



Heat of hydration of Portland high-calcium fly ash cement incorporating limestone powder: Effect of limestone particle size



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HIGHLIGHTS

- Ternary blended cement of high-calcium fly ash and limestone powder were studied.
- High-calcium fly ash and limestone powder without PC was also studied.
- Limestone powder of different particle size is reported.
- Compressive strength, XRD, TG and heat of the paste mixes are reported.
- Accelerated heat of hydration was found at early age with finer particle size.

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ABSTRACT

This study investigated the limestone powder (LS) particle size effect on the heat of hydration of Portland cement (PC) and high-calcium fly ash (FA) systems. The LS used has a maximum particle size of 5 and 20 μm , respectively. Isothermal calorimetry was used to measure the heat evolution during the hydration process of the cement and PC–FA pastes. The results show the particle size of LS had a significant influence on the observed heat of hydration both on rate of reaction and total amount of heat release. The hydration rates of both PC–LS and PC–FA–LS systems were accelerated by 5 μm LS, while 20 μm LS did not. For PC–FA–LS system, the total heat release at 48 h when using 5 μm LS were higher than FA at 30% replacement level whereas 20 μm LS showed similar total heat release. However, the total heat release of these ternary mixtures with both LS particle sizes was still lower than PC control. X-ray diffraction (XRD) technique and thermogravimetric (TG) analysis were applied to identify the hydration products. XRD results show the formation of carboaluminates and ettringite was still found at 28 days, indicated the stabilization of ettringite when both LS particle size were used. The reaction between LS and PC or FA was confirmed by TG results in the reduction of CaCO_3 content.

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1. Introduction

Fly ash is a by-product generated from burning coal in the generation of electricity. Two classes of fly ash are defined by ASTM C618 [1] that is low-calcium Class F fly ash and high-calcium Class C fly ash. The main difference between these classes is the amount of calcium, silica, alumina, and iron content in the ash and the type of coal used. High-calcium fly ash is widely used as a cementitious material since it has a high lime (CaO) content and pozzolanic activity [2,3]. It should be noted that high-calcium fly ash would provide more rapid strength gain than lower calcium fly ash [4]. The use of fly ash in cement and concrete still continue to increase

due to it can replace part of Portland cement without adversely affecting concrete properties. However, one issue to consider when using fly ash as cement replacement is that early age performance (i.e. strength development, heat of hydration, setting time) may be delayed [5,6], especially at high-volume replacement.

Limestone is used in cement and concrete for various purposes, namely, as a raw material for clinker production and as coarse or fine aggregate. Moreover, limestone powder (LS) has been suggested for use as an additive or replacing part in Portland cement to improve the early ages strength, workability and stability of fresh concrete [7,8]. Generally, LS used with Portland cement is assumed as an inert filler which produce mainly physical effects in the cement pastes known as the filler effect. However, LS has also been reported to have a chemical effect, in which carboaluminate hydrate is formed during the hydration of Portland limestone

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cement. In normal Portland cement hydration, the dissolved sulfate ions will react with tricalcium aluminate ($3\text{CaO}\cdot\text{Al}_2\text{O}_3$, C_3A) and/or tetracalcium aluminoferrite ($4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{Fe}_2\text{O}_3$, C_4AF) forming ettringite ($6\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{SO}_3\cdot 32\text{H}_2\text{O}$, $\text{C}_6\text{A}_3\bar{\text{S}}_3\text{H}_{32}$) and the remaining C_3A and C_4AF can react with the ettringite to form monosulfoaluminate ($4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SO}_3\cdot 12\text{H}_2\text{O}$, $\text{C}_4\text{A}\bar{\text{S}}\text{H}_{12}$). In the presence of LS, the carbonate ion from limestone powder can interact with the aluminate hydrate from Portland cement hydration to form carboaluminates [9–11]. This leads to the stabilization of ettringite and results in an increase of the total volume of hydration products [12,13]. However, this chemical interaction of limestone does not have a significant effect on properties of Portland limestone cement especially at high level limestone content due to the limited aluminate phase in Portland cement. Thus, additional aluminate content obtained from supplementary cementitious materials such as fly ash, slag or metakaolin may be required.

The use of limestone powder incorporating with supplementary cementitious materials, especially fly ash in Portland cement has attracted great interest in recent years. De Weerd et al. [13] investigated the heat of hydration of ternary Portland cements containing limestone powder with a median particle size of $5\ \mu\text{m}$ and siliceous fly ash ($\text{CaO} = 6.3\%$). They found that the cement paste with a minor limestone powder replacement at 5% had similar rate of heat of hydration compared to PC control. Whereas, Vance et al. [14] found that fine limestone powder with median particle size of 0.7 and $3\ \mu\text{m}$ accelerated the early age hydration of both PC–LS and PC–FA–LS mixes, but similar rate of heat of hydration to PC control was observed when using $15\ \mu\text{m}$. The reported fly ash used in those previous studies [13,14] is low-calcium fly ash, however, the by-product of coal burning from Mae Moh power plant in Northern Thailand used in this study is a high-calcium fly ash. Additional use of LS as a partial cement replacement has economical benefits and provides interesting alternative as the LS was found to interact with PC and FA. Therefore, this research work aimed to investigate the reaction of LS in the Portland high-calcium fly ash cement in terms of its effect on heat of hydration when using different particle size of LS ($5\ \mu\text{m}$ and $20\ \mu\text{m}$). This is done by using the isothermal calorimetry, thus giving the rate of heat of hydration and total heat release during the first 48 h. In addition, the hydration products and other phases in both the binary (PC–LS) and ternary (PC–FA–LS) mixtures were characterized by using X-ray diffraction and thermogravimetric analysis. Moreover, the reaction between high-calcium fly ash and limestone powder (FA–LS) was also verified using different LS particle sizes.

2. Materials and methods

2.1. Materials

The mixtures were prepared using ordinary Portland cement Type I (PC) complying with ASTM C150. Class C fly ash (FA) was obtained from Mae Moh coal fired power plant in Lampang province, Thailand. Limestone powder (LS) with two different particle sizes ($5\ \mu\text{m}$ max. and $20\ \mu\text{m}$ max.) was supplied by the Siam Research and Innovation Co., Ltd. (Thailand). It is recognized that the identification of very small sizes of limestone powder is difficult due to the agglomeration of the particles. Thus, the microscopy technique was employed to determine the true particle sizes of the limestone powder used in this study. The morphologies of limestone powder were examined using low vacuum scanning electron microscopy (SEM, JEOL JSM-5910LV) at a magnification of $3000\times$ (Fig. 1). The SEM observations revealed that the LS powder was of an angular shape and with a maximum particle sizes of $5\ \mu\text{m}$ and $20\ \mu\text{m}$ as shown in Fig. 1a and b, respectively. The chemical compositions determined by X-ray fluorescence technique (XRF) and the specific gravity of Portland cement, fly ash and limestone powder are given in Table 1.

The percentages of fly ash or limestone powder used to replace part of Portland cement in the binary mixtures were 0–30% by weight. Ternary mixtures were produced by using fly ash in combination with limestone powder as a partial cement replacement at 30 wt.%. Mixtures of high-calcium fly ash and limestone powder (without PC) were also prepared by replacing part of fly ash with limestone powder. The mix proportions of the binary and the ternary mixtures prepared in this studies are given in Table 2.

2.2. Methods

The heat evolution of all the pastes were measured by isothermal conduction calorimeter (I-Cal 4000), using a constant water to binder (w/b) ratio of 0.5. The mixtures were mixed by hand for 30 s and about 25 g of the paste was carefully weigh out and transferred to testing cups. The cups with of the pastes were then covered with plastic lids and quickly placed into the calorimeter. All measurements were performed during the first 48 h of hydration at a control temperature of $20\ ^\circ\text{C}$.

The specimens for compressive strength test were prepared using a w/b ratio of 0.5. The mixtures were mixed and cast into 50 mm cube steel molds. After casting, the molds containing the specimens were covered with a plastic sheet and stored in the laboratory environment ($23\ ^\circ\text{C}$ and 70 RH) for 24 h. The paste specimens were then demoulded and immersed in saturated lime water ($23 \pm 2\ ^\circ\text{C}$). The compressive strength was determined using a Denison compression machine with a loading capacity of 3000 kN. The loading rates applied during the compression tests was $0.6\ \text{MPa/s}$. For each mixture, the compressive strength was measured at the age of 1 (or 3) and 28 days. The reported values are the average of three specimens. Small fractured pieces from the tested specimens were collected for XRD and TG tests.

The X-ray diffraction (XRD) tests were conducted on the 28 days pastes. The collected small pieces of pastes were first immersed in acetone to stop the hydration. The specimens were then dried in oven at $60\ ^\circ\text{C}$ and ground to fine powder. XRD measurements were performed using a diffractometer (Rikagu SmartLab 9 kW) equipped using K α -filtered $\text{CuK}\alpha$ radiation and operating at 40 kV and 20 mA. Step scanning was made from 5° to 65° using a step interval of 0.02° and a scan speed of $5^\circ/\text{min}$.

Thermogravimetric (TG) and derivative thermogravimetric (DTG) analysis were performed on the finely ground pastes at the age of 28 days by using Netzch STA 449C, Jupiter. The samples were heated over a range of $30\text{--}1000\ ^\circ\text{C}$, at a constant rate of $10\ ^\circ\text{C}/\text{min}$ in a nitrogen atmosphere.

3. Results and discussion

3.1. Calorimetry

The isothermal calorimetry heat profiles show the heat of hydration per gram of the binder (PC/FA/LS) evolved during the first 48 h of hydration of PC–FA, PC–LS, FA–LS and PC–FA–LS pastes at a constant w/b ratio of 0.5 (Figs. 2–8). The profile of the plain PC (100PC) is given as a reference in each system. Fig. 2 shows that the shape of the calorimetry heat profile of PC–FA pastes were similar to that of 100PC. The replacement of PC by fly ash results in a delay of the hydration heat development compared to plain PC. Moreover, a reduction in the total released heat with the increasing FA content has been observed, probably due to the slow reaction of fly ash and the cement dilution effect. This is in agreement with the results reported by Rahhal and Talero [15] and Nocuñ-Wczelik [16] where the heat evolution rate decreases with the increase of the fly ash content because of the lower rate of pozzolanic reaction.

The calorimetry heat profiles of PC–LS pastes using different LS particle sizes of 5 and $20\ \mu\text{m}$ are shown in Figs. 3 and 4, respectively. In the presence of limestone, the calorimetry heat profile indicates two humps of the main hydration peak. The first hump is caused by the hydration of calcium silicates, mainly tricalcium silicates (C_3S) [17]. The second hump, on the back of the silicate hydration, corresponding to renewed reaction of the aluminate phases [18]. As shown in Fig. 3a, the heat of hydration occurs earlier for all pastes containing $5\ \mu\text{m}$ LS in comparison with the plain PC. For the 30LS sample, the maximum heat of hydration was observed around 6.5 h and for plain PC at 10 h, indicating an acceleration of the cement hydration in the presence of finely ground limestone, which conformed to the observed results by Lothenbach et al. [19]. Moreover, the total heat released during 48 h of pastes containing $5\ \mu\text{m}$ LS up to 10 wt.% is higher than that of plain PC illustrating again the accelerating effect of limestone (Fig. 3b). This might have occurred due to the formation of carboaluminates during the hydration of Portland limestone cement [7]. However, carboaluminates which appeared within 48 h may be formed as hemicarboaluminates. Ipavec et al. [20] reported that carboaluminate phases in Portland cement–calcite system always form from hemicarboaluminate and is converted with time into

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