



Contents lists available at ScienceDirect

Construction and Building Materials

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

Influence of physico-chemical characteristics on the carbonation of cement paste at high replacement rates of metakaolin

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HIGHLIGHTS

- Cement paste with fluidized bed and flash metakaolin was tested at replacement levels of up to 50%.
- Carbonation of the cement paste was evaluated in high CO₂ and humidity levels.
- Physico-chemical characterization using water porosity, absorption, TGA and XRD.
- Flash metakaolin was less susceptible to carbonation than fluidized bed.

ARTICLE INFO

Article history:

Received 20 March 2017

Received in revised form 31 July 2017

Accepted 3 October 2017

Keywords:

Cement paste

Metakaolin

Durability

Cement hydration

Carbonation

ABSTRACT

The study detailed in this paper examined the influence of two types of metakaolin (MK1 and MK2) at high replacement rates on the carbonation rate of cement paste. “Flash” calcination produced MK1, which had a high amount of quartz mixed in, while “fluidized bed” calcination produced MK2, which was relatively pure. The metakaolins were incorporated into the cement paste at rates of up to 50% by weight of cement and cured for 90 days. With regard to durability, MK replacement increased water porosity and susceptibility to carbonation, while water adsorption and primary sorptivity decreased. Thermogravimetric analysis (TGA) and X-ray Diffraction (XRD) analysis of the hydration phases showed a decrease in portlandite (Ca(OH)₂) content with MK replacement, increasing carbonation susceptibility. Decreased portlandite showed a strong correlation with an increased carbonation rate, indicating that these are primary factors in metakaolin cement paste carbonation. With TGA deconvolution, MK1 significantly increased CSH content up to a replacement level of 20%, while replacement levels over 30% appeared to reduce CSH formation. MK2 replacement did not have as significant an effect on CSH formation, corresponding with the higher primary sorptivity and greater susceptibility to carbonation penetration of MK2.

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1. Introduction

The use of kaolinite-based materials in construction has been studied since the 19th century. While Portland cement is the most popular binding material used in concrete materials today, there have been supplementary cementing materials (SCMs) introduced that not only reduce the environmental impact of concrete, but also improve its performance with respect to durability

[16,20,36]. SCMs such as fly ash, blast furnace slag, silica fume and cement kiln dust have been used with success to partially replace Portland cement, while using industrial by-products that would otherwise be in landfills [24,25,35,39].

Metakaolin is a manufactured supplementary cementing material that has gained popularity as an alternative to Portland cement and SCMs [14]. Metakaolin is obtained through the calcination of kaolinitic clay at temperatures of 650–900 °C [29]. This can be done by i) “flash process,” in which the clay is immediately calcined after crushing as it goes through a flame burner, or ii) the “fluidized bed” process where the clay is baked in a kiln for several hours before the crushing process, making it much slower than the “flash” production process. The product is further dehydroxylized

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(at around 100 °C) by the application of heat over a period of time [13,26,51].

Some SCMs that are by-products from industrial processes – particularly those of high quality that come from industrial processes such as coal burning and steel manufacturing [37] – have become less available, as demand for their use as SCMs has exceeded production, and both are on the decline for environmental and economic reasons [38]. Metakaolin is produced mostly from raw materials that are readily available. Although this increases the ecological footprint, it allows metakaolin to be more controlled during manufacturing, as raw materials can be better quality-controlled than recycled ones [8,50]. With other types of SCMs, the manufacturing process is generally controlled by the producers of the primary product [20].

In terms of environmental impact, Portland cement clinker manufacturing produces significant carbon dioxide emissions, with the production of each ton of Portland cement clinker releasing 0.7 t of carbon dioxide into the atmosphere [23]. With metakaolin production, less carbon dioxide is released; for every ton of metakaolin produced, only 175 kg of CO₂ are released, which is 17.5% of the amount produced by Portland cement. This process also consumes less energy and produces less thermal energy [17].

High silica and alumina content in metakaolin leads to pozzolanic activity when it reacts with Portland cement (primarily calcium hydroxide) and water [15,44], leading to the production of CSH and various other hydration products. Pozzolanic activity from the addition of SCMs to cement paste has been shown to improve both late-age compression strength [17,31,44] and concrete and tensile strength [31,46]. However, MK has been found to have more pozzolanic activity compared to other SCMs – especially at early ages – which can be attributed to the relatively high presence of Al and fineness [43].

The total water porosity of metakaolin has been shown to both increase [17,29,32] and decrease [6,8,44] with metakaolin replacement. However, the resulting pore structure is more refined [6,8,11,55], leading to improved performance against free/thaw cycles [27,55] and chloride penetration [7,27]. The pozzolanic reaction of silica from metakaolin also leads to a reduction in calcium hydroxide (portlandite), which has been shown to reduce the chance of sulfate attack [3] and alkali-silica reaction [45,47].

However, a significant challenge to metakaolin incorporation in concrete has been a reduced resistance to carbonation [9]. Carbonation occurs during the exposure of cement paste to CO₂ environments, notably in high CO₂ environments such as structures near factories, power plants and high-traffic areas. Carbonation is a diffusion mechanism where CO₂ penetrates the pore network, reacting with calcium and hydroxide ions – such as those from portlandite – precipitating CaCO₃ [18], as shown in Eq. (1) [36].



However, this does not always imply a negative effect, as carbonation can fill in pore space in cement paste. In pastes with elevated metakaolin content, however, the reduction in portlandite from pozzolanic reactions, combined with the lower pH of the pore solution, reduces the chemical barrier (the dissolved portlandite) between the CSH and CO₂. While the carbonation of portlandite and CSH occur simultaneously, the reduction in portlandite causes a more intense decalcination and polymerization of the CSH by the CO₂ [18]. These mechanisms reduce the resistance of the paste, causing cracks, which, along with the reduction in the pH of the pore solution, make it more susceptible to corrosion [10]. Carbonation is also affected by pore volume and structure, curing conditions, temperature, w/c ratio of the binder, and binder content [28,53]. The presence of alkaline ions Na²⁺ and K²⁺ can also have an effect in maintaining high pH [18].

The objective of this research was to investigate the influence of metakaolin type and high rates of replacement on cement paste carbonation. The metakaolin replacement level varied from 0 to 50% by weight of cement, with two types of metakaolin extracted from two different sources and manufactured using different processes. Several physico-chemical properties related to the durability of cement paste were also investigated. Physical testing consisted of water porosity and water absorption analysis, while chemical testing consisted of Thermogravimetric Analysis (TGA) and X-ray Diffraction (XRD). This relatively high replacement range allowed for the observation of some of the less apparent results of metakaolin replacement. While there has been study of the effects on carbonation on concrete and cement paste with metakaolin replacement, the effects of carbonation on the physical and chemical properties of the cement paste have yet to be examined in such detail.

2. Experimental program

2.1. Materials

2.1.1. Cement

The type of Portland cement (Table 1) used in this study was Lafarge CEM I 52.5R, as designated by the European standard EN 197-1:2000 [1]. The most important element in cement clinker is calcium – due to its limestone origins – while silica quantity is also significant. These two elements are found in C₃S and C₂S, which are the two most common compounds in cement at 62 and 10% by mass, respectively. The C₃S is primarily responsible for early CSH formation, while C₂S hydrates at later ages. The excess calcium ions present in the concrete pores after placement are essential for pozzolanic activity with SCMs, along with water [36].

2.1.2. Metakaolin

Two types of metakaolin, manufactured in different ways, were used in this study. MK1 came from a relatively impure source, with a high amount of quartz. It was produced via “flash” calcination (Table 2). MK2 came from a relatively pure source of kaolin and was produced via “fluidized bed” calcination [13].

The particle size distribution of MK2 has more fine particles than cement and MK1, with a similar distribution to cement for particles 10 μm and larger. Metakaolins have a much higher water demand than Portland cement due to their higher fineness [52]. MK1 has a much higher quantity of quartz than MK2, and with that, a higher estimated SiO₂ content. Due to the higher quantity of metakaolin particles, the water demand is higher for MK2 than MK1, and much higher than for cement, as typical with metakaolin [5]. The distribution of MK1 particles is coarser compared to cement, although the origin of the coarser and more angular [52] particles appeared to be the quartz from the Scanning Electron Microscope (SEM) observations shown in Fig. 1. The LOL value for MK2 is higher, indicating a lower purity for the metakaolin particles themselves. However, assuming the quartz constituted the larger particles in MK1, the particle size of the actual metakaolin in MK1 was finer than for MK2, indicating a potentially more reactive metakaolin component.

2.2. Mix design

The mix design for the cement paste samples had two variables. The first was the replacement percentage, with metakaolin replacing cement at 0, 5, 10, 15, 20, 25, 30, 35, 40 and 50%. The second was the type of metakaolin used. A water to cement ratio of 0.4 was used for each sample to allow enough water for hydration of the cementing materials, especially since metakaolin has a high water demand [34].

2.3. Preparation of cement paste

2.3.1. Mixing

The cement paste was mixed with a mechanical mixer in accordance with EN 196-1 [19], then placed in cylindrical plastic molds with a diameter of 30 mm and a height of 100 mm. The cylinders were filled to the top with paste while on the vibrating table, and the samples were covered in plastic wrap on the top exposed side and placed in a curing room at 20 °C and 80% RH for 24 h.

2.3.2. Curing

The samples were de-molded by cutting the molds from the outside with a precision saw. They were stored in a container full of tap water at room temperature (20 °C) for 90 days. The water in the container was replaced every two weeks.

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