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Research paper

Effect of hydrothermal curing on the hydration characteristics of artificial pozzolanic cement pastes placed in closed system

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ABSTRACT

This paper describes the effect of high temperature on the hydration characteristics of artificial pozzolanic cement pastes containing metakaolin with or without silica fume for different hydrothermal ages in well closed stainless steel capsule. Pastes were hydrothermally treated in well closed stainless steel capsule at 180 °C for different hydrothermal ages (2, 4, 6, 12 and 24 h). Hydration characteristics of the hydrothermal OPC–MK–SF pastes were studied by the determination of compressive strength, bulk density, porosity and chemically combined water contents at different hydrothermal ages. The phase composition and morphology of the formed hydrates were studied using X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM). Results indicated that the compressive strength of multi-blended mixes containing silica fume 5% is by mass higher than the corresponding Portland cement control at later age up to 6 h of hydrothermal curing age.

1. Introduction

The object of producing pozzolanic cements has been of considerable scientific and technological interest because such addition of pozzolana increases the chemical resistance to sulfate attack, interpermeability, lowering heat of hydration, and thermal expansion (Hewlett, 1998). Recently, the use of pozzolanic cement is increasing world-wide because it needs less energy for production. Hydration of cement is a complex exothermic chemical reaction consisting of several major phases which correspond with the participation of different clinker minerals. The process of hydration heat evolution is affected by the cement composition, cement fineness, and ambient temperature as the most important factors (Hewlett, 2004; Taylor, 1997). Therefore, it is useful to know both the differential and integral characteristics of the hydration heat development for particular cement under different conditions. Autoclaving building products possess several advantages including the marked development at shorter times of autoclaving at relatively high steam pressures. In addition, the utilization of industrial solid wastes in the production of high strength building products is of prime importance for environmental protection and development (Amin and Hashem, 2011). The pozzolanic activity of calcined clays depends on the type and amount of clayed minerals, the nature and amount of impurities, the thermal treatment used for its activation and the specific surface obtained after calcination. Four test methods for assessment of the pozzolanic activity on seven calcined clays were analyzed (Tironi et al., 2013). The MK is a valuable pozzolanic and thermally activated aluminosilicate material obtained by calcining kaolin clay within the temperature range of 700–850 °C. MK is usually added to concrete in amounts of 5–15% by weight of cement. Addition of MK causes increase in mechanical strength, enhancement of long term strengths, decrease of permeability, porosity, reduction of efflorescence, increase of resistance to soluble chemicals like sulfates, chlorides and acids (Ambroise et al., 1994; Khatib et al., 1996; Kostuch et al., 1993; Shatat et al., 2014).

The SF and dealuminated kaolin (DK) from Egyptian sources have been characterized chemically and mineralogically, and a comparative study of their reactivities toward lime was conducted using isothermal conduction calorimetry and an accelerated chemical method (Mostafa et al., 2001). Silica fume is a byproduct of the reduction of high-purity quartz with coal in electric furnaces in the production of silicon and ferrosilicon alloys. Silica fume is also collected as a byproduct in the production of other silicon alloys such as ferrochromium, ferromanganese, ferromagnesium, and calcium silicon. Silica fume consists primarily of amorphous (non-crystalline) silicon dioxide (SiO_2). The individual particles are extremely small, approximately 1/100th the size of an average cement particle. Because of its fine particles, large surface area, and the high SiO_2 content, silica fume is a very reactive pozzolana when used as a blend in cement and concrete (ACI Committee 226, 1987; Cheng Yi and Feldman, 1985; Kumar and Roy, 1983). The quality of silica fume is specified by ASTM C 1240 and AASHTO M 307. Toutanji and El-Korchi (1996) studied the effect of silica fume on the compressive and uniaxial direct tensile strength of Portland cement pastes and mortars. Many authors studied the durability of OPC blended with silica fume against sulfate ions (Al-Dulaijan, 2007; Toutanji and

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El-Korchi, 1996), fire (Hekal, 1997a, 1997b; Morsy and Shebl, 2007; Zelic et al., 1999) and acid solutions (Lefebvre et al., 1997; Pavlik and Uncik, 1997). The role of SF in the reaction kinetics and mechanisms of the early-stage hydration of Portland slag cement–silica fume pastes (W/S = 0.5 at 20 °C) has been studied (Zelic et al., 2000). The reaction stages have been analyzed and explained by kinetic terms. The delay in CH formed during hydration between 8 and 10 h from the beginning of hydration has been noticed, and a mechanism has also been discussed. The results of this study have revealed evidence of the accelerator effect of SF during the first 8 h of hydration when it still exists as chemically inert filler. The addition of SF resulted in a further densification of the microstructure of the hardened ordinary Portland cement and burnt clay with silica fume pastes; this was reflected on the observed improvement in the compressive strength values at all ages of hydration (Amin et al., 2012; Shatat, 2013). The effects of substituting cement with 10%, 20% and 30% natural zeolites on concrete durability were compared to the effects of substituting 5%, 10% and 15% metakaolins and 5%, 7.5% and 10% silica fumes, along with water-to-cement ratios of 0.35, 0.40, 0.45 and 0.50. Results show that, in general, the zeolite is not as active as silica fume or metakaolin, although it could be used as a substitute for pozzolans because it has better durability characteristics and is economical and environmentally friendly as well (Valipour et al., 2013). The identification of mineral phases produced during the pozzolanic reaction in a ternary system, formed by a pozzolan mix (activated paper sludges and fly ash) and calcium hydroxide, cured at 40 °C during 1, 3, 7, 14, 28 and 90 days of reaction was studied (Frías et al., 2013). The knowledge of hydrated phases during the pozzolanic reaction will be determinant for the use of this activated waste in the manufacture of future ternary blended cements.

Hydrothermal treatment of these wastes is almost associated with building products having improved binding characteristics. The properties of these autoclaved products are always governed by the chemical composition and physical state of the formed hydration products which act as the main binding centers (Abo-El-Enein et al., 1997; Luke, 2004; Siauciunas and Baltakys, 2004). Hydration characteristics of the autoclaved CKD–sludge–silica fume pastes were studied by the determination of compressive strength and chemically combined water contents at different autoclaving ages, the results indicate that autoclaved cement kiln dust (CKD)–sludge pastes possess a considerably compressive strength at all different autoclaving ages. This is mainly attributed to the hydrothermal interaction between the lime released from CKD with sludge to give calcium silicate hydrates (Amin and Hashem, 2011).

The object of this investigation is to study the hydration characteristics of hydrothermal specimens made with Portland cement and metakaolin in presence of silica fume. The phase composition and the morphology of the formed hydrates were studied using X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM).

2. Materials and experimental techniques

Raw materials used in the present work are: OPC from Assuit Cement Co., Egypt and condensed SF which is a by-product of silicon or ferrosilicon alloy industries. It was obtained from the Ferro-Silicon Company, Kom-Ombo, Egypt. The SF particles are spherical and have an average diameter of about 0.1 μm. It consists of ~99% amorphous silica with a specific surface area of 20,000 $m^2\ kg^{-1}$. These characteristics account for the substantial pozzolanic activity of SF in terms of both its capacity of binding lime and the rate of hydration reaction, and kaolin was collected from Kalabsha, Aswan, Egypt. Kaolin was calcined in an electrical muffle furnace with a heating rate of 10 °C/min at 800 °C for 3 h, to give metakaolin (MK). The various mixtures and the chemical compositions of the starting materials are shown in Tables 1 and 2 respectively. Each dry mixture was homogenized for 1 h in porcelain ball mill provided with four balls to obtain complete homogeneity then kept in airtight containers until the time of cement paste preparation. The mixing of each dry cement mixture was carried out with the required water of standard consistency which is previously determined according to ASTM Designation C 187-98 (ASTM Designation: C 187-98, 2002). The resulting paste was then pressed by hand molding pressure into stainless steel cylindrical molds of 3.14 cm^2 cross-section and 2 cm height. The molds were vibrated for 1 min to remove any air bubbles and voids. Immediately after molding, the cylindrical specimens were cured in the humidity cabinet at about 100% relative humidity at room temperature (23 ± 2 °C) for 24 h in order to attain the final setting of the specimens. The specimens then were demolded and cured under tap water in stainless steel capsule (as shown in Fig. 1.) keeping the occupied volume equal to 0.67 of total volume capacity. The capsule was tightly closed to avoid sealing of water vapor. The capsule was heated in electric oven for appropriate temperature and time. At the end of the hydrothermal treatment process, the capsule was removed from the oven and cooled to room temperature. Then, hydration characteristics of the hydrothermal pastes were studied by the determination of compressive strength, bulk density, porosity and chemically combined water contents at different hydrothermal ages.

Bulk density was determined using Archimedes' principle (De Gennaro et al., 2004). The compressive strength was measured using a manual compressive strength machine for a set of three cubes according to ASTM designation (ASTM designation: C109-80, 1983). Free water content was determined using domestic microwave oven (Olympic electric model KOR-131G, 2450 MHz, 1000 W) (Pavlík et al., 2003). The combined water content was determined using hydration stopped specimen after being ignited in porcelain crucibles at 1000 °C for 1 h in a muffle furnace. The total porosity of the hardened cement paste was calculated from the values of bulk density, free and total water contents as described elsewhere (Copeland and Hayes, 1956). Free lime CaO were determined at the various curing times after stopping the hydration of the hardened pastes (Grim, 1962; Searl and Grimshaw, 1971). X-ray diffraction (XRD) analyses were carried out by Philips X-ray diffractometer PW 1370; Co. with Ni filtered CuKa radiation (1.5406A).

Table 1
Mix composition in wt.% of blended cements.

Symbol	OPC	MK	Silica fume
PC	100	0	0
MK	75	25	0
SF5	75	20	5
SF10	75	15	10

Table 2
Chemical composition of the starting materials, wt.%.

Oxide contents	Portland cement	Silica fume	Kaolin
SiO_2	20.48	92.90	44.18
Al_2O_3	4.76	1.10	36.75
Fe_2O_3	4.69	0.82	1.36
CaO	62.15	0.42	0.26
SO_3	2.63	–	–
MgO	1.47	0.52	0.16
Na_2O	0.46	0.64	0.18
K_2O	0.27	1.12	0.25
TiO_2	0.58	–	2.94
P_2O_5	0.15	–	–
L.O.I.	1.85	1.56	13.55
Total	99.51	99.72	99.63