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Characterization of the high-calcium fly ash geopolymer mortar with hot-weather curing systems for sustainable application

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ABSTRACT

Fly ash geopolymers are an alumino-silicate material and thus enable the utilization of waste containing alumino-silicate effectively. Geopolymeric reaction occurs as a result of the activation of fly ash with alkali solutions. In Thailand, a large amount of high-calcium fly ash is available due to the use of low-grade lignite coal feedstock for pulverized coal combustion process and the calcium content becomes very high. In this study, heat curing at 35 °C as a representative of a high ambient temperature (hot weather) and low cost was investigated. Curing at temperature of 65 °C and room temperature of 25 °C were also conducted to compare the results. Geopolymeric products were tested for compressive strength and characterized by XRD, IR, SEM and TGA techniques. The results showed that heat curing enhanced the geopolymerization resulting in the formation of Si—O—Al network product. Heat curings at 35 °C and 65 °C led to the formation of calcium silicate hydrate (C-S-H) and alumino-silicate (geopolymer bonding). Without heat curing, the product was predominantly C-S-H compound and the matrix was as strong as the heat-cured product. The immersion of samples in 3% sulfuric acid solution revealed that the performance of the heat-cured samples were better than those cured at room temperature. In addition, application of research results was to produce the geopolymer brick with outdoor heat exposure of 35 °C. Pedestrian pathway was demonstrated.

1. Introduction

Fly ash-based geopolymer is a green binder as the source materials are often from waste containing alumino-silicate. The carbon footprint of this material is, therefore, 9 times smaller than that of ordinary Portland cement [1]. Geopolymeric reaction occurs when alumino-silicate source materials (e.g. fly ash) react with base and sodium silicate solutions [2]. Sodium silicate solution provides the external dissolved SiO_2 to the system. NaOH solution is a common base and usually used to leach out the glassy phases of alumino-silicate source materials resulting in the gel formation [3,4]. A moderate temperature of 60–100 °C is applied to enhance the geopolymerization reaction [1,5,6]. Amorphous to semi-crystalline geopolymer products are formed by co-polymerisation of individual alumino-silicate species [7]. With heat curing, this material has desirable mechanical properties due to the amorphous alumino-silicate product with some semi-crystalline phases. Fast high strength gain can be achieved. In addition, geopolymer is more durable than OPC mortar under the acid and sulfate environment e.g. less loss of mass and decline in mechanical strength [6]. Owning to its good resistance to acid attack, it makes geopolymer very favorable to the sustainable development [8]. Generally, a temperature between 40 and 80 °C is used for the curing of fly ash geopolymer to obtain a strength gain up to 65 MPa within a short period [8]. However, application of heat curing requires additional effort and equipment in the construction site and thus hinders the extensively use of geopolymer as commercial building materials. Several attempts have been made successfully to produce the fly ash geopolymers with the reasonable strength (20–23 MPa at 28 days) at low curing temperature of 25 °C [9–11].

In Thailand, fly ash used in construction is mainly obtained from a pulverized coal-fired power plant in the North where lignite coal is the feed stock. The calcium content is rather high and increases with the increasing depth of coal mining. The high-calcium fly ash geopolymer consists of alumino-silicate product and C-S-H. C-S-H results from the reaction between calcium in fly ash and alkali solution in mixture leading to the strength development with curing age. High calcium content of fly ash in cement-based or geopolymeric composites, generally, results in unsoundness and

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can lead to crack formation [12,13]. In addition, rapid setting and low strength under acid environment of materials can be obtained [13]. Heat curing up to 80 °C helps to accelerate the geopolymerization reaction in order to gain high strength, particularly when high-calcium fly ash geopolymer has been used in acid environment as waste water pipe [14]. However, the production cost is higher for heat curing process that limits the manufacturing of heat-cured fly ash geopolymer products. Therefore, high-calcium fly ash is an interesting source material for geopolymer preparation at room temperature with a reasonable strength development owing to C-S-H formation [9,10]. Durability in acid environment should be focused and improved for products with this type of curing.

Many researchers have investigated the use of high-calcium fly ash in geopolymer [4,9,10,15–17]. It was reported that high-calcium fly ash has a potential as a source material for geopolymer synthesis. For the utilization of geopolymer in oil well cementing operations, the high early strength of high- calcium fly ash geopolymer was achieved with high pressure and high temperature of curing [16]. For normal heat curing of 65 °C, high-calcium fly ash geopolymer presented the high strength gain up to 70 MPa after curing in presence of alumino-silicate bond formation [4]. Due to the high calcium content of fly ash, this geopolymer could also set and harden at room temperature and strength was developed with time [9,10,15]. C-S-H was also formed in the composite owing to the reaction between calcium, amorphous silica in fly ash, and silica from water glass in the presence of base [15].

Thailand locates near the equator with hot weather all year round. Normal temperature during daytime is approximately 30 °C with high temperature up to 40 °C in the summer time. Researchers have reported that the use of 40 °C-temperature curing gives fly ash geopolymer with sufficient compressive strength for use as structural masonry units [18,19]. In order to utilize the hot climate to increase the alumino-silicate bond formation, this research, therefore, proposed the synthesis of high-calcium fly ash geopolymer with heat curing at 35 °C. Heat curing at 35 °C (representing a hot weather curing), normal curing temperature of 65 °C and room temperature curing of 25 °C were also tested for comparison.

2. Experimental program

2.1. Materials

In this research, lignite coal fly ash with high-calcium content was used as alumino-silicate source material for geopolymer preparation. With low grade of lignite coal, high calcium content was detected by X-ray fluorescence (XRF, PANalytical PW-2404) as shown in Table 1. The XRD pattern of this fly ash is presented in Fig. 1. High calcium content of 30.0 %wt was detected in the fly ash. In addition to calcium, quartz (SiO_2), aluminium oxide (Al_2O_3) and magnetite (Fe_3O_4) were the main compositions. It was classified as class C fly ash according to ASTM C618. The median particle size (D50) of fly ash as measured by particle size analyzer (Malvern Mastersizer S) was 9 μm. For alkali activators, sodium silicate solution (water glass, $Na_2O{\cdot}SiO_2$, 27 %wt SiO_2 and 8 %wt Na_2O) was used as external dissolved silica, and 8 M NaOH solution was used as a leaching solution. From preliminary study, it was found that high concentration of NaOH (more than 8 M)

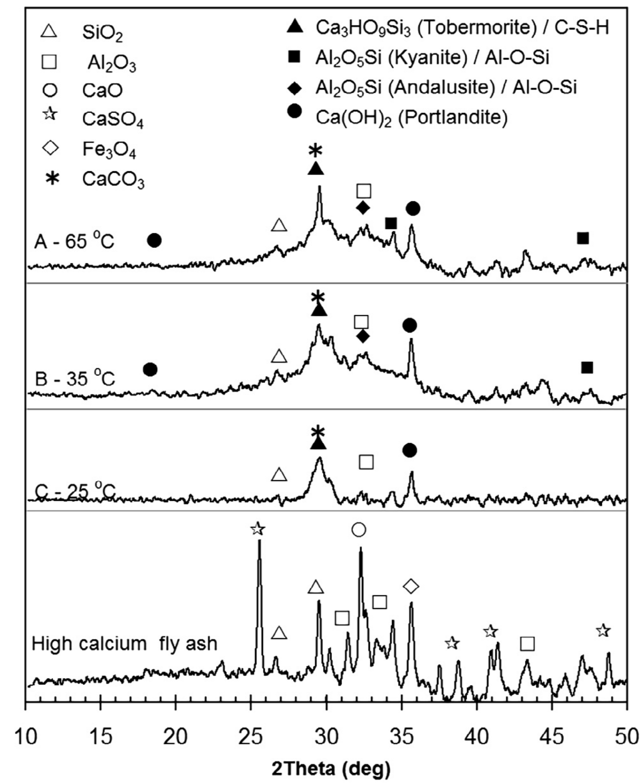

Fig. 1. XRD patterns of fly ash and composites.

resulted in low workability. Therefore, 8 M NaOH was used without extra water added to the mixture. Graded-fine river sand (passing sieve No. 30, 0.6 mm opening) with fineness modulus of 1.9 was used to prepare the geopolymer mortar for strength test.

2.2. Geopolymer preparation and test program

Geopolymer paste and mortar were prepared and tested. Paste composition was 55 %wt fly ash, 15 %wt 8 M NaOH and 30 %wt water glass. Mix proportions are showed in Table 2.

Molar ratios of paste mixture are following; $SiO_2/Al_2O_3 = 5.3$, $Na_2O/SiO_2 = 0.4$ and $H_2O/Na_2O = 11.9$ which is in the ranges recommended by Davidovit [2]. The high-calcium fly ash was thoroughly mixed with alkali activator (NaOH solution and water glass) for 1 min before molding in small plastic mold. Initial setting time was 8 min. Samples were covered with cling film to avoid the rapid water evaporation at the surface during the heat curing. Three curing conditions viz., 65 °C for 24 h (A: normal temperature curing), 35 °C for 72 h (B: representing a hot weather curing), and room temperature for 7 days (C: 25 °C controlled temperature) as shown in Table 3. Hot air oven was used in heating process of 65 °C and 35 °C. For 35 °C curing, 72-h or 3-day curing time was selected based on the calculation of equivalent heat transfer by conduction formula. Owing to the use of high-calcium fly ash, specimens hardened within 1 h after mixing. However, all samples were demolded after the temperature curing and the specimens were then kept in water (25 ± 2 °C) until the age of testing. The use of high-calcium fly ash resulted in the final products with the coexistence of

Table 1
Chemical compositions and LOI of high calcium fly ash.

Compositions (%)	SiO_2	Al_2O_3	CaO	Fe_3O_4	SO_3	MgO	K_2O	Na_2O	Others	LOI
Fly ash	25.0	13.2	30.0	15.0	7.1	2.1	1.8	1.4	1.1	3.4

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