Construction and Building Materials 110 (2016) 48–53

Contents lists available at ScienceDirect

Construction and Building Materials

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

Quantitative analysis on ground blast furnace slag behavior in hardened cement pastes based on backscattered electron imaging and image analysis technology

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HIGHLIGHTS

- BSE and image analysis technology quantify GBFS content in hardened cement paste.
- Quantitative analysis is governed by slag dosage, fineness, W/B and curing age.
- A linear correlation between areal fraction of slag and other factors is proposed.

ARTICLE INFO

Article history:
Received 9 December 2015
Received in revised form 2 February 2016
Accepted 3 February 2016
Available online 9 February 2016

Keywords:
Backscattered electron microscope
Image analysis
Hardened cement paste
Ground blast furnace slag

ABSTRACT

Grounded blast furnace slag is one of common supplementary cementitious materials in Portland cement concrete, and is capable of improving the performance of concrete and reducing the costs. Once hardened, the dosage of slag in concrete matrix is hard to evaluate. Three types of ground blast furnace slag (GBFS) are used to prepare cement-slag pastes with different water binder ratios, and the finenesses and original dosages of GBFS are various. Backscattered electron (BSE) images combining with image analysis method are used to analyze the areal fraction of GBFS in the pastes. The feasibility of this method is analyzed, and it is proved that the method is effective in quantitative analysis on the content of GBFS in hardened cement pastes. The multivariate linear correlations between areal fraction of GBFS, curing age, GBFS fineness, original dosage of GBFS, are created. It is capable of obtaining original dosage of GBFS based on existing parameters.

1. Introduction

Ground granulated slag is one of the most important supplementary cementitious materials in cement paste, and has been widely used in modern concrete because of its several well-known advantages. For instance, slag is capable of improving the resistance of concrete to chloride ingress, it can also reduce the total heat generation and peak temperature reached during the early stage of mass concrete structure, which lessens the risk of early-age thermal cracking [1–3]. Meanwhile, the addition of slag affects the mechanical properties of the cement paste as well. In addition, once cement paste has hardened, it is difficult to determine the fraction of ingredients in hardened concrete, especially the dosage of slag. Thus, the determination of the slag content in cement paste is necessary, and under the circumstance, there is an increasing demand for a reliable and rapid method for estimating the slag content in hardened cement paste.

Different methods have been used to study the content of slag in hardened concrete, Xie et al. [4] reported an empirical relationship between the electrical conductivity of concrete, aggregate fraction and slag to binder ratio that might be useful in determining slag content in unknown mixtures. However, the electrical conductivity is affected by many other factors, especially the water to binder mass ratio and degree of hydration. In Sisomphon's work [5,6], a method for determining the slag content in hardened concrete was studied, but it was required to obtain the oxide compositions of original cement, slag and aggregates by X-ray fluorescence in advance. By using X-ray diffraction technique or HCl dissolving technique to determine the content of sand in the extracted mortar, followed by calculating the amount of calcium oxide in the cement paste, the slag-binder ratio could be determined. However, its precision would be diminished if aggregates

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http://dx.doi.org/10.1016/j.conbuildmat.2016.02.015
0950-0618/

were not properly removed from the hardened paste, and this method was not suitable for concretes containing calcareous aggregates. Hooton and Rogers [7] proposed the X-ray diffraction technique to determine the slag content in hardened concrete by igniting the mortar fraction of concrete at 950 °C ~1050 °C. After cooling, the mortar was mixed with 10% CaF_2 (as internal standard) and analyzed by X-ray diffraction technique. To estimate slag content, the melilite/CaF_2 peak intensity ratio is measured and compared with ignited slag mixture from the same source as calibration, the slag content could also be observed by optical microscopy on thin section made from the hardened concrete. This method involved the preparation of thin sections and determination of the slag content by point counting, and the distribution of slag particle size must be measured in advance. Grantham [8] proposed a mathematical approach to obtaining the content of the individual components of concrete. The method has been successfully used by a number of UK laboratories and has provided another possibility of resolving the composition of concrete. However, this method can only be used in specific situations where the chemical composition of original materials in concrete is known.

Among different quantitative analyses of cement-based materials by using scanning electron microscopy, Scrivener [9,10] quantitatively studied the degree of cement hydration by BSE. Since then, BSE quantitative analysis technique had been widely used in the research of cement-based materials. Backscattered electron imaging and image analysis (BSE-IA) technique for polished surfaces has been proposed as a method to analyze cement and concrete microstructures. This technique is widely applied for both qualitative analysis and quantitative analysis. Zhang et al. [11] proposed the application of gray-value processing method of backscattered electron image in the study on the hydration degree of cement and slag in slag-cement composites. The results suggested: BSE images of well-polished samples are capable of exhibiting the morphology of hydration products and unreacted grains in the pastes. Based on the analysis on compositions of hardened cement samples by BSE and calorimeter, Kocaba et al. [12] proposed the methods for determination of reaction degree of slag in blended cement pastes. The results seem to show that calibration of calorimetry with BSE-IA is a promising method to quantify the reaction degree of slag. Ouki and Hills [13] successfully used BSE image analysis to quantify the observable porosity and cement hydration in cement paste. Brough and Atkinson [14] identified the aggregate-paste interfacial transition zone by BSE/EDS pattern. Wong and Yio et al. [15,16] successfully utilized FE-SEM in the backscattered electron mode to estimate the initial cement content, water content, w/c, slag/binder ratios and curing ages of hardened cement-based materials that have unknown mixture proportions and degree of hydration. Based on the above research foundations, the extension of the method to Portland cement mortars and concretes is presented by Wong et al. [17].

Based on the above experimental work, SEM backscattered electron microscope has been successfully applied in the quantitative and qualitative analysis of cement hydration. However, quantitative study on slag content in cement paste is not sufficient. In this paper, the effect of type of blast furnace slag, water binder ratio, fineness of slag, original dosage of slag and curing age are considered. The BSE images are categorized into bright and dark regions and are colored by image processing function, the areal fraction of ground blast furnace slag is obtained. The correlation between areal fraction of slag, water binder ratio, slag fineness, original slag dosage and curing age is created. This method provides a new idea for quantitative study on mineral admixtures in hardened cement paste.

2. Experimentation and methods

2.1. Materials

(1) Reference cement used in this study was ASTM Type I/II (China United Cement Corporation, China). Its density was 3000 kg/m^3. Its physical and mechanical properties are listed in Table 1, and its chemical composition is given in Table 2.

(2) Ground blast furnace slag, its main oxide compositions were CaO, SiO_2 and Al_2O_3. The blast furnace slags used in this work were from three vendors, and were ground into three different finenesses, respectively. The specific surface areas are given in Table 3.

XRD patterns were obtained by a Standard laboratory-based bulk powder X-ray diffractometer with CuKα radiation (λ = 1.5418 Å). The XRD patterns for three ground blast furnace slag samples are shown in Fig. 1. All samples major phases are amorphous phases with minor amount of quartz, anatase and other traces in the range of 20–40° 2θ. The content of crystal phases is quite low, therefore X-ray fluorescence was conducted, and the results are shown in Table 4.

2.2. Mix proportions

To comprehensively investigate the effects of type, fineness and mass fraction of slag, and water binder ratio on backscattered-based SEM images, cement pastes were prepared with ground blast furnace slag in different types and finenesses, and their mix proportions are given in Table 5. In this study, the water binder ratios are 0.3, 0.4 and 0.5; the mass fractions of slag in total binding materials are 20%, 40% and 60%. The preparation method of samples can be found elsewhere [18]. The curing environmental conditions are 20 ± 2 °C and relative humidity 95%, and curing ages are 7 days, 28 days and 90 days.

2.3. Methods

2.3.1. Preparation for BSE

After curing at each designated age, small pieces (less than 20 mm diameter) in the central hardened paste samples were prepared with a diamond wafering blade saw. The small pieces were oven-dried at 40 °C until the weight of samples was stable. After vacuuming, the sample surface was epoxy-impregnated. The samples were polished by sand papers with different finenesses (P80, P360, and P2000) for 4–6 min, then the samples were polished by diamond wafering blade with grades of 9 μm, 3 μm, and 1 μm for 1 h. After each grade of polishing, the sample surface was cleaned by ultrasonic cleanser. Then, the samples were dried again. The samples were glued to aluminum stubs and coated with gold by a sputter

Table 2
Chemical composition of reference cement (%).

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2Oeq	f-CaO	Loss	Cl^-
21.1	4.31	3.57	61.33	2.37	2.7	0.56	0.53	2.93	1.011

Table 3
Specific surface area of different slag (m^2/kg).

Type/fineness	Coarse	Medium	Fine
I	295	334	386
II	310	347	403
III	303	341	392

Table 1
Physical and mechanical properties of cement.

Compressive strength at 3 days MPa	Compressive strength at 28 days MPa	Flexural strength at 3 days MPa	Flexural strength at 28 days MPa	Initial setting time min	Final setting time min
25.1	46.5	4.5	8.6	135	205