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Microstructure, hydration and nanomechanical properties of concrete containing metakaolin

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HIGHLIGHTS

- Microstructure and nanomechanical properties of concrete containing metakaolin.
- Metakaolin transforms portlandite into C–S–H gel by means of pozzolanic reaction.
- Addition of metakaolin reduces porosity and creates nucleation sites for hydration.
- Metakaolin modifies the relative proportions various phases of C–S–H gel.

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ABSTRACT

This paper presents the results of an experimental investigation carried out to evaluate the microstructure, hydration and nanomechanical properties of concrete containing metakaolin. The properties of concrete containing metakaolin at 0%, 5%, 10% and 15% by mass of cement were studied for their compressive strength, sorptivity and carbonation resistance at two different water–binder ratios. It was found that 10% of the Portland cement could be beneficially replaced with the metakaolin to improve the sorptivity and carbonation resistance of concrete. In order to have a better understanding of the microstructure, hydration and nanomechanical properties various analytical techniques such as XRD, MIP and nanoindentation studies were carried on cement paste samples (with and without 10% MK). Test results showed that the incorporation of metakaolin modifies the cement paste in four different ways. Firstly, by transforming portlandite into C–S–H gel by means of pozzolanic reaction, secondly by reducing the porosity, thirdly by creating nucleation sites for hydration and finally, by modifying the relative proportions various phases of C–S–H gel.

1. Introduction

Durability has been one of critical considerations whilst designing reinforced concrete structures with long service life due to number of economic and environmental reasons [1–3]. Durability of concrete is its ability to resist chemical and physical attacks during its service life. There are different causes of deterioration, such as corrosion of reinforcement bars due to carbonation or chloride ingress, freezing and thawing action, sulphate attack and alkali-aggregate reaction. Carbonation is the reaction of the cement hydration products penetrating into the concrete from the atmosphere. This reduces the pH of concrete pore solution from 12.6 to less than 11 [4]. As a consequence, the steel passive oxide film may be destroyed and uniform accelerated corrosion may occur [5,6]. On the other hand, when chloride concentration of concrete exceeds a certain threshold value, which is dependent on several material and environmental factors, the reinforcement steel would start to corrode [7–9]. It is generally recognised that the utilisation of cementitious and pozzolanic by-products, such as fly ash, slag and microsilica, improves the concrete durability [10–13].

Due to its high pozzolanic properties, the utilisation of metakaolin (MK) as a supplementary cementitious material in concrete has received considerable interest in recent years [14–17]. MK is a thermally activated alumino-silicate material obtained by calcining kaolin clay within the temperature range 650–800 °C [18]. It contains typically 50–55% SiO_2 and 40–45% Al_2O_3, and is highly reactive. An important difference between MK and other pozzolans is that MK is a primary product, whilst others are either secondary products or by-products. Thus, MK can be produced

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with a controlled process to achieve the desired properties. Also, the white colour of MK results in a concrete with lighter colour, and is suitable for colour matching and other architectural applications. The use of MK is reported to increase the concrete strength especially during the early ages of hydration [19,20]. After 14 days of curing the contribution that MK provides to concrete strength is reduced. The increase in the compressive strength of MK concrete is thought to be due to the filling effect where MK particles fill the space between cement particles, acceleration of cement hydration and the pozzolanic reaction of MK [21–23].

Metakaolin is increasingly used to produce high-strength, high-performance concrete with improved durability. Extensive research is reported in the literature concerning different properties of paste, mortar and concrete containing MK, such as pozzolanic reaction, compressive and flexural strength and shrinkage cracking [24,25]. However, limited information is available on the effect of MK on the sorptivity and carbonation resistance properties of concrete. Therefore, this research was conducted to investigate the effect of MK on these two durability properties of concrete. In order to have a better understanding of the microstructure, hydration and nanomechanical properties various analytical techniques such as XRD, MIP, SEM and nanoindentation studies were carried on cement paste samples (with and without 10% MK).

2. Experimental programme

2.1. Materials

Commercial Swan General Purpose Portland cement (Type GP) with normal consistency of 28.5% and specific surface area of 352 m^2/kg (blain fineness) was used. The coarse aggregate used was crushed basalt with 20 mm and 10 mm size fractions mixed in the ratio 1:1 by mass and the fine aggregate used was medium graded natural sand. Both materials were obtained from local sources in Western Australia. The chemical composition and physical properties of cement and MK used in this study are reported in Table 1. A polycarboxylate-based superplasticiser was used in the concrete mixes to keep the workability constant with a slump of 50(±5) mm. The dosage of superplasticiser was increased with an increase in the MK content in order to offset the increased water demand, which is due to their high fineness.

Table 1
Chemical composition and physical properties of cement and MK used.

Parameters	PC	MK
SiO_2 (%)	21.41	52.10
Al_2O_3 (%)	5.11	41.00
Fe_2O_3 (%)	2.61	4.30
CaO (%)	61.50	0.09
MgO (%)	1.78	1.36
SO_3 (%)	3.03	–
Na_2O (%)	0.33	0.01
K_2O (%)	0.61	0.62
P_2O_5 (%)	0.16	–
Loss on ignition (%)	2.58	0.50
Specific gravity	3.18	2.63
Specific surface area (m^2/kg)	352	12000

The X-ray diffraction (XRD) patterns of cement and MK are shown in Fig. 1. In cement, typical peaks of alite (A), belite (B) and ferrite (F) phases were detected. On the other hand, XRD patterns of MK indicate that the crystalline phases in MK consisted of kaolinite, mica and quartz.

2.2. Mix proportions

The investigation on concrete was carried out at water–binder ratios (W/B), 0.5 and 0.6, and the MK replacement level was 0%, 5%, 10% and 15% by mass of cement. Details of the mix proportions for both the series are summarised in Table 2. The investigation on cement paste was carried out at a fixed W/B of 0.4, and the MK replacement level was 0% and 10% by mass of cement. No superplasticiser was used in the cement paste.

2.3. Manufacturing and curing of test specimens

Six blocks of 250 × 250 × 100 mm size and three cubes of 100 mm size were cast for each mix. The blocks were used for the permeation and durability studies, whereas the cubes were used to determine the compressive strength. Concrete was manufactured using a 160 kg capacity pan mixer (speed of rotation was 26 rotation per minute and temperature and relative humidity of room were 20(±1) °C and respectively 65(±1)%). All specimens were cast in two layers and compacted on a vibrating table with a frequency of 60 Hz until air bubbles appearing on the surface stopped. The specimens in their moulds were covered with a plastic sheet and kept in the casting room at 20(±1) °C for 24 h. These were then demoulded and placed in a water bath at 20(±1) °C for 3 days. The specimens were then sealed in polythene sheets and transferred to a storage laboratory maintained at a constant temperature of 20(±1) °C and a relative humidity of 65(±1)% until required for testing. This curing regime was considered to result in most of the hydration reactions being completed along with the prevention of any leaching of calcium hydroxide from the specimens.

For nanoindentation test, small samples were obtained by initially cutting cement cube samples using a tile cutter into 10 × 10 × 50 mm sticks. The cement sticks were then cut using a Precision Saw down to smaller 10 × 10 × 8 mm samples. The samples were then placed into moulds and cast into epoxy resin. Initial grinding and polishing of samples was performed using silicon carbide papers of reducing gradation 52 μm, 35 μm, 22 μm and 15 μm to expose the surface of the cement as shown in Fig. 2. Following the exposure of the cement surface, samples were impregnated using red-pigmented epoxy resin to provide structural support to the fragile porous cement matrix, which can be easily damaged during indentation as shown in Fig. 3. Once impregnated, samples were put through a final stage of grinding and polishing using reducing carbide papers of 52 μm, 35 μm, 22 μm, 15 μm and diamond suspensions of reducing gradations 9 μm, 6 μm, 3 μm and 1 μm and 0.05 μm on a polishing cloth. Samples were then mounted onto sample disks, placed into samples trays and installed into the indenter ready for nanoindentation tests.

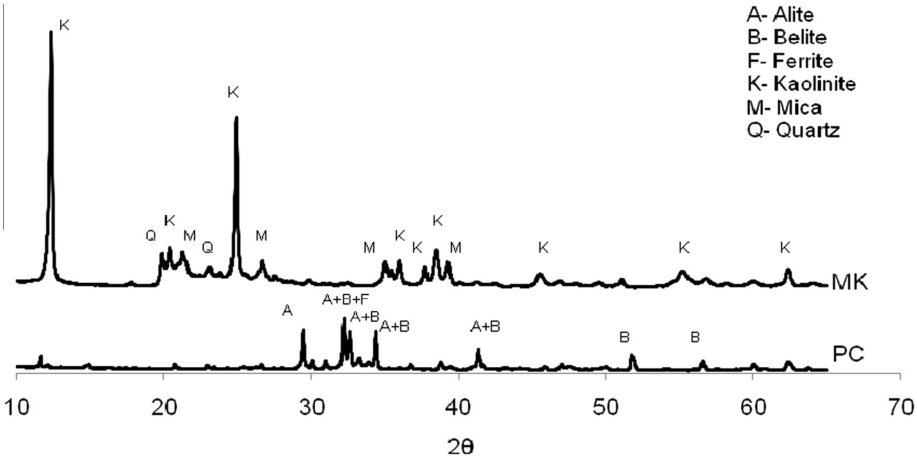

Fig. 1. XRD patterns of cement and MK used.