Construction and Building Materials 154 (2017) 219–228

Contents lists available at ScienceDirect

Construction and Building Materials

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

Study on hydration products by electrical resistivity for self-compacting concrete with silica fume and metakaolin

P. Ghoddousi *, L. Adelzade Saadabadi

Department of Civil Engineering, Iran University of Science and Technology, Iran

HIGHLIGHTS

- Influence of SF and MK used in SCCs on electrical resistivity was studied over time.
- The hydration products of SF-MK cement pastes from SCCs were evaluated by XRD, TGA.
- The $Ca(OH)_2$ content and electrical resistivity have a good relation.
- The electrical resistivity can be applied as a simple method to calculate CH content.

ARTICLE INFO

Article history:
Received 5 May 2017
Received in revised form 20 July 2017
Accepted 24 July 2017

Keywords:
Electrical resistivity
Hydration products
Calcium hydroxide
Thermogravimetric analysis
X-ray diffraction

ABSTRACT

This paper investigates the hydration products of self-compacting concrete (SCC) made with binary and ternary blends of silica fume(SF) and metakaolin(MK). X-ray diffraction (XRD) and thermogravimetric analysis (TGA) test were used to measure the hydration products of pastes corresponding to self-compacting concretes. For this purpose, the reference SCC mixture at constant water to cementitious material (W/CM) ratio of 0.45 and total cementitious material content of 450 kg/m^3 was prepared. Other mixtures contained binary (92%PC + 8%SF, 80%PC + 20%MK) and ternary (72%PC + 8%SF + 20%MK) cementitious blends of metakaolin and silica fume. Four pastes corresponding to SCC mixtures with the same W/CM and cementitious materials were considered. Also, the electrical resistivity of SCC mixtures was measured to assess the relationship between electrical resistivity over time and calcium hydroxide (CH) content. The correlation coefficient of the relationship between $Ca(OH)_2$ content and electrical resistivity was found to be 0.95 with positive slope for reference SCC and 0.97 with negative slope for samples containing pozzolans. It can be suggested to apply the electrical resistivity as a simple method to calculate CH content in cement paste.

1. Introduction

In recent years, the use of binary and ternary blends of pozzolans of silica fume (SF) and metakaolin (MK) as supplementary cementitious materials (SCM) in self-compacting concrete structures exposed to aggressive environments has been increasing. ASTM C618 describes a pozzolan as a siliceous or silico-aluminous material which in itself possesses little or no cementitious value but chemically react with hydrated lime (CH) at ordinary temperature to form compounds possessing cementitious properties during pozzolanic activity. Pozzolanic activity is corresponding to the reaction of SiO_2 and Al_2O_3 in pozzolanic materials with calcium hydroxide formed during portland cement hydration process. In this process, silica precipitates in the form of calcium silicate hydrate, so-called secondary C–S–H phase. the calcium hydroxide in cement paste decreases due to pozzolanic reaction [1–4]. Juenger and Siddique [5] conducted a review research on supplementary cementitious materials (SCMs). New materials, replacement amount, modifying materials and developing better test methods to improve properties of concretes were areas which most recent research has focused on. Most concrete properties strongly depend on cement and SCM hydration process with time. The hydration process of concrete incorporating fly ash (FA), silica fume and MK has also been researched extensively [6–8]. Using X-ray powder diffraction (XRD) and thermoanalytical methods, Mlinarik and Kopecsko [9] evaluated the changes in quality and quantity of phases in concrete containing ordinary Portland cement and 17% metakaolin at the ages of 1, 7, 28, 120, 180 and 360 days. Results confirmed that the addition of metakaolin through pozzolanic reaction decreases the portlandite (calcium

* Corresponding author.
E-mail addresses: Ghoddousi@iust.ac.ir (P. Ghoddousi), leyla_adelzade@civileng.iust.ac.ir (L. Adelzade Saadabadi).

http://dx.doi.org/10.1016/j.conbuildmat.2017.07.178

hydroxide) quantity from 7.1 to 5.6 at 3 days, 11.3 to 8.5 at 7 days, 12.9 to 7.5 at 28 days, and at the ages of 120 and 180 days quantities of portlandite in metakaolin-containing samples are almost the half of the quantities in the references. The comparison between the diffractograms of cement pastes showed that the addition of 15% metakaolin reduces the peak maximum intensity count of calcium hydroxide from 248 to 112 in 28 days, which is due to the transformation of hydroxide (CH) into secondary C–S–H gel [10].

X-ray diffractometry and thermogravimetry analyses were widely applied to investigate the hydration process of cement-based systems [11–15]. Thermogravimetric analysis also was applied to determine the CH amount in cement paste and cement-pozzolan systems by measuring the mass loss of CH decomposition [11,8,16]. This method was used by researchers to determine pozzolanic activity of pozzolanic materials, specially fly ash [17–19]. Since the baseline of CH amount does not remain horizontal during the heating process, A procedure, which was suggested by Taylor and Turner [20], was used to evaluate the weight loss pertaining CH decomposition for pozzolans, such as silica fume and metakaolin. Straight tangential lines to the weight loss curve are drawn at the beginning, stationary point and end of CH dehydration. The two intersection points of the tangents define the initial and final temperatures of the process to calculate the weight loss involved. In the case of a paste with pozzolanic material, this peak represents the unreacted CH content. The amount of reacted calcium hydroxide by the pozzolan in the paste, i.e. the calcium hydroxide fixation (%CH) can be determined from the given data.

According to the Soriano et al. [15], thermogravimetric study on pastes with 15% cement replacement by metakaolin showed negative fixed lime percentages during the early curing age. Due to the high fineness, the addition of MK accelerates the cement hydration process. At 20 °C of curing, MK pastes yielded positive values of fixed lime for curing times of 3,7,28 days. Based on the results obtained by Cruz et al. [21] on high metakaolin and fly ash percentages in the paste, it can be concluded that at day 3, 100% of the CH released by the cement showed reaction, whereas at curing days of 15 and 21 only 80% of the CH was consumed. For longer curing ages such as 90 and 180 days, all the CH released by the hydration reaction of cement was fixed by FA, therefore pozzolanic reactivity of FA took place later than MK.

Many Studies were conducted on properties of self-compacting concrete incorporating silica fume and MK with TGA and XRD [2–4].

Using an X-ray qualitative diffractometry, the effect of 10% silica fume and metakaolin on the microstructural properties of self-compacting concrete (SCC) was evaluated. The results showed low calcium hydroxide and ettringite in silica fume and metakaolin cement pastes at 28 days curing [22]. The microstructural properties were examined using SEM and EDS on mixtures with high volumes of metakaolin, ground granulated blast-furnace slag and fly ash with two w/b ratios. The SEM results showed that MK produce higher amounts of C–S–H gel in higher w/b ratio. MK has a greater effect on the microstructural strength of the transition zone than GGBS. The results of EDS analysis demonstrated that lower Ca/Si ratios indicate the improvement of compressive strength [23].

The simple method is essential to evaluate cement hydration over time. In this case, electrical resistivity measurement was recently used to investigate the time-dependent properties of cement based materials. The Knudsen model [24] can be applied to relate electrical resistivity with the degree of cement hydration, thereby illustrating that hydration parameters vary over time, as seen in the following parabolic expression [21]:

$$\frac{R_t}{(R_{max} - R_t)^2} = a.t + b \quad (1)$$

where R_{max} is the asymptotic value of R, a and b are constants, and t is the time of hydration.

Based on Eq.1, it can be stated that electrical resistivity is a function of degree of cement hydration. Electrical resistivity can be applied to cases where determining the degree of hydration is difficult. Cruz et al. [21] concluded that during the first 28 days, the fitting of Eq. (1) for electrical resistivity of mortar with inert filler and high percentages of metakaolin and fly ash gave maximum value for the determination coefficient > 0.95. The adjustment was acceptable as it showed electrical resistivity as a parameter of hydration.

Establishing a relationship between durability indicators and electrical resistivity is important because it would allow the larger areas of a structure to be tested, indirectly. Numerous studies on the application of electrical methods on cement-based materials indicate that important parameters of concrete can be monitored effectively. The application of electrical methods on cementitious materials were reported before such as: monitoring changes in pore solution and connectivity with time [25,26], assessing pozzolanic reactivity of pozzolans [21,27,28] indicating durability, [24–35] and determining the setting time [36].

The electrical resistivity was proposed to predict the amount of calcium hydroxide and hydration degree for concretes containing fly ash [37].

There is little study on the relationship between electrical resistivity and CH to determine cement hydration products of CH in self-compacting concretes with SF and MK. Therefore the main objective of the present study was to investigate the relationship between the CH amount from TGA results and the electrical resistivity of SCCs with binary and ternary blends of MK and SF. An investigation was undertaken to determine the effects of pozzolanic admixtures, including metakaolin and silica fume on hydration products and electrical resistivity of self-compacting concrete (SCC) at the ages of 1, 3, 7, 28 and 365 days.

2. Experimental program

2.1. Materials

In this study, ordinary portland cement type II according to ASTM C150 [38] and limestone powder were used to develop self-compacting concrete mixtures and pastes. The utilized mineral admixtures used were silica fume and metakaolin. Table 1 illustrates chemical characteristics of the cementitious materials used in the mixtures. Self-compacting concrete mixtures were generally made from natural river sand with a specific density of 2.44, water absorption of 3.1% as fine aggregate, 19 mm maximum size stone of 2.54 specific density, and coarse aggregates of 1.8% water absorption. Fig. 1 illustrates the particle size distributions of the aggregate used for SCC mixtures.

A poly carboxylate super plasticizer with a 1.01 g/cm3 density, referred to as the p100 was utilized to achieve the SCC workability requirements according to EFNARC [39].

2.2. Mixture proportions

Four self-compacting concrete mixtures were designed for purposes of this research. The water to cementitious material ratio of the mixtures was kept constant at a value of 0.45, and a total cementitious materials content of 450 kg/m^3 was considered. The reference mixture contains 450 kg/m^3 of cement. Other mixtures contained binary (92%PC + 8%SF, 80%PC + 20%MK) and ternary (72%PC + 8%SF + 20%MK) cementitious blends, in which the proportion of portland cement was replaced with SF and MK. As reported for appropriate durability properties of concretes in