaOH concentration, metakaolin/activator and sodium silicate/sodium hydroxide ratios [18,19] on the mechanical properties of the mixed geopolymer pastes. The geopolymer powder could be used as the ready-mixed precursor to form one-part-mixing geopolymers by just adding water. In this article, the changes in physical properties and extent of strength increment were studied by varying the curing temperature and time.

Most importantly, geopolymer powder can be advantageously used to produce geopolymer ceramics. Sintering caused the formation of ceramics product due to the formation of crystalline phases. For as-cured monolithic geopolymers, direct high-temperature sintering caused excessive shrinkage and cracking and consequently lower the strength of the final products [20,21]. Based on Xie et al. [22], the formation of geopolymer powder, powder metal-

lurgy and lastly sintering could address the cracking issue which occurred in the as-cured geopolymers. They successfully produced leucite glass-ceramics from geopolymer powder based on potassium precursors. For sodium-based geopolymers, nepheline-predominated crystallites formed when sintered at elevated temperatures [23,24]. Nepheline ceramics have hitherto been produced through sintering-crystallization, controlled devitrification and vitrification process of zeolites [25,26], fly ash and slag [27] in the temperature range of 600–1200 °C. In their study, the mechanical strength of the prepared nepheline ceramics was not studied. The production of nepheline ceramics using geopolymer powder has not been studied. Thus, this work investigated another choice method to produce nepheline ceramics from geopolymer.

To recap briefly, the physical and mechanical properties of one-part-mixing geopolymers and geopolymer ceramics prepared from sodium-based geopolymer powder were investigated in this work.

2. Experimental work

2.1. Materials

Clay-based aluminosilicate source was used in this work. For the preparation of one-part-mixing geopolymers, metakaolin was used. Kaolin was subjected to thermal treatment at 800 °C for 2 h to obtain metakaolin. On the other hand, kaolin was utilised in the formation of geopolymer ceramics. The chemical composition of kaolin and metakaolin employed was determined using XRF analysis is tabulated in Table 1. The microstructures of kaolin and metakaolin particles are shown in Fig. 1. It can be seen that both kaolin and metakaolin are plate-like and needle-like structures. However, the plate-like structure was more open in metakaolin [18,28].

The activator solution was a mixture of sodium hydroxide (NaOH) solution and liquid sodium silicate (Na_2SiO_3). The NaOH pellet has purity of 99% while the liquid Na_2SiO_3 contains 30.1% SiO_2 , 9.4% Na_2O and 60.5% H_2O and $\text{SiO}_2/\text{Na}_2\text{O}$ modulus of 3.2. Based on Jansson et al. [29], the modulus of Na_2SiO_3 affected the pH, viscosity and the setting time of the geopolymer mixtures. Low modulus close to 1 led to precipitation of hydrous sodium metasilicate crystals [30]. Thus, higher modulus Na_2SiO_3 was chosen as it becomes more siliceous that provides more free Si species for geopolymer structure formation [31]. The NaOH stock solution was prepared and cooled down to room temperature before use. The activator solution was prepared 24 h before mixing process.

To avoid confusion, the following terms are used in the present study:

- i) Fresh paste is the paste before pre-curing;
- ii) Pre-cured geopolymers is the sample that has set after pre-curing;
- iii) Fresh one-part-mixing geopolymer paste is the paste after the geopolymer powder was mixed with water;
- iv) One-part-mixing geopolymer is the hardened geopolymer paste produced from the mixing of geopolymer powder and water;
- v) Green body is the geopolymer ceramic before sintering; and
- vi) Geopolymer ceramic is the sintered ceramics produced from the geopolymer powder.

2.2. Formation of geopolymer powder

The aluminosilicate source was mixed with the prepared activator solution until a homogeneous paste was achieved. The paste was moulded and pre-cured in the oven. Then, the pre-cured geopolymer was pulverized and sieved to produce geopolymer powder. The experimental procedure is illustrated in Fig. 2.

In order to produce metakaolin geopolymer powder, the metakaolin/activator and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratios were 0.8 and 0.2, respectively. The fresh paste was pre-cured at 80 °C for 4 h. These mixing ratios and pre-curing condition were selected

Table 1
Chemical composition of kaolin and metakaolin as determined by XRF analysis.

Compound	Mass (%)	
	Kaolin	Metakaolin
SiO_2	54.5	54.1
Al_2O_3	32.4	33.0
K_2O	5.58	5.67
TiO_2	1.33	1.34
Fe_2O_3	4.32	4.41
MnO_2	0.09	0.11
ZrO_2	0.08	0.09

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