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The Incorporation of Sodium Hydroxide (NaOH) Concentration and CaO-Si Components on Ground Granulated Blast Furnace Slag Geopolymers.

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Abstract. This paper investigates the incorporation of sodium hydroxide (NaOH) concentration and CaO-Si component on ground granulated blast furnace slag geopolymers (GGBFS). An optimal mechanical property of GGBFS with various NaOH concentrations had been determined by performing a number of compressive strength tests. It was found that GGBFS with 10M NaOH concentration resulted in high compressive strength at 65.31 MPa after 28 days curing. The microstructure analysis of the GGBFS geopolymers using SEM and FTIR revealed the formation of calcium silicate hydrate ($\text{Ca}_5\text{Si}_6\text{O}_{16}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$) and calcium carbonate (CaCO_3) phase within the geopolymer backbone chain. The optimum NaOH concentration allows the acceptable formation of CaO-Si bond on the GGBFS geopolymers main structure and thus leads to optimum compressive strength.

1. Introduction

Geopolymers is an inorganic material consisting of three-dimensional structures and amorphous phase activated by alkali activation of an aluminosilicate sources at ambient or elevated temperature through a geopolymerization process. Also, an aluminosilicate materials contains silica (Si) and aluminium (Al) react with a highly alkali solution through dissolution and polycondensation reaction in producing geopolymer binder [1-3]. The fundamentals of geopolymerization are the dissolution of solid aluminosilicate oxide in MOH solution (M = alkali metal), dissipation of dissolved Al and Si to an inter-particle space, the formation of gel phase by polymerization and finally the hardening of geopolymer gel [4, 5]. The mixing of Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) is the most used as alkali activator in the geopolymerization reaction [6-8]. The dissolution of aluminosilicate materials occurs with the presence of alkali hydroxide while sodium silicate is essential as binder, plasticizer or dispersant [9]. Alkali solution influence a certain atoms of Al and Si to dissolve the aluminosilicate materials, produce monomers in liquid phase, and further to polycondense to form rigid network [10, 11]. In order to enhance the polymerization reaction, sodium silicate is favoured in activating solution owing to its soluble silica species [12, 13].



According to Deb et al. [14], the inclusion of ground granulated blast furnace slag up to 20% in fly ash based geopolymers activated with sodium hydroxide and sodium silicate obtained excellent compressive strength. High calcium geopolymer concrete by fly ash Class C with the mixture of alkali activator of 15M NaOH concentration was prepared by Topark-Ngam et al. [15] whereas exhibited a compressive strength of 54.4 MPa after 28 days of curing. Also, the strength continues to develop with time similar to ordinary Portland cement concrete. Researchers found that the common sodium hydroxide concentration utilized for forming geopolymer concrete ranging from 8M to 16M and the highest strength according to these studies were observed at concentrations in the range of 12 M and 16 M [16-20]. Otherwise, Hamidi et al. [21] exhibited the lowest value of flexural strength of using 4M and gained the optimum value by using 12M.

The present work deals with the synthesis and characterization geopolymer paste prepared from ground granulated blast furnace slag activated with the mixing of sodium hydroxide and sodium silicate solution under ambient environment condition. The requirement of geopolymer concrete formed at ambient temperature will be crucial to be suitable for site application. Thus, this works determines the effect of sodium hydroxide concentrations on GGBFS geopolymers paste properties because researchers about solely slag binder are limited.

2. Experimental

2.1. Material

Ground granulated blast furnace slag (GGBFS) was collected from Ann Joo Integarted Steel Sdn. Bhd., Penang, Malaysia. This industrial by-product was sun-dried first before kept in the oven within temperature 100 °C to 105 °C for 24 hours for drying. The chemical composition of GGBFS raw materials were analyzed by X-ray fluorescence (XRF) spectroscopy and summarized in table 1. The amount SiO₂ + Al₂O₃ and CaO in the GGBFS are 40.9% and 50.37% respectively.

Table 1. Chemical composition of GGBFS.

| Compound | CaO | SiO ₂ | Al ₂ O ₃ | MgO | Fe ₂ O ₃ | TiO ₂ | LOI |
|----------|-------|------------------|--------------------------------|-----|--------------------------------|------------------|------|
| Wt % | 50.37 | 30.4 | 10.5 | 3.2 | 0.53 | 0.98 | 2.87 |

Sodium hydroxide (NaOH) flakes with 99% purity and sodium silicate solution were used as the alkaline activator and purchased from the South Pacific Chemical Industries Sdn. Bhd., Malaysia. The NaOH with various concentrations (6M to 14M) expressed in molarity (M) were prepared before 24 hours of its use to ensure proper dissolution and to completely dissipate the hydration heat. The liquid alkaline activators were prepared by mixing NaOH solution with Na₂SiO₃ solution at fixed ratio of 2.5 until clear solution was obtained.

2.2. Preparation of GGBFS Geopolymers

The homogenous slurry was prepared as the dry GGBFS fine powder (~150 μm) mixed with alkali activator solution and solid-to-liquid ratio at fixed of 2.5 and 1.0 respectively. The GGBFS slurry were molded and compacted as followed ASTM C109 with the acrylic mould was 50 mm x 50 mm x 50 mm and kept cured at room temperature for 28 days. The details design of mixtures is presented in table 2.

Table 2. Details of mixing conditions for GGBFS geopolymer paste with various NaOH concentration.

| Mixture | NaOH concentration (M) | Solid-to-liquid ratios | Na ₂ SiO ₃ /NaOH ratios | Code |
|---------|------------------------|------------------------|---|-----------------|
| M1 | 6 | 1.0 | 2.5 | 6M/1.0SL/2.5SN |
| M2 | 8 | 1.0 | 2.5 | 8M/1.0SL/2.5SN |
| M3 | 10 | 1.0 | 2.5 | 10M/1.0SL/2.5SN |
| M4 | 12 | 1.0 | 2.5 | 12M/1.0SL/2.5SN |
| M5 | 14 | 1.0 | 2.5 | 14M/1.0SL/2.5SN |

2.3. Testing and Analysis Method

Five different GGBFS geopolymers paste were prepared by varying NaOH concentration. Alkali activator with different NaOH concentration of 6, 8, 10, 12 and 14M was added to the dry GGBFS powder with the fixed of solid-to-liquid and Na₂SiO₃/NaOH ratios of 1.0 and 2.5 respectively. The solids and solutions were mixed for at least for 1 min to maintain homogeneity. The compressive strength of GGBFS geopolymers were tested accordance to ASTM C109/109M-05. The bulk density was measured after 28 days of curing by measuring the dimension and mass of the samples. The mineralogical, microstructural and chemical study on GGBFS geopolymers were analyzed by XRD, SEM and FTIR analysis respectively.

For these characterizations of GGBFS geopolymers, the fracture samples of materials after strength test were collected. Then, the samples were finely ground to a size less than 150 μm . The mineralogical analysis for the GGBFS raw material and GGBFS geopolymers was made mainly by XRD analysis using XRD-6000, Shimadzu X-ray diffractometer with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$) operating at 40kV, 30 mA, at 2θ range from 10° to 80° with scan rate of 0.01°. The XRD pattern was analyzed using X'pert Highscore Plus software. The functional group was done through spectrum RXI spectrometer. The specimen was powdered samples scanned from 4000 cm^{-1} to 650 cm^{-1} .

3. Results and Discussions

3.1. Compressive Strength Measurement

The compressive strength of GGBFS geopolymers paste synthesized at five different NaOH concentrations as illustrated in figure 1. An increase in the NaOH concentration from 6M to 10M exhibited an increase of 14.05% of the GGBFS geopolymer strength after 28 days curing. The optimum NaOH concentration for synthesis of GGBFS geopolymer was 10M which displayed a stable strength while achieving its highest strength of 65.31 MPa. However, by increasing the NaOH concentration from 12M to 14M, the compressive strength decreased to 32.2% of 44.07 MPa.

By referring to figure 1, the compressive strength increased with the increasing of NaOH concentration through the leaching of silica and alumina from aluminosilicate material [22]. The higher concentration provides greater dissolving ability to slag particles and forms more reactive bonds for the monomers consequently boosting the inter-molecular bonding strength of the geopolymer [23-25]. Additionally, the highest density of the samples as shown in Figure 5 related the highest compressive strength. However, the strength reduction obtained as the GGBFS geopolymers paste activated by using NaOH concentration above 10M. This was believed so by the excess hydration ions (OH⁻ and Na⁺) at a high concentration of NaOH. The excess of OH⁻ induced aluminosilicate gel precipitation at early ages of geopolymerization reaction while much Na⁺ prevents the formation of oligomers at later stages which resulting in lower strength, respectively [26-27].

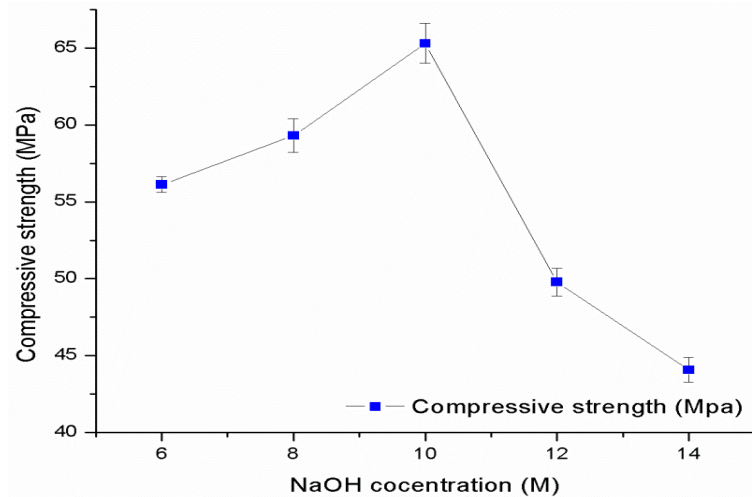


Figure 1. Compressive strength of GGBFS geopolymers at various NaOH concentrations.

Similar results were obtained by Lee et al. [28], whereby the excessive sodium hydroxide concentration was associated with the geopolymer strength deterioration as there was hydrolytic attack on the primary aluminosilicate gel. The excessive OH⁻ ion from dissolution at higher NaOH concentration could lead to ionic congestion which may cause a hindrance to oligomers precursor – (Al(OH)₄ and Si(OH)₄)⁻ transport. According to Alonso & Palomo [29], higher NaOH concentration causes a higher dissolution rate, however, higher amount of ions dissolved in the geopolymerization reaction. It was unacceptable for the system due to limitation of movement of the dissolved ions.

During the slag geopolymerization, the silica ion (SiO₄⁻) dissolved from slag material and was provided by alkali activator to support the geopolymer network that promoted the reactions with Ca²⁺ released from the slag. Then, further reaction of silicate ions from the activator was consumed while the slag dissolution continued. The polycondensation of these components was induced to gelation, therefore resulting in calcium silicate hydrate (CSH) with some of the Si⁴⁺ substituted by Al³⁺ supplied by the slag, and also the incorporation of Na⁺ alkalis into the charge balancing site and was attached onto the geopolymer structure as shown in figure 2.

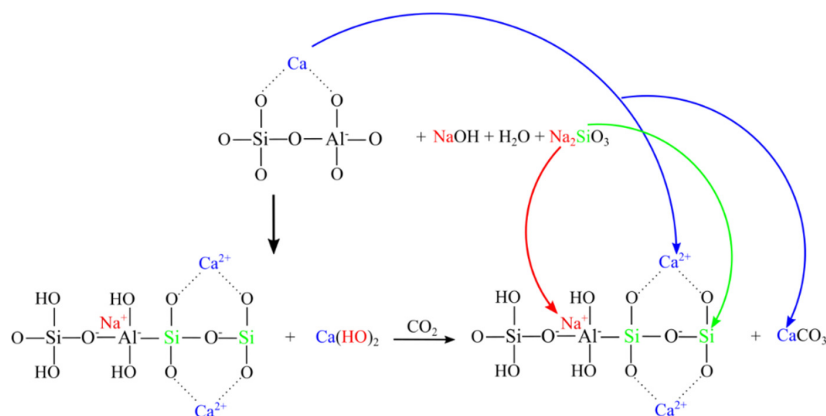


Figure 2. The ground granulated blast furnace slag geopolymers by NaOH activation.

3.2. Microstructural Analysis

Figure 3(a) shows the microstructure of ground granulated blast furnace slag particle. It reveals the morphology of GGBFS particles as irregular-shaped and sharp-edged particles with unsmooth surface. figure 3(b) to figure 3(f) reveals the microstructure of GGBFS geopolymers activated by different NaOH concentration (6M to 14M). The GGBFS geopolymers activated by 6M NaOH as shown in figure 3(b) appeared rough surface with unreacted and/or partially reacted remnant GGBFS particles. The edges of slag particles were slightly altered due to alkali activation with partially reacted particle on the surface.

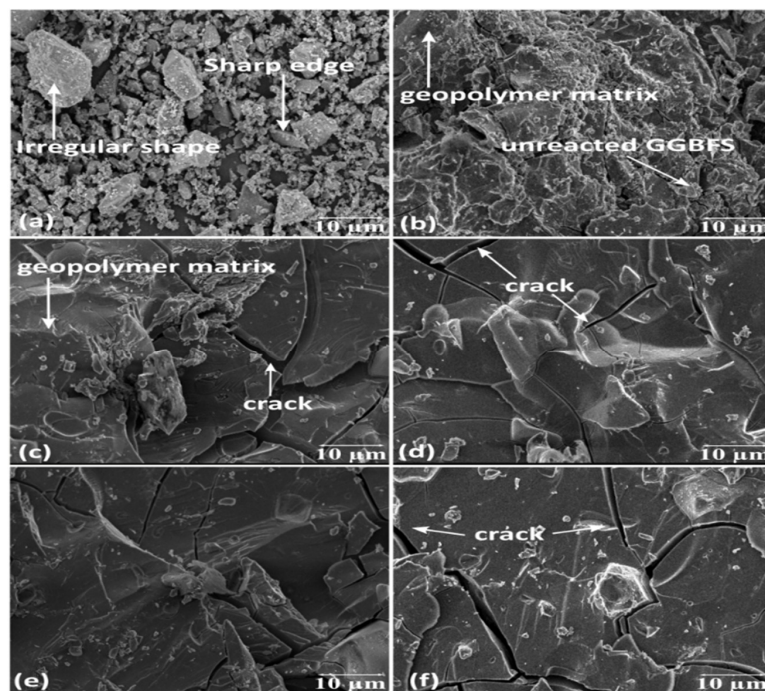


Figure 3. SEM micrograph of GGBFS geopolymer on various NaOH concentration with x1000 magnification; (a) Raw GGBFS (b) 6M/1.0SL/2.5SN (c) 8M/1.0SL/2.5SN (d) 10M/1.0SL/2.5SN (e) 12M/1.0SL/2.5SN and (f) 14M/1.0SL/2.5SN.

Otherwise, figure 3(c-f) the continuous mass of aluminosilicate and crack was observed on the GGBFS geopolymer surface. The crack appeared primarily due to excessive evaporation of water during the hardening process. Figure 3(d) shows less unreacted GGBFS particles activated with alkali activator at 10M exhibited a higher compressive strength of 65.31 MPa for 28 days test. Additionally, the increase of NaOH concentration in the geopolymer system resulted in the enhanced leaching of silica and alumina from GGBFS particles to the alkali solution [30, 31]. The surface became denser and produced a stronger geopolymer matrix that increased the compressive strength of the geopolymer sample. The alkali activator of mixture M3 was more effective and showed the most reaction compared others, leading to a higher strength.

Meanwhile, geopolymer matrix still can be seen in samples at Figure 3(e) and 3(f) concentration with the presence of larger cracks and loose microstructure. This caused the significant decrease in strength of the geopolymer samples. The similar observation on the microstructure was supported by Santa [32]. The bottom ash geopolymer synthesized by high concentrations of NaOH obtained the carbonation particles that caused by the excess of reagent. Hence, the NaOH solution was not favourable to the dissolution as it led to the presence of cracks. The presence of excess microcracks

morphology demonstrated by delayed geopolymerization reaction. The delayed geopolymerization reaction ascribed to fast activation in the early stage [33]. In this study, the formed GGBFS geopolymer gel occurred in fast setting due to higher CaO as tabulated in table 1.

3.3. Functional Group Identification Analysis

Figure 4 presents the FTIR spectra of ground granulated blast furnace geopolymers activating various NaOH concentrations and tested after 28 days. Fourier transform infrared (FTIR) spectrums are divided into three regions. The first region is located at wavenumber higher than 1600 cm^{-1} . The most bands appeared in this region were assigned at range 3200 cm^{-1} to 3300 cm^{-1} and 1640 cm^{-1} to 1650 cm^{-1} . The broad band around 3200 cm^{-1} to 3300 cm^{-1} was the OH-stretching vibration. Meanwhile, the absorption band near 1640 cm^{-1} to 1650 cm^{-1} was attributed to the OH-bending.

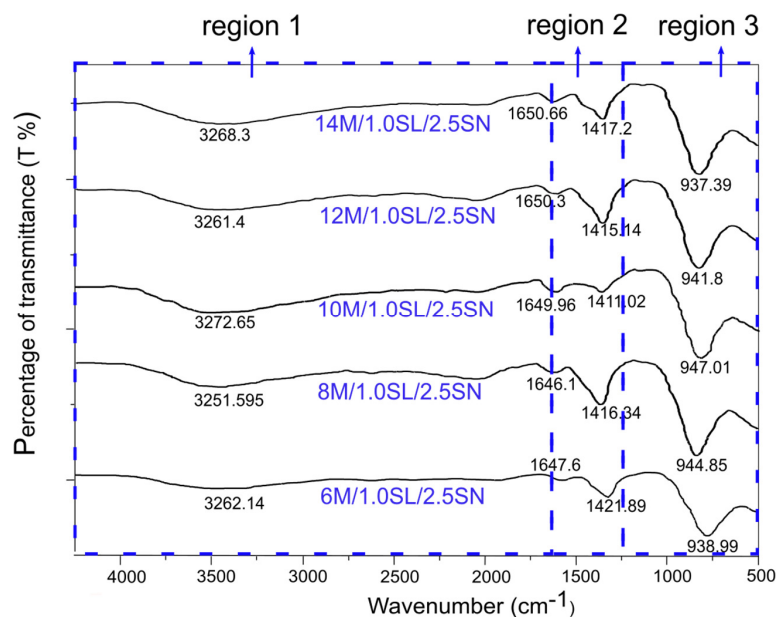


Figure 4. FTIR spectra of GGBFS geopolymers for various design synthesized at different NaOH concentrations.

These broad absorption bands were assigned to the bound water molecules which are surface absorbent or entrapped in the large cavities of the polymeric framework [34]. Furthermore, the intention of these bands was greater in FTIR spectra of attributed higher degree of water molecules absorption in the geopolymer mass and synthesized with high water amounts which related to weakened mechanical strength as determined in the compressive strength results (Figure 1). The second region is located in between wavenumber 1300 cm^{-1} and 1600 cm^{-1} . Absorption bands at 1415 cm^{-1} to 1425 cm^{-1} are designed to the stretching vibrations of O-C-O bonds which exhibited the presence of sodium bicarbonate due to the atmospheric carbonation of the high alkali NaOH aqueous phase which is diffused on the GGBFS geopolymer material surface [35-36]. The third region of the FTIR spectra is located at wavenumber below 1300 cm^{-1} and includes the absorption bands near 950 cm^{-1} which assigned the Si-O-Al/Si-O-Si asymmetrical stretching vibrations. These major bands comprises the major fingerprints of the geopolymerization [37-38]. The transformation of six coordinated Al into other coordination during the hydration reaction was exhibited in the range of 770 cm^{-1} to 795 cm^{-1} [39-40].

4. Conclusions

The result exhibited that the compressive strength was affected by the NaOH concentrations. This parameter plays a role on the pattern of GGBFS strength, efficient as well as environmentally friendly and economical. The GGBFS geopolymers for varying NaOH concentration showed fluctuate of compressive strength. The compressive strength results displayed with an increase of NaOH concentrations up to 10M had a positive impact on the compressive strength. The presence of OH⁻, Na⁺ maximizes the dissolving ability of GGBFS particles and forms more reactive bonds which it boots the inter-molecular bonding strength of geopolymers. The contribution of Ca-O component within the geopolymer structure led to the formation of calcium silicate hydrate (CSH) and the hydration product of calcite (CaCO₃), thus enhances the mechanical properties and strengthen the structure.

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