Advanced Powder Technology 24 (2013) 703–707

Contents lists available at SciVerse ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

Role of microwave radiation in curing the fly ash geopolymer

Prinya Chindaprasirt [a], Ubolluk Rattanasak [b,*], Sompop Taebuanhuad [b]

[a] Sustainable Infrastructure Research and Development Center, Department of Civil Engineering, Faculty of Engineering, Khon Kaen University, Khon Kaen 40002, Thailand
[b] Department of Chemistry and Center for Innovation in Chemistry, Faculty of Science, Burapha University, Chonburi 20131, Thailand

ARTICLE INFO

Article history:
Received 14 August 2012
Received in revised form 19 November 2012
Accepted 27 December 2012
Available online 10 January 2013

Keywords:
Geopolymer
Microwave radiation
Microstructure
Compressive strength

ABSTRACT

Fly ash geopolymer requires rather long heat curing to obtain reasonable strength development at an early age. However, the long heat curing period limits the application of the fly ash geopolymer. High strength development and a reduction in heat curing duration have been considered for energy saving. Therefore, this research proposed a process using 90-W microwave radiation for 5 min followed by conventional heat curing for high-calcium fly ash geopolymer. Results showed that the compressive strengths of geopolymer with microwave radiation followed by conventional heat curing were comparable to those of the control cured at 65 °C for 24 h. Microwave radiation gave the enhanced densification. In addition, SEM images showed that the gels formed on the fly ash particles owing to the promoted dissolution of amorphous phases from fly ash. This method accelerated the geopolymerization and gave the high compressive strength comparable to the conventional curing.

1. Introduction

Coal-fired power plants currently produce 40% of the global electricity for households and industries. Besides generating power energy, coal wastes, i.e. fly ash and bottom ash are obtained and utilized related to the environmental aspect [1,2]. In addition to the use of fly ash to partially replace Portland cement, the high content fly ash usage has been introduced and termed "geopolymer" which is a low heat cured alumino-silicate material [3]. Owing to the endothermic reaction, heat curing at 65 °C was employed in order to gain high early strength of lignite fly ash geopolymers with good physical and mechanical properties [4,5]. However, the 24-h or longer heat curing periods limit the application of geopolymer. Although the room temperature curing has been considered for energy saving, the prolonged curing duration was required to obtain reasonable strength for lignite fly ash geopolymer [6].

Heat curing has been applied to construction materials especially for the precast concrete to improve the strength development process. This concrete attains sufficient strength in short curing time, so the moulds can be reused, and the final products can be rapidly delivered to the site [7]. For the conventional heating technique, heat is distributed in the specimen from the exterior to the interior leading to the non-uniform and long heating period to attain the required temperature. In contrast, the microwave technique allows a uniform and fast heating due to the interaction between the polar molecules and microwave electric fields [7–11]. Application of microwave to the fresh concrete results in removal of water, collapse of capillary pore and densification of sample. However, microwave radiation is mostly used in concrete curing and the radiation period is usually long (more than 1 h with high wattage) [7,8]. This could limit the use of microwave curing due to the energy cost.

Therefore, this research proposed the process to reduce the microwave curing duration and energy using the short-time microwave radiation in addition to the convention heat curing for the fly ash geopolymer. Microwave radiation could play the role in the geopolymer formation and strength enhancement. Additionally, this method could shorten the curing time of geopolymer, accelerate the geopolymerization and give the high compressive strength at an early age compared to the conventional curing.

2. Experimental procedure

2.1. Materials

Coal fly ash from Mae Moh power plant in the north of Thailand was used as raw material. This fly ash was generated from the pulverized coal combustion process (1200 °C) using lignite coal as the feed. The properties of this fly ash are tabulated in Table 1. In addition to Al_2O_3 and SiO_2, this fly ash had high contents of CaO and magnetite (Fe_3O_4). Sodium hydroxide pellet (NaOH) dissolved in the deionized water to obtain 10 M NaOH solution, and sodium silicate solution (Na_2SiO_3) with SiO_2:Na_2O mass ratio of 3.2 was also used. The viscosities of 10 M NaOH and Na_2SiO_3 were 9.3, and 60.6 cps (centipoises), respectively. The graded river sand with fineness

* Corresponding author. Tel.: +66 38 103066; fax: +66 38 393494.
E-mail address: ubolluk@buu.ac.th (U. Rattanasak).

0921-8831/$ - see front matter © 2012 The Society of Powder Technology Japan. Published by Elsevier B.V. and The Society of Powder Technology Japan. All rights reserved.
http://dx.doi.org/10.1016/j.apt.2012.12.005

Table 1
Properties of fly ash.

Chemical composition (wt%)	SiO_2	39.2
	Al_2O_3	19.7
	CaO	16.9
	Fe_3O_4	12.1
	SO_3	2.8
	Others	9.3
Median particle size (μm)		19.0

modulus of 2.8 and specific gravity of 2.65 was employed to prepare the mortar specimen for strength test.

2.2. Geopolymer preparation

To prepare the liquor, 10 M NaOH and Na_2SiO_3 were mixed in a container with the Na_2SiO_3/NaOH mass ratio of 1.5. The liquor was then added to the fly ash in a pan mixer and the paste was thoroughly mixed. The fly ash-to-liquor mass ratio of 1.86 (fly ash = 65 wt% and liquor = 35 wt%). The paste was continuously mixed for 5 min and then cast into 50-mm cubic acrylic mould (1-cm thickness). The paste specimens were vibrated for 10 s and covered with cling film to avoid the moisture evaporation during the heat curing.

For geopolymer mortar preparation, sand was added to the paste mixture with the sand-to-fly ash mass ratio of 1.5 and mixed for another 2 min. The mixture was then cast into 50-mm cubic acrylic mould. The curing condition was the same as for the paste preparation. Compressive strength test was performed on the geopolymer mortars.

2.3. Microwave and conventional curing temperature profile

In this study, the 2.45-GHz household microwave and the conventional oven were employed. Owing to preliminary results, 90- and 180-W microwave power levels were applied on the geopolymer mortars for 3, 5 and 10 min to obtain the temperature profile at the middle of the samples. Higher wattage resulted in rapid evaporation in short time, heat evolution and cracks on the surface of specimens. Result is reported as an average of five samples.

In addition, the effect of microwave and conventional curing on the compressive strength of geopolymer mortar was evaluated. 90-W microwave curing for 5 min was selected in addition to the convention heat curing at 65 °C for 3, 6 and 12 h. After heat curing, specimens were cooled down and cured continuously at 25 °C. The compressive strength was tested at the age of 7 days. The reported results were the averages of five samples. Table 2 shows the curing conditions. Curing at 65 °C for 24 h (system 1) was the control.

2.4. XRD, SEM and degree of reaction

Geopolymer paste was prepared for the testing of XRD, SEM and degree of reaction. 90-W microwave radiation for 5 min in addition to the conventional heat curing at 65 °C for 3, 6 and 12 h was used. XRD and SEM analyses were performed on the hardened samples. In addition, degree of reaction was determined both on geopolymer paste and fly ash by identification of unreacted fly ash in specimen using the dissolution of the powdery sample in 2 M HCl and 5 wt% Na_2CO_3 [12–14]. The hardened geopolymer pastes were ground to obtain particles that passed a 150-μm sieve. A 100-mL beaker filled with powdered samples (5 g) and 2 M HCl (30 mL) was placed in a 60 °C water bath and stirred for 20 min to accelerate the dissolution. Solid phase was then filtered using a vacuum filter. The remaining solid was washed with warm water thrice to completely remove HCl. Acetone was applied in the last filtration to remove water before drying at 70 °C for 2 h. Degree of reaction was calculated using Eq. (1). The degree of reaction of the fly ash particles was also determined and assigned as "blank." All the results were subtracted with blank to obtain the corrected degree of reaction. The reported results were the averages of three samples.

$$\text{Degree of reaction} = \frac{m_{\text{sample}} - m_{\text{residue}}}{m_{\text{sample}}} \times 100 \quad (1)$$

where m_{sample} is the weight of powdery sample (g) and m_{residue} is the weight of dried residue (g).

3. Results and discussion

3.1. Temperature profile of microwave curing

The temperature profiles of the 50-mm cube geopolymer mortar are reported in Fig. 1. Application of 90- and 180-W microwave power levels generated high temperature in the specimen in a short period of time. At 5 min microwave radiation, 90- and 180-W gave slightly different temperatures. For 10-min microwave radiation, the temperature difference was noticeable. Increase in the microwave radiation time led to high temperature, however, longer radiation time and increase in wattage resulted in rapid evaporation, high heat evolution and sequential cracks on the surface of specimens owing to the energy beyond the requirement for water removal in materials [15]. For this reason, 90-W microwave radiation for 5 min was selected for further study.

3.2. Effect of microwave and conventional curing on the compressive strength

To study the effect of curing conditions on the properties of geopolymer mortars, the compressive strength was primarily focused in this section. Table 3 presents the strengths of the specimens

Table 2
Curing conditions of geopolymer.

System	Curing conditions	Note
1	65 °C oven curing for 1, 3, 6, 12 and 24 h	65 °C oven curing
2	65 °C oven curing for 1, 3, 6, 12 and 24 h → 90 W MW curing for 3 min	65 °C oven curing before MW radiation
3	90 W MW curing for 3 min	MW radiation only
4	90 W MW curing for 5 min	MW radiation only
5	90 W MW curing for 10 min	MW radiation only
6	90 W MW curing for 3 min → 65 °C oven curing for 1, 3, 6, 12 and 24 h	MW radiation before 65 °C oven curing
7	90 W MW curing for 5 min → 65 °C oven curing for 1, 3, 6 and 12 h	MW radiation before 65 °C oven curing
8	90 W MW curing for 10 min → 65 °C oven curing for 1, 3, 6 and 12 h	MW radiation before 65 °C oven curing
9	25 °C curing for 7 and 28 days (room temp., RT)	No heat curing