



Efficiency of metakaolin in steam cured high strength concrete



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HIGHLIGHTS

- The effect of metakaolin on the steam cured high strength concrete is investigated.
- The effect of incorporation of aluminium on the microstructure of C-S-H is discussed.
- The metakaolin participates the formation of C-(A)S-H gels.
- The metakaolin hinders deleterious effects of heat treatment on concrete.

ARTICLE INFO

Article history:

Received 23 April 2016

Received in revised form 22 May 2017

Accepted 2 July 2017

Keywords:

Metakaolin

High strength concrete

Microstructure

Volume stability

C-S-H gel

ABSTRACT

This paper presents a study on investigating the effect of metakaolin (MK) on the hydration, microstructure and volume stability of steam cured high strength concrete (HSC) with a low water to binder ratio (w/b) of 0.25 cured at 80 °C. The structure of C-A-S-H series with a Ca/Si ratio of 1.0 and Al/Si ratio varying from 0 to 0.3 are investigated to discuss the effect of aluminium on the microstructure of C-S-H gel. The experimental results show that the hydration of cement is accelerated due to the presence of MK. The Ca (OH)₂ is consumed and aluminium substituting silicon in the C-(A)S-H chains can be obtained, which leads to high strength, low average pore diameter and low total porosity of HSC. The cracks caused by steam curing tend to be moderated after adding MK, and no visible interfacial transition zones (ITZ) between aggregate and matrix are observed. The MK decreases the volume expansion of steam cured HSC caused by heat treatment as well as the drying shrinkage, leading to a better volume stability. Moreover, incorporation of aluminium results in modification of the layer stacking of C-(A)S-H and reduction of its porosity, which will support the improvement of strength, microstructure and volume stability of steam cured HSC.

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1. Introduction

Considering the advantages such as the reduction of construction time, low environmental impacts [1] and constant supply of precast elements [2], the precast high strength concrete pipe piles (HSCP) have been widely used for geotechnical engineer during the past decades. However, the high early strength of HSCP is ensured by heat treatment (steam curing & autoclaved-curing), high proportion of cement and high silica fume content. And widely use of CEM 52.5 cement with high clinker content commonly leads to environmental problems. Because the manufacture of Portland cement clinker produces large amount of CO₂ [3]. Also, a large amount of energy is consumed during the two-step heat treatment

of HSCP, especially for autoclaved curing. Hence, there is an issue that associates with reduction the energy consumption and CO₂ emission for HSCP industry.

Considering the performance of HSCP, methods for decreasing the energy consumption and CO₂ emission of HSCP have been proposed, the most typical ones are replacement of cement by supplementary materials, such as GGBS, fly ash, MK and limestone powder [4,5]. The other one is the application of energy-saving curing process [6].

Among these supplementary materials, concrete with MK has potential to achieve high strength and durability on the same magnitude to that of silica fume. Moreover, it has a larger particle size than that of silica fume which leads to less water demand, good workability and less plastic cracking [7]. In addition, the cost of silica fume has been high since its demand constantly rising [8]. Then, MK can be considered as one of most quality enhancing additives for high-strength and high-performance concrete.

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Additionally, MK is generally obtained by calcining kaolinite clay at 500–800 °C [9], where conversion from kaolinite to amorphous mineral phase occurs and becomes pozzolanic [10–12]. It mainly reacts with calcium hydroxide dissolved from cement during hydration. And the additional C-S-H gels, together with crystalline products are formed, which depend principally on the MK/CH ratio and curing temperature [13,14]. In previous researches, the positive effects of MK on the properties of concrete have been investigated. These researches showed that MK could significantly improve the mechanical strength [15], pore structure [16,17], resistance to chemical attack [9], and durability [18–20] of concrete.

For precast concrete, like HSCP, in order to reduce production cycle and acquire high strength as fast as possible, curing temperature higher than 70 °C is usually used. But, concrete cured at 60 °C has high volume of pores that is larger than 150 nm in diameter [21], which it is larger than that of concrete cured at room temperature, and further influences the durability of concrete. Then, for HSC cured at high temperature (>70 °C), it is necessary to offset the increasing volume of the harmful pores. In addition, concrete cured at high temperature, usually above 70 °C, shows significant expansion in the later age [22,23]. The expansion would lead to cracking of concrete, and further affects its mechanical properties, microstructure and durability. This kind of damage is related to delayed ettringite formation [24]. Hence, the volume of steam cured (especially for >70 °C) concrete is usually significantly influenced by temperature, and this change of volume can lead to cracks in cement paste. In addition, reduction of the long-term strength and durability would be found [25]. MK is considered as a effective material for controlling expansion, because it contains large amounts of reactive Al_2O_3 [26]. Many studies are mainly focus on the shrinkage of cement or concrete containing MK curing at normal temperature. Test results show that the early age autogenous shrinkage can be reduced with the inclusion of MK content, and the long-term autogenous shrinkage increases with the increasing MK replacement level [27,28]. And, the drying shrinkage is also remarkably reduced [29]. However, the effect of MK on the autogenous shrinkage or expansion of concrete curing at high temperature (>60 °C) is rarely reported, especially for the shrinkage under steam curing condition.

Usually, the high strength of HSCP is obtained through applying low water to binder (w/b) ratio and heat treatment. The MK is also a solution for the HSCP to limit the clinker content in concrete. A number of researchers have reported performance of concrete containing MK at low w/b ratio. Guang Jiang et al. found that the pozzolanic reactivity of MK is higher than that of silica fume at w/b ratio of 0.17 [30]. While, performance of MK in concrete with low W/B ratio of 0.3 is similar to that of higher water to binder ratios is also reported [31]. C.-S. Poon et al. found that the rate of

pozzolanic reaction of MK blended cement paste (w/b = 0.3) is higher than that of silica fume and fly ash blended cement paste at an early age [17]. Moreover, many studies [5,32,33] have demonstrated that performance of concrete containing MK is better than that of reference without MK under steam-cured conditions (50–70 °C). The improvement can be explained by the occurrence of the pozzolanic reaction of MK and thermo-activated under steam curing conditions [32]. Unfortunately, there is limited study that has evaluated the performance of concrete containing MK under a very low w/b ratio (<0.3) cured at high temperature (>70 °C).

Consequently, based on these premises, the objective of this research is to investigate the effects of MK on the hydration, microstructure and volume stability of steam cured HSC. The HSC is obtained by adding MK and cured at 80 °C. Properties of HSC are quantified and compared with those of references. In order to identify the effect of MK on microstructure of HSC, the C-S-H and C-A-S-H are synthesized and used to discuss the effects of aluminium on the microstructure of C-S-H gels.

2. Experiment

2.1. Materials and sample preparation

A type I Portland cement and MK is used to prepare cement paste and HSC. Quartz sand (fineness modulus 2.8) and crushed limestone (density 2.55 g/cm³) with grain size between 5 mm and 25 mm are used as aggregates. The chemical compositions of the Portland cement and MK are listed in Table 1 and their technical properties are listed in Table 2.

Mix proportions of HSC samples are listed in Table 3. Cement paste mixtures (P0, P1) containing 0% and 10% MK were prepared with the same w/b ratio of concrete specimens. After mixing and vibration, the concrete mixtures were casted in molds sizing of 100 mm × 100 mm × 100 mm and the cement paste mixtures were casted in molds sizing of 40 mm × 40 mm × 40 mm. All samples were cured according to the curing cycle demonstrated in Fig. 1.

In order to identify detail microstructure changes of C-S-H after incorporation of aluminium, the C-S-H and C-A-S-H were synthesized. The C-S-H and C-A-S-H samples were prepared by solid materials ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) in boiled deionized water at a water/solid ratio of 20, after mixing continuously by magnetic stirrer for 1 h, they were curing in 80 °C water bath for 14 days. The C-A-S-H with a Ca/Si ratio of 1.0 and Al/Si ratio varies from 0 to 0.3 were obtained by changing the proportion of $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$. In addition, the C-A-S-H gels were also prepared with metakaolin and calcium

Table 1
Chemical compositions of the Portland cement and MK (wt%).

Oxide	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	SO ₃	MgO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	ZrO ₂	LOI
Cement	19.37	3.92	68.30	3.69	0.81	1.61	0.13	0.59	–	–	–	1.09
MK	50.27	34.46	0.29	0.75	0.21	–	–	0.69	0.42	0.06	0.01	–

Table 2
Technical properties of Portland cement and MK.

Properties	Blaine fineness (m ² /kg)	Density (g/cm ³)	Setting time (min)	
			Initial	Final
Cement	419.7	3.10	237	298
Metakaolin	1248.0	2.48	–	–

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