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Effects of sodium hydroxide and sodium silicate solutions on compressive and shear bond strengths of FA–GBFS geopolymer

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HIGHLIGHTS

- FA–GBFS geopolymer activated with sodium silicate (cured at 23 °C) resulted mainly in amorphous gel.
- Activated with sodium hydroxide resulted in significant crystalline CSH.
- Incorporation of GBFS enhanced compressive strength and microstructure of FA geopolymer pastes.
- Shear bond strength depended on strength and amount of NASH gel of FA–GBFS geopolymer.

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ABSTRACT

This article investigated the effects of sodium hydroxide and sodium silicate solutions on the properties of fly ash (FA)–granulated blast furnace slag (GBFS) geopolymer. Three types of geopolymer pastes viz., FA paste, FA + GGBS paste and GGBS paste were tested. They were activated with three types of alkaline solutions viz., sodium hydroxide solution (NH), sodium silicate solution (NS), and sodium hydroxide plus sodium silicate solution (NHNS). NH with 10 molar concentration, alkaline liquid/binder ratio of 0.60 and curing at ambient temperature of 23 °C were used for all mixes. The results indicated that the reaction products and strengths of geopolymer depended on the types of source materials and alkali activators. The use of NH and NHNS solutions resulted in the formation of crystalline calcium silicate hydrate (CSH) which co-existed with amorphous gel. Whereas the use of NS solution resulted in mainly the amorphous products with only a small amount of crystalline CSH in GBFS paste. The increase in GBFS content enhanced the compressive strength and microstructure of geopolymer pastes due to the formation of additional CSH. The shear bond strength between Portland cement concrete substrate and geopolymer paste was found to relate to both compressive strength and amount of NASH gel of geopolymer paste.

1. Introduction

The manufacturing of Portland cement (OPC) results in high emission of carbon dioxide (CO_2) to atmosphere causing greenhouse effect [1]. To solve this problem, the use of geopolymer as an alternative binder for application in concrete industry is recommended [2–5]. Geopolymeric material is formed using source materials containing silica (SiO_2) and alumina (Al_2O_3) such as fly ash (FA), calcined kaolin or metakaolin and granulated blast furnace slag (GBFS) activated with alkali solutions [6–10]. Specifically, sodium hydroxide based geopolymer is more attractive as it gives lower carbon footprint than sodium silicate based geopolymer. For normal strength concrete, Turner and Collins [11] showed that the CO_2 footprint of a sodium silicate based geopolymer concrete was approximately 9% less than comparable OPC concrete.

Fly ash is a by-product from coal burning in thermal power plants. Both low calcium fly ash [6,12] and high calcium fly ash [8,13] are suitable source materials due to their high SiO_2 and Al_2O_3 contents. One obstacle with the use of fly ash is the low

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strength development with ambient temperature curing [6]. A number of researchers [14–16] have incorporated additives containing calcium to enhance the strength development of fly ash geopolymer. The presence of calcium has a positive effect on the mechanical properties of geopolymeric material [9] by forming additional calcium silicate hydrate (CSH) which coexists with the geopolymer products [17,18]. However, a certain amount of calcium can lead to a decrease on compressive strength of geopolymers [19,20]. Pacheco-Torgal et al. [21] reported that when calcium hydroxide percentages above 10% are used, strength decrease after curing for 14 days is noticed.

GBFS is usually utilized as a binder in concrete industry due to its high content of CaO; and SiO_2 and Al_2O_3 in an amorphous state [22,23]. Therefore GBFS shows pozzolanic and binding properties in an alkaline medium [24]. A number of researchers [6,24,25] found that the incorporation of GBFS to FA geopolymer resulted in additional calcium in the system and the mechanical properties and microstructure of geopolymer were improved.

Several researchers indicate that sodium silicate and sodium hydroxide based activators are suitable considering the mechanical properties of GBFS and FA geopolymers [6,25,26]. The main reaction products formed as a result of alkali activation of GBFS are CSH and/or CASH gels similar to those of PC [27], whereas the main product of alkali activation of FA is NASH gel [28]. As GBFS is a glassy phase material, it is, therefore, easier to activate than fly ash. Fly ash contains a larger portion of crystalline phase and usually requires temperature between 40 and 85 °C to accelerate the reaction [24]. Previous studies suggested that the incorporation of GBFS results in the enhancement of strength development of fly ash geopolymers [29,30]. Bond strength is one of the most important properties of high performance binder for application as a repair material. Several tests i.e. pull-out, splitting, flexural, and slant shear are used to evaluate the bond strength of repair materials [3]. The slant shear test was successfully used to study the shear bond strength of concrete repair [31] and bond between concrete substrate and geopolymer [3,4].

Therefore, the use of GBFS to improve the strength of FA geopolymer is very attractive. The objective of this study is to investigate the mechanical properties viz., compressive strength and shear bond strength, and microstructure of FA–GBFS geopolymer with types of alkaline solutions. The obtained results should be beneficial to the understanding and to the future applications of FA–GBFS geopolymer.

2. Experimental details and testing analysis

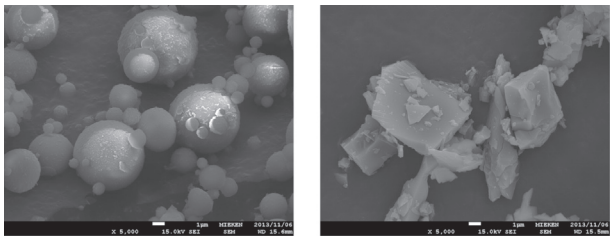
2.1. Materials

The materials used in this research were fly ash (FA) from Hekinan power plant and ground granulated blast furnace slag (GBFS) from Nippon Steel & Sumitomo Metal Corporation. 10 M sodium hydroxide (NH) and sodium silicate (NS) with 11.67% Na_2O, 28.66% SiO_2, and 59.67% H_2O were used as activators.

The chemical compositions and physical properties of FA and GBFS are shown in Tables 1 and 2. The specific gravity of FA and GBFS were 2.20 and 2.91. The median particle sizes of FA and GBFS were 12.3 and 12.4 μm with the corresponding Blaine finenesses of 3100 and 4950 cm^2/g, respectively. Fig. 1 shows the scanning electron micrographs (SEM) of FA and GBFS. The FA consisted of spherical particles with smooth surface, while the GBFS consisted of irregular and angular particles similar to the previously published results [32]. The XRDs of FA and GBFS as shown in Fig. 2 showed that as-received FA mainly consisted of amorphous phase as shown by a hump around 18–28 °2theta with some crystalline phases of mullite ($Al_6Si_2O_{13}$), quartz (SiO_2), magnesioferrite (Fe_3O_4) and calcium oxide (CaO), while as-received GBFS mainly consisted of amorphous phase as shown by a hump around 25–35 °2-theta [27] with a small amount of magnetite.

Table 2
Physical properties of FA and GBFS.

Materials	Specific gravity	Median particle size (μm)	Blaine fineness (cm^2/g)
FA	2.20	12.3	3100
GBFS	2.91	12.4	4950



(a) FA (b) GBFS

Fig. 1. SEM of FA and GBFS.

2.2. Mix proportion and curing

The mix proportions of geopolymer pastes are shown in Table 3. Constant liquid alkaline to binder ratio of 0.60 was used for all mixes. Three types of geopolymer pastes viz., FA paste, FA + GGBS paste and GGBS paste and three types of alkaline solutions viz., sodium hydroxide solution (NH), sodium silicate solution (NS), and sodium hydroxide plus sodium silicate solution (NHNS) were used. The NHNS solution was prepared with NS/NH ratio of 2.0. For the other series the NH and NS solutions were used directly. For the mixing of pastes, FA and GBFS were dry mixed until the mixture was homogenous. Right after, the liquid solution was added and the mixing of paste was done for another 3 min.

2.3. Testing and analysis

2.3.1. Compressive strength of geopolymer pastes

After being mixed, the fresh geopolymer pastes were placed into cylindrical moulds of 50 mm in diameter and 100 mm in height. They were covered with vinyl sheet and placed in ambient temperature curing (23 °C). The samples were demoulded at the age of 1 day and immediately wrapped with vinyl sheet to protect moisture loss and kept in the 23 °C controlled room until the testing age. The compressive strengths were tested at the ages of 7, 28, and 60 days. The reported results were the average of three samples.

2.3.2. X-ray diffraction (XRD) and scanning electron microscopy (SEM) of geopolymer pastes

At the age of 28 days, the geopolymer paste samples were broken and the middle portion was collected and ground to fine powder. The XRD scans were performed at 5–60 °2theta with an increment of 0.02°/step and a scan speed of 0.5 s/step. The amorphous phases of geopolymer pastes were determined by quantitative XRD analysis using Bruker's TOPAS software. The specimen was placed on a brass stub sample holder with double stick carbon tape. The specimen was dried using infrared light for 5 min and then coated with a layer of gold using a blazer sputtering coater. The micrographs were recorded at 20 kV and 1000× magnification.

2.3.3. Shear bond strength between concrete substrate and geopolymer paste

The shear bond strength was evaluated using the slant shear test of concrete substrate and geopolymer paste. The slant angle of 30° to the vertical is recommended by ASTM C882 [33] because the failure stress is close to the minimum stress [31]. However, stiffer angle of 45° is also officially used for the standard

Table 1
Chemical compositions of FA and GBFS (by weight).

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	LOI
FA	52.31	27.04	6.85	3.32	1.23	1.29	1.15	0.99	1.60
GBFS	30.53	13.67	0.33	46.00	5.09	0.36	0.24	–	0.22