



Influence of curing conditions on properties of high calcium fly ash geopolymer containing Portland cement as additive



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ABSTRACT

This paper investigated the mechanical properties and microstructure of high calcium fly ash geopolymer containing ordinary Portland cement (OPC) as additive with different curing conditions. Fly ash (FA) was replaced with OPC at dosages of 0%, 5%, 10%, and 15% by weight of binders. Setting time and microstructure of geopolymer pastes, and flow, compressive strength, porosity and water absorption of geopolymer mortars were studied. Three curing methods viz., vapour-proof membrane curing, wet curing and temperature curing were used. The results showed that the use of OPC as additive improved the properties of high calcium fly ash geopolymer. The strength increased due to the formation of additional C–S–H and C–A–S–H gel. Curing methods also significantly affected the properties of geopolymers with OPC. Vapour-proof membrane curing and water curing resulted in additional OPC hydration and led to higher compressive strength. The temperature curing resulted in a high early compressive strength development.

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1. Introduction

Clinker production process in the cement industry requires a lot of energy and emits a large amount of carbon dioxide (CO₂) to the atmosphere. The production of cement in 2005 accounted for around 7% of CO₂ world emission [1]. The negative environmental impact must be reduced by a reduction in the use of Portland cement. Fly ash, silica fumes and agro-waste ashes can be used as pozzolanic materials to reduce Portland cement usage and to curb emission of CO₂. Geopolymer is now receiving more attention as an alternative binder for use as building material. It is made from aluminosilicate source materials activated with high alkali solutions [2].

Fly ash is a by-product from coal burning to produce electricity. For Thailand, the major source of fly ash is from Mae Moh power station in the north. Approximately 3 million tons is produced annually and it is used extensively in the construction industry as supplementary cementitious materials to replace part of Portland cement. Many researchers have shown that it can be used for making good geopolymer [3–5].

At ambient temperature of around 25 °C, the fly ash geopolymer gains strength slowly [6]. To obtain reasonable strength fly ash geopolymers, temperature curing at 40–75 °C is normally required [7]. This is difficult for the construction practice in real construction. A number of researchers have, therefore, tried to improve the strength development of fly ash geopolymers. Some additives

such as ground granulated blast furnace slag, flue gas desulfurization gypsum and Portland cement are used [6,8,9].

The use of OPC to improve strength of fly ash geopolymer is very attractive as OPC is a common commodity in construction industry. The reaction of OPC and water is an exothermal process at normal temperature and the developed heat should be beneficial to the enhancement of mechanical properties of geopolymer. In this research, the mechanical properties and microstructure of geopolymer with various levels of OPC replacement and curing condition were studied. The knowledge should be useful in the understanding and future utilization of the high calcium fly ash geopolymer containing OPC as additive.

2. Experimental details

2.1. Materials

Fly ash (FA) from Mae Moh power plant in Lampang province in northern Thailand, and ordinary Portland cement (OPC) were the materials used. The Blaine finenesses of FA and OPC were 2460 and 3600 cm²/g. The alkaline solutions were 10 M sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) with 15.32% Na₂O, 32.87% SiO₂, and 51.81% H₂O. Local river sand with specific gravity of 2.62 and fineness modulus of 2.90 was used for making geopolymer mortar.

The chemical compositions of materials are given in Table 1. The FA consisted of 36.0% SiO₂, 14.9% Al₂O₃ and 19.7% Fe₂O₃, and the CaO content was high at 19.4%. The sum of SiO₂ + Al₂O₃ + Fe₂O₃

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Table 1
Chemical composition of FA and OPC.

Materials	Chemical composition (%)								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	LOI
FA	36.0	14.9	19.7	19.4	2.6	0.7	1.0	4.6	1.1
OPC	20.8	4.7	3.4	65.3	1.5	0.4	0.1	2.7	0.9

content of the FA were 70.6%. The high calcium content suggested that it was class C fly ash as specified by ASTM: C618.

2.2. Sample preparation

2.2.1. Mix proportion

Table 2 shows the mix proportion of the geopolymers. The mixes were named FA, 5PC, 10PC, and 15PC mixes with FA replaced by OPC at the dosages of 0%, 5%, 10%, and 15% by weight of binder, respectively. Constant liquid alkaline/binder ratio (L/B) of 0.40 and Na₂SiO₃/NaOH ratio of 0.67 were used for all mixes. The SiO₂/Al₂O₃ and CaO/SiO₂ ratios were computed and also presented in Table 2. For the geopolymer mortar mixes, sand/binder ratio of 2.75 was employed.

2.2.2. Mixing and curing method

For mixing of pastes, FA and OPC were thoroughly mixed until a uniform mixture was obtained which took approximately 2 min. For mortar mixes, sand was incorporated in the mixes with FA and OPC. Then NaOH was added and mixed for 5 min and Na₂SiO₃ was incorporated and mixed for another 5 min. The mixing was done in an air conditioned room with temperature around 25 °C.

After mixing, the fresh geopolymer pastes and mortars were placed into 50 × 50 × 50 mm cube moulds as described in ASTM: C109. The samples were covered with damp cloth and vinyl sheet and placed in 23 °C controlled room for a period of 24 h. They were then demoulded and put into different curing conditions as follows.

- (1) Vapour-proof membrane curing (MC). The demoulded samples were wrapped with a vapour-proof membrane to prevent moisture loss and kept in a control 23 °C room.
- (2) Temperature curing (TC). The samples were wrapped with a plastic membrane to prevent moisture loss and then oven-cured at 40 °C for 24 h. After that, the samples were demoulded and kept in a 23 °C room.
- (3) Wet curing (WC). The demoulded samples were immersed in water in a 23 °C room until the testing age.

2.3. Setting time and flow

The setting time of geopolymer pastes were tested using Vicat apparatus in accordance with ASTM: C191. The flow of fresh geopolymer mortars were tested in accordance with ASTM: C1437. The reported results were the average of two tests.

Table 2
Mix proportions of geopolymer.

Mixes	FA:OPC	Na ₂ SiO ₃ /NaOH	SiO ₂ /Al ₂ O ₃	CaO/SiO ₂
FA	100:0	0.67	2.77	0.47
5PC	95:5	0.67	2.80	0.54
10PC	90:10	0.67	2.83	0.61
15PC	85:15	0.67	2.86	0.69

2.4. Compressive strength, porosity and water absorption

Compressive strengths of geopolymer mortars were tested at the ages of 3, 7, 28, and 90 days in accordance with ASTM: C109. The reported results were the average of three samples.

Porosity and water absorption of geopolymer mortars were tested at the age of 7 days in accordance with ASTM: C642. The measured values were calculated using Eqs. (1) and (2). The reported results were the average of two samples. The methods for measuring porosity and absorption were used successfully in a number of researches [10,11].

$$\text{Porosity (\%)} = \left(\frac{W_a - W_d}{W_a - W_w} \right) \times 100 \quad (1)$$

$$\text{Absorption (\%)} = \left(\frac{W_a - W_d}{W_d} \right) \times 100 \quad (2)$$

where Porosity is the vacuum saturation porosity of geopolymer mortar (%), Absorption is water absorption of geopolymer mortar (%), W_a is weight in air of saturated sample (g), W_d is weight of dried sample (g), and W_w is the weight in water of saturated sample (g).

2.5. SEM and XRD analysis

Scanning electron microscopy (SEM) and X-ray Diffraction (XRD) were used to study the microstructure of geopolymer pastes. The XRD scans were performed between 5 and 70° 2θ, with an increment of 0.02 deg/step and a scan speed of 0.5 s/step. The amorphous structure in geopolymer was determined by quantitative XRD using Bruker's TOPAS software.

3. Results and discussions

3.1. Setting time and flow

The results of setting time of geopolymer paste and flow of mortar are shown in Figs. 1 and 2. The addition of OPC resulted in the reduction of setting time of pastes and flow of fresh mortars. For the 0%, 5%, 10% and 15% OPC mixes, the initial setting times were 124, 66, 39 and 28 min. while the final setting times were 144, 82, 53 and 47 min. respectively, and the flow values reduced from 136% to 111%. As the OPC contained a reasonably high CaO content (65.3%), the incorporation of OPC, therefore, resulted in the

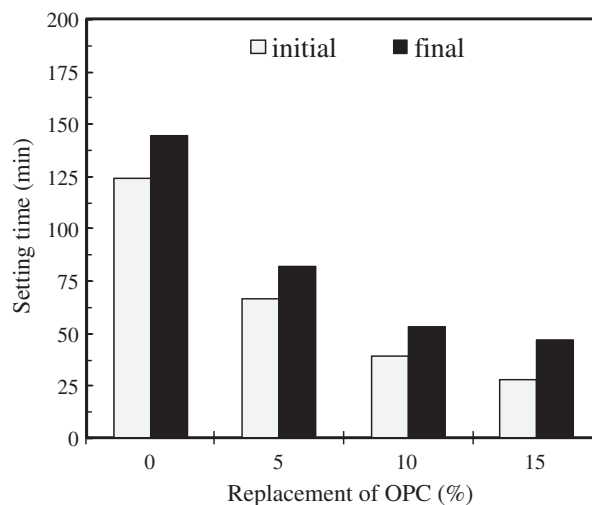


Fig. 1. Setting time of geopolymer paste.

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