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Hydration and characteristics of metakaolin pozzolanic cement pastes

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Abstract The industrial area produces lots of solid waste materials with CO_2 emission. One of the most effective ways to solve these problems is the utilization of these waste materials. The production process of cements from its raw materials produces a lot of CO_2. The most effective way to decrease CO_2 emission of cement industry is the substitution of a proportion of cement with supplementary cementing materials. Cement blended with metakaolin (MK) is also required as a countermeasure to reduce the amount of CO_2 generation. Metakaolin (MK), $Al_2Si_2O_7$, is a highly amorphous dehydration product of kaolinite, $Al_2(OH)_4Si_2O_5$. The aim of our research was to investigate the effect of up to 20 wt% substitutions of OPC by MK on the hydration characteristics of MK-blended cement pastes. The physico-chemical properties of the hardened cement pastes were studied up to 90 days of hydration. The hydration products of some selected samples were investigated using XRD, DTA and DTG techniques. The results indicated that substitution of up to 20 wt% OPC by MK as pozzolanic materials resulted in an increase in the standard water of consistency, acceleration of the initial setting times, high compressive strength values at earlier ages and improvement of the mechanical and durability properties.

Introduction

Cement industry produces the 7% of the global CO_2 emission [1]. Researchers investigate the opportunities of how to decrease this level. The application of different supplementary materials can be the proper solution for this problem. With the development of industry, more and more by-products or wastes have been generated, causing serious environmental pollution problems. To solve this problem, a way must be found to consume or decrease such wastes. It has been discovered that

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many industrial wastes can be recycled as a substitute (replacement) for cement or aggregate in concrete [2].

Supplementary cementitious materials (SCMs) are now commonly used to reduce the clinker factor of cement. These materials can improve concrete properties such as compressive strength, durability and impermeability through hydraulic or pozzolanic activity. The main component of SCMs additive is usually an active amorphous SiO_2. The availability of common used industrial by-products such as fly ash, blast furnace slag, rice husk ash, silica fume, and metakaolin. Kaolinitic clays are widely available in the earth's crust, and a heat treatment between 600 and 800 °C of such clays leads to the dehydroxylation of the crystalline structure of kaolinite to give metakaolin [3–5].

Metakaolin (MK), $Al_2Si_2O_7$, is a largely amorphous dehydration product of kaolinite, $Al_2(OH)_4Si_2O_5$, which exhibits strong pozzolanic activity [6–9]. MK is processed from kaolin clay by calcination at moderate temperature (650–800 °C). At higher temperatures (>900 °C), the metakaolin undergoes further reactions to form crystalline compounds, the end-products being free silica and mullite. It contains silica and alumina in an active form which will react with CH. The principal reasons for the use of clay-based pozzolans in mortar and concrete have been materials availability and durability enhancement. In addition depending on the calcining temperature and clay type, it is also possible to obtain enhancement in strength, particularly during the early stages of curing. The very early strength enhancement is due to a combination of the filler effect and accelerated cement hydration [10]. Subsequently, these effects are enhanced by the pozzolanic reaction between MK and the CH produced by the hydration of the cement. The reactivity of MK has been linked to its content of penta-coordinated aluminum ions that are formed during the dehydroxylation process [9,11]. The pozzolanic activation of MK by various activators (calcium hydroxide (CH), sulfates as well as alkali hydroxide) and the properties of these binders have been previously reported [2]. The principal reaction between MK and CH was derived from cement hydration, in the presence of water. This reaction forms additional cementitious CSH gel, together with crystalline products, which include calcium aluminate hydrate and alumino-silicate hydrates (C_2ASH_8, C_4AH_{13} and C_3AH_6). The crystalline products depend principally on the MK/CH ratio and reaction temperature [4,13]. This reaction, which is even slower than the hydration of plain Portland cement improves the binding properties of blended cements [11].

Production of concrete with the incorporation of industrial waste not only provides an effective way to protect the environment, but also leads to better performance annually, and it is possible to completely consume most of the industrial waste in the world, provided that suitable techniques for individual waste incorporation are available [14]. Many researchers have shown a lot of interest in MK as it has been found to possess both pozzolanic and microfiller characteristics [6]. The replacement with 30 wt% of MK leads to substantial improvement in strength and transport properties of blended concrete when compared to that of unblended concrete [15]. Inclusion of MK as partial replacement of cement enhanced the compressive strength of concrete, but the optimum replacement level of OPC by MK was about 20 wt% [14]. Dinakar et al. [16] studied the effect of incorporating MK on the mechanical and durability properties of high strength concrete for a constant W/B ratio 0.3. MK mixture with cement replacement of 5, 10 and 15 wt% was prepared. The results showed that 10 wt% replacement level was the optimum level of MK content.

The objectives of this study were to investigate the substitution of OPC by MK up to 20 wt% on the hydration characteristics of MK-blended cement pastes. The physico-chemical properties of cement pastes were determined up to 90 days. The hydration products of some selected samples were investigated by using XRD, DTA and DTG techniques.

Materials and methods of investigation

The materials used in this study are ordinary Portland cement (OPC) and kaolinite clay. OPC was provided by Suez Cement Company, Suez plant, El-Ain El-Sokhna, and the kaolinite clay was derived from Ras Abu Zneima Zone, South of Sinai, Egypt.

Metakaolin (MK) is a product from dehydroxylation of a clay mineral, kaolinite, which is very fine powder prepared by firing in a muffle furnace from room temperature up to 800 °C for 2 h. The ground MK passed through 90 μm B.S. sieve. The chemical and physical properties of starting materials are shown in Table 1.

The mineralogical composition of MK is seen from XRD pattern in Fig. 1. It shows the presence of quartz as the main mineral and amorphous alumino-silicate phase, and the amorphous phase is formed as a result of reactions between SiO_2 and Al_2O_3 as well as fluxing oxides impurities oxides at high temperature. The mix composition of MK-pozzolanic cement is shown in Table 2.

Mixes were prepared by substitution of OPC with 5, 10, 15 and 20 wt% of MK. The dry constituents of each mix were mechanically mixed for one hour in a porcelain ball mill using four balls to attain complete homogeneity, then kept in airtight containers for further investigation. The standard water of consistency as well as initial and final setting times was determined

Table 1 Characteristics of OPC and metakaolin.

	OPC	MK
(a) Oxide composition, %		
CaO	62.72	0.28
SiO_2	20.68	55.10
Al_2O_3	4.90	34.10
Fe_2O_3	3.35	5.24
MgO	2.64	0.25
SO_3	2.65	0.01
Na_2O	0.11	0.10
K_2O	0.14	0.02
TiO_2	0.12	2.00
P_2O_5	0.10	1.00
L.O.I	2.73	1.50
(b) Blaine surface area (cm^2/g)	3400	11,000
(c) Residue on sieve (%):		
90 μm	0.00	1.00
45 μm	12.00	12.80
(d) Specific gravity (g/cm^3)	3.15	2.65
(e) Insoluble residue (%) HCl/Na_2CO_3	0.45	63.45