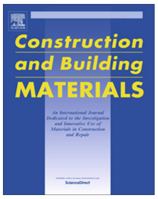
Construction and Building Materials 135 (2017) 306–314

Contents lists available at ScienceDirect

Construction and Building Materials

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



The correlation between porosity and mechanical properties of multicomponent systems consisting of Portland cement–slag–silica fume–metakaolin

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HIGHLIGHTS

- Refinement of the structure as a result of applied SCMs (BFS, SF and MK).
- Synergic effect of SCMs ensured high strengths in every curing time.
- Improvement of strength in comparison with numerous reported binary or ternary systems.
- Determination of relationship between strengths and porosities.

ARTICLE INFO

Article history:
Received 15 July 2016
Received in revised form 25 October 2016
Accepted 21 December 2016

Keywords:
Blended cements
Metakaolin
Silica fume
Ground granulated blast-furnace slag
Porosity

ABSTRACT

Pore structure and mechanical strength of quaternary systems consisting of ordinary Portland cement (PC) replaced in 25, 30 and 35 wt% by ground granulated blast-furnace slag (BFS), silica fume (SF) and metakaolin (MK) were evaluated up to 365 days. Refinement of the structure as a result of cement supplementary materials (SCMs) demonstrated mainly by the rise of gel pores portion, whereas the portion of middle capillary pores decreased. Despite dilution effect in blended samples, both the compressive (CS) and flexural strength (FS) increased throughout all the curing time and reached the values higher than that of referential sample. Development of strength in time reflected different activity of SCMs. The highest values of strength corresponding to the lowest total porosities were determined in the samples containing the most SF (CS = 125 MPa, FS = 18 MPa after 365 days), whilst the highest amount of BFS together with the highest substitution level led to the lowest strengths among blended samples (CS = 99 MPa, FS = 15 MPa).

1. Introduction

Even though the blended cements are in the center of attention for a relatively long time, their possibilities are not still exhausted. Continue improvement of their properties is based on the combined effect of various binders' materials and allowed e.g. the development of self-compacting concrete or high performance cements [1–5].

Performance of multicomponent materials depends on the variety of initial factors, such as chemical and mineralogical composition, selected preparation conditions (e.g. water content, initial and curing temperature, dosage of plasticizer) [6]. Many preparation parameters (workability, water to binder ratio, bleeding and drying shrinkage) are affected by fineness of initial materials. The rate by which cement and supplementary materials react with water also considerably depends on their fineness. Higher fineness increases reaction kinetics owing to the corresponding increase of surface area [7] and ensures mainly the early age strength acquirement [8,9].

The fineness characteristics of materials include particle morphology, particle size, particle size distribution and specific surface [10]. It is necessary to be aware that characterization of material fineness only by average particle size or specific surface area is not sufficient. Two cements with the same average particle size but different distributions will exhibit dissimilar rate of early hydration. A better parameter for describing the fineness of the

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http://dx.doi.org/10.1016/j.conbuildmat.2016.12.105

material (at least in terms of knowing the early times reactivity) is the specific surface area. However, specific surface area does not depose about the size of the largest particles and about the breadth of the particle size distribution. Both of these parameters affect the workability at a given water to binder ratio, the degree of reaction and correspondingly the strength development over long periods of time. Different particle size distribution of materials possessing the same specific surface area will lead to the different water demands. Wider size distribution increases the packing density of the system and thus effectively reduces water demand [11]. Consequently, it is expected that increasing of the packing density would have a positive contribution to strength gain potential.

Besides the fineness of initial materials, the curing procedure has an important impact on the porosity and the related final material properties. According to Ramezanianpour and Malhotra [12], the continuous moist curing of concrete is essential to achieve the lowest porosity and the highest compressive strength. They reported that the use of BFS produces a very poor permeable concrete however it is more sensitive to curing process than the use of SF or pure PC and this sensitivity increases with slag content in the particular mixtures.

It is well known that also incorporation of MK up to 20 wt% leads to the decrease of total porosity [13,14]. On the contrary, the substitution of more than 30 wt% of PC by MK results in the increase of porosity, which was attributed to its fineness and corresponding increase of water to binder ratios. The flowability of fresh cement mixtures deteriorated due to the MK content can be improved by addition of BFS. In the same manner, the presence of BFS helps to decrease the water demand and the need of plasticizer use in blended cements with SF [15]. Accordingly, it helps to prevent from the resulting decrease of the compressive strength.

In general, the contribution of SCMs to concrete strength can be divided into three elementary factors associated with fineness: the filler effect, the acceleration of PC hydration (nucleation sites) and the pozzolanic reaction of admixtures with $Ca(OH)_2$ [16–18]. SCMs can produce more efficient packing at the cement paste-aggregate particle interface, reduce the amount of bleeding and form a denser, more homogeneous initial transition zone microstructure and also a narrower transition zone. The acceleration of PC hydration in the presence of substitution materials is apparent from the higher rates of heat evolution but also from nearly the same or even higher content of $Ca(OH)_2$ determined at very early times of hydration than the expected amounts due to the consummation of arising hydroxide by pozzolanic reactions [16]. Pozzolanic reactions improve material strength by the formation of additional binding phases (C-S-H, C_4AH_{13}, C_3AH_6 and C_2ASH_8) [19–22]. The contribution of particular factors differs according to the activity of the used substitution materials, their replacement level but also the curing time. For example according to [16] dealing with MK blended cements, whilst the filler effect shows immediately, the acceleration of PC has its major impact within the first 24 h and the maximum effect of the pozzolanic reaction occurs between 7 and 14 days. The main contribution of SF as highly active pozzolan to concrete strength at normal curing temperature takes place from about 3 to 28 days. After this time, the effect of SF on strength development becomes negligible. Unlike the MK and SF, reactivity of BFS is much lower and its contribution to the strength gain was demonstrated at medium and later ages of hydration.

Majority of published studies focus on the binary or ternary systems, whereas only few researches deal with the replacement of cement by three supplementary materials together. The reason why we have studied the four compound systems is the synergic effect of materials that can moves the barriers and bring other improvement of final material properties. The following SCMs with different reaction activity have been selected to this goal: BFS, SF and MK.

In our previously published paper [23], evaluation of pozzolanic activity and heat profiles of 2 days samples from the referred four compounds systems using isothermal calorimetry and thermal analysis were already discussed. It was proved that the suitable combination of SF, BFS and MK can prevent from the decrease of strength due to the partial replacement of PC. On the contrary, significantly higher values of compressive strength than the values announced in the number of studies devoted to binary or ternary systems may be attained, which makes these materials promising for the use as e.g. high performance cements. In addition, the formation of phases resistant to higher temperatures (such as tobermorite, C-A-S-H) indicates their possible applications in hydrothermal conditions of deep geothermal wells. Present article continues to study referred systems up to 365 days and investigates the correlation of porosity, compressive and flexural strength.

2. Experimental

The following initial materials were used to prepare the samples: Portland cement (CEM I 52.5 N, Holcim (Slovensko), a.s., Slovakia,), ground granulated blast-furnace slag (Kotouč Štramberk, spol. s r.o., Czech Republic), silica fume (Oravské ferozliatinárske závody, a.s., Slovakia) and metakaolin L_{05} (Mefisto, České lupkové závody, Czech Republic). Composition of particular samples is listed in Table 1. The concept was based on STN-EN 197-1 that limits the content of Portland clinker to 65 wt% for binary blended cements in order to assure necessary $Ca(OH)_2$ quantity for alkali activation or pozzolanic reactions. Keeping into mind, the combination of SCMs to replace PC up to 35 wt% was used. As both, MK and SF, are very reactive since the initial stage of hydration, the decreasing proportion of SF with increasing proportion of MK was chosen in order to establish their influences on the hydration and relating properties of final materials. On the contrary, BFS acts after longer period and its effect does not overlap to the significant extent with those of MK and SF. Its amount was chosen to be the same as MK. The results of chemical analysis and physical characteristic of the used Portland cement and supplementary cementitious materials are shown in Table 2. Chemical analysis of cement was done according to EN 196-2. Mineral composition of the used cement is displayed in Table 3.

The whole experimental procedure of mortar preparation, storage and measurement of flexural and compressive strength was done in accordance with STN EN-196-1. Standard sand satisfying the tests for CEN Reference sand was added to binders in the weight ratio of 3/1. The amount of sulphate ions consumed by metakaolin to form additional ettringite was compensated by gypsum addition [24]. Except referential sample, gypsum was added to each mixture in the amount corresponding to 1.25 wt% of metakaolin content. According to our preliminary results it should assure necessary quantity of gypsum needed for the formation of both ettringites (from C_3A and MK) and also the formation of C-S-H and C-A-S-H from MK by pozzolanic reaction.

Prepared mixtures were homogenized along with the gradual adding of water using the cement mixer. Quantity of water was adjusted to achieve suitable workability of pastes (Table 1). The demand for water was reduced by Plasticizer Stachement® 2353 (Stachema Bratislava, Slovakia). Plasticizer concentration in water of 0.05 vol% was kept constant. After the whole amount of water was added, 10 min of additional homogenization followed. Three prisms with the dimensions of 160 × 40 × 40 mm were prepared from the mortar of each composition. Prisms were covered with moist tissue and foil and kept at laboratory temperature of 20 ± 0.5 °C for 24 h. Demolded samples were immersed in the water and stored at laboratory temperature until the strength