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Effect of free lime content on properties of cement–fly ash mixtures

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HIGHLIGHTS

- The free lime particles have irregular shape and are distributed outside the fly ash particles.
- Higher free lime in fly ash leads to early setting and higher compressive strength especially at early age.
- The autoclave expansion and the expansion due to alkali-aggregate reaction increases as free lime in fly ash increases.
- Carbonation depth, chloride and sulfate resistances are only slightly affected by the content of free lime in fly ash.
- It is possible to utilize the tested fly ash with free lime content up to 4.51% as mineral admixture in concrete.

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ABSTRACT

This research aims to clarify the limitation of free lime content in fly ash according to EN-450. The form and existence of free lime in fly ash by SEM, EDX, and free lime content by a titration method, were observed. Some basic properties of fly ashes with various free lime contents and durability properties of mortar incorporating the fly ashes were also preliminary studied. It was found that free lime particles were mostly distributed outside the fly ash particles and that a minor amount of free lime was encapsulated inside the fly ash particles in the fly ash formation process, meaning that the originally high free lime and added free lime similarly affects properties of fly ash mixtures. A free lime addition method was selected by adding free lime to the originally collected fly ash in order to vary free lime content of the fly ash. The studied basic properties consisted of normal consistency, autoclave expansion, initial and final setting time, water requirement, and compressive strength. In terms of durability, alkali-aggregate reaction, carbonation, shrinkage, and chloride and sulfate resistances, were investigated. Test results revealed that physical properties such as normal consistency and water requirement were unaffected by free lime, and that a free lime content up to 4.51% had only slight chemical effects on fly ash–cement mixtures, i.e., faster setting, higher compressive strength, especially at early age, and higher autoclave expansion. However, the values of autoclave expansion were still within the standard limit. Regarding durability, mixtures with the high free lime fly ash led to higher expansion due to alkali-aggregate reaction. The expansion was smaller than that of a cement-only mixture. Carbonation depth, shrinkage, and chloride and sulfate resistances were only slightly affected by the added free lime.

1. Introduction

Fly ash, a by-product from electricity generating power plants, has been widely used in the concrete industry. Since it is not an industrial product, its chemical and mineralogical compositions vary greatly depending on many factors such as the type of coal and burning conditions. Other countries' standards [1–3] cannot be directly applied because of differences in characteristics and properties of fly ashes produced in different countries.

Much research has been conducted in Thailand to develop fly ash classification and standards for the country, i.e., TIS 2135 [4]. Recently, fly ash produced from Mae Moh electricity generating power plant started to show signs of high free lime content. Too

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high free lime content fly ash may affect both mechanical properties and durability of concrete [5,6]. Antiohos and Tsimas [7] reported that unusually high percentages of free lime and sulfur trioxide are concentrated in the smaller size particles of fly ash. The behaviors of concrete with high free lime content fly ash in Thailand have not been sufficiently studied. With reference to ASTM C618 [2], no limit is set for either total calcium oxide content or free lime content of fly ash. Nonetheless, EN-450 [1] limits the amount of free lime content of fly ash to 1%, or up to 2.5% if the result of an autoclave expansion test is satisfied.

This research was initiated to provide understanding and preliminary information on the effect of free lime content of fly ash in Thailand on properties of concrete. The research consists of two parts: (i) clarifying the form and existence of free lime in fly ash and (ii) studying properties of cement–fly ash mixtures with different free lime content. To eliminate effects of physical properties of the fly ash and focus only on the effects of free lime, the addition of free lime to originally collected fly ash was done in order to vary free lime content.

2. Experiment

2.1. Form and existence of free lime in fly ash

Microscopic study of Mae Moh fly ash content was carried out by SEM (Scanning Electron Microscope), EDX (Energy-dispersive X-ray), whereas free lime content was determined by a titration method.

The SEM picture of Mae Moh fly ash with high free lime content, illustrated in Fig. 1, shows that there are two types of particle shape, i.e., round (#1) and irregular (#2). It is generally known that Mae Moh fly ash particles are spherical. Therefore, there is no doubt that the round particles are fly ash. The irregular particles, often found in Mae Moh fly ash with high free lime content, are assumed to be free lime. To prove this hypothesis, it is necessary to determine the composition of the particles. The EDX technique was implemented on the selected areas of particles #1 and #2 and the results are shown in Figs. 2 and 3, respectively. The main composition of particle #1 is silica, which is common for fly ash. Unlike particle #1, particle #2 indicates a calcium rich particle. To reaffirm the outcome, an SEM picture of Mae Moh fly ash (which is the oven-dried filtered residue of fly ash in the process of free lime determination) was taken as depicted in Fig. 4. The round shape particles are obviously observed while the irregular shaped particles, presumed to be free lime, are absent. This indicated that free lime particles were washed out in the free lime determination process. According to SEM and EDX techniques, it is concluded that particles of free lime in Mae Moh fly ash have irregular shapes and are distributed among the fly ash particles.

However, free lime may also be encapsulated inside fly ash particle during the fly ash formation process. To quantitatively determine this amount of free lime, original Mae Moh fly ash (MM) was ground into two different finenesses, referred to as MM1 and MM2. Free lime contents of all fly ashes were determined by a titration method. Initially, 1 gram of fly ash was dissolved in 50 cm^3 of ethylene glycol at a temperature of 60–70 °C for 30 min. The solution was then filtered by #1 filter paper. In this process, another 30 cm^3 of ethylene glycol at the temperature of 60–70 °C was added to make sure that all particles were washed out. The filtrate was titrated with 0.1 N hydrochloric acid using Bromocresol green as an indicator while the residues on filter paper were collected and dried in an oven. These residues were later sampled and used for the second test of free lime determination. The free lime content was computed from:

$$\text{Free lime content } (\%) = (M_{fl}/M_{fa}) \times 100 \quad (1)$$

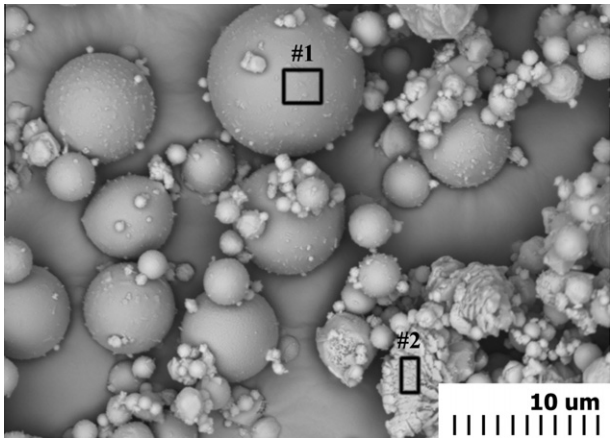

Fig. 1. SEM image of Mae Moh fly ash (particles #1 and #2).

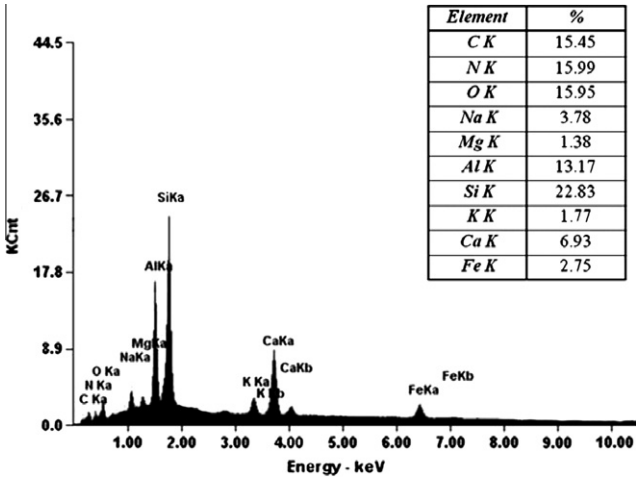

Fig. 2. EDX of particle #1.

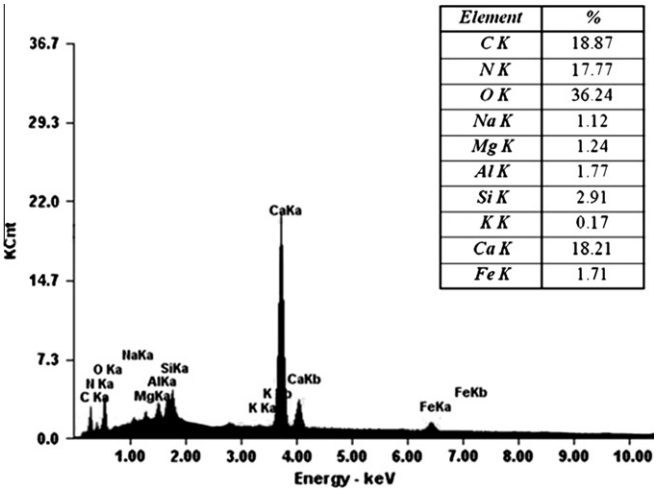

Fig. 3. EDX of particle #2.

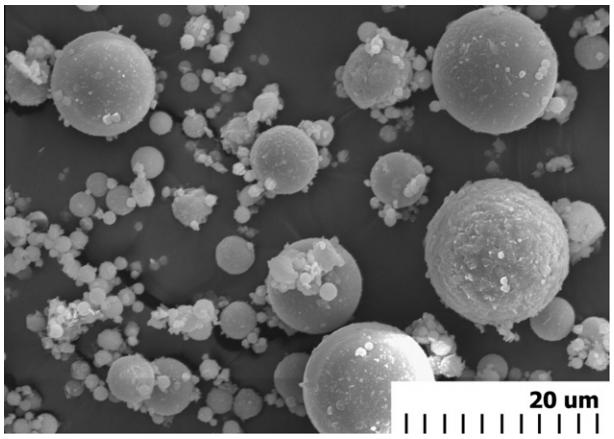

Fig. 4. SEM image of oven-dried filtered Mae Moh fly ash.