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Rheological characterization of SCC mortars and pastes with changes induced by cement delivery

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ABSTRACT

The present paper deals with the influence of different date produced cement on the rheological properties of SCC mortar/paste mixes. Twelve reference SCC mortar/paste mixes (corresponding to four different types of cement and three different levels of aggregate content) displayed workability variation due to different deliveries of cement, supplied from the same factory. Physical and chemical analysis of cements was carried out for the different deliveries. Mortar and corresponding paste mixes were characterized in fresh, hardening and hardened states. Spearman's correlation coefficient was used to investigate whether there is any kind of association within cement characteristics and between cement characteristics and SCC mortar/paste test results. Existing models for predicting the mini-slump flow diameter and the flow time of pastes were implemented and compared with experimental data. The target paste properties for different fine aggregate content were defined in terms of both empirical test results and rheological parameters, linking mortar and paste properties adequate for SCC.

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1. Introduction

Rheology of cement paste largely dictates rheology of concrete, given a specific aggregate skeleton [1,2]. The key role of the paste is clearly shown by the strong effect on concrete workability of the powder materials, water/powder ratio and of superplasticizer dosage. Furthermore, important phenomena in fresh concrete, like air entrapment and aggregate segregation are determined from paste rheology (along with the stabilising effect of smaller aggregate particles), which provides more or less freedom of motion to aggregates and air bubbles. In spite of the importance of paste rheology, there is no generally accepted procedure for its study. Therefore, direct comparison between different works and the definition of rheology-based acceptance criteria for SCC paste is often difficult. Ultimately, it would be desirable to predict concrete behaviour from paste characteristics. Tests on paste are easier to carry out and require less material. Although rheological tests require more expensive equipment and specific training, they allow a more fundamental and precise characterization of cement suspensions.

The rheology of a superplasticized cement suspension is mainly controlled by: interparticle interactions and state of flocculation of particles (mainly determined by type and amount of superplasticizer added, but also mixing power and history); particle packing (determined by particle size distribution); and type and amount of hydration products formed (mainly determined by cement chemistry and fineness but also cement–superplasticizer interaction). The rheology of superplasticized cement paste, mortar or concrete can be affected by various factors [3–6], namely,

- the amount, the chemical composition and the molecular structure of the admixture;
- the chemical composition of cement (especially C_3A content and availability of soluble sulfates during the workable period);
- the specific surface of cement;
- the presence of mineral additions or of other types of admixtures;
- technological aspects such as mixing power and instance of admixture introduction;
- environmental aspects such as humidity and temperature.

It is therefore difficult to ascertain the main factors and interactions existing between the different components in a superplasticized cement suspension which is further complicated by the ongoing hydration reactions of cement.

According to BIBM et al. [7] all cements which conform to EN 197-1:2001 can be used for production of SCC. Generally, cement is seen as one of the constituent materials of concrete with less

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variation due to a stringent quality control during production. However, recent studies have shown that cement variations have a greater effect on workability and on early reactions of concrete than is generally thought [8–10]. Kubens and Wallevik [10] found that the effect of the production date of cement on rheology was only slight in mixtures without admixtures but more prominent in mixes containing dispersing admixtures. Yield stress results of blank, polycarboxylate ether and melamine mixes (7 samples), containing CEM I 52.5 R, exhibited coefficients of variation of 15%, 44% and 39%, respectively. A similar trend was observed for mixes containing other cements (three CEM I 42.5 R from different producers, and one CEM II/B-S 32.5 R) [10]. Juvas et al. [8] found similar conclusions when comparing the spread flow results of mortars without admixture, containing a typical melamine plasticizer and a polycarboxylate plasticizer, from 50 samples of daily collected CEM I 52.5 R cement type. Larger variations were observed with mixtures containing admixtures when compared to plain mixtures.

The rheology of plain mortar mixtures, with a water/cement ratio of about 0.50, is controlled largely by water [11]. In these mixtures, the cement grains are not uniformly dispersed throughout the water but tend to form small flocs which trap water within them. By adding water these cement flocs are kept apart influencing the rheology of the paste [11]. Later in time, the first hydration products begin to interfere with the unrestricted movement of the cement flocs. Dispersing admixtures are used to both break up the flocs and to maintain the dispersion in a way that causes the cement particles to distribute more uniformly throughout the aqueous phase, reducing the yield stress value for a given water content and so increasing the fluidity of the mix. Not only has the fluidity increased, but more sites on the surface of the cement grains are available for interaction with superplasticizer and hydration [12]. If water is removed from the system, lowering the water/cement ratio to 0.3–0.4, like in the case of mixtures mentioned above [8–10], the fluidity can be reduced back to what it was before the addition of admixture. However, the average inter-grain distance will also reduce, so less hydration will be necessary before the space between the cement grains is filled with hydration product to give setting and strength development [12]. Finally, there will be less void space, not filled with any hydration product, resulting in fewer capillaries and therefore improved final strength and better durability [12]. In conclusion, the mixtures containing superplasticizers are more complex systems and at low w/c ratios a small difference in the dispersion effect of the admixture changes the fluidity remarkably.

Wallevik et al. [9] reported that the fluctuations observed in mortar, due to cement variations, can also be observed at the concrete level. It is suggested that the influence of cement variations on concrete properties can be more or less pronounced depending on cement content in the concrete mixture [9]. In the case of self-compacting concrete, a high-range water reducer must be incorporated and often mixtures have a higher content of cement when compared to conventional concrete mixtures. For these reasons, cement variations have become an important factor when discussing the robustness of SCC production.

A methodology was developed for the design of mortar mixtures which are adequate for SCC [13]. This methodology was developed in three phases: first, the experimental phase conducted according to a central composite design; second, the statistical analysis and model fitting of data collected during the experimental phase and, third, the numerical optimization of mixture parameters using the models derived in the previous phase. Contour plots and interaction diagrams representing the range of mixture parameters where SCC can be found were obtained for each combination of cement type + mineral addition + superplasticizer [13]. This methodology was used to design the SCC mortar mixes investigated in the present study. A central composite design (2^4 factorial statistical design augmented with 8 axial runs plus 4 central runs) was used to establish numerical models relating mixture parameters to spread flow (Dflow), flow time (Tfunnel) and compressive strength (at 28 days) of mortar. SCC mortar mix proportions were established based on the following mixture parameters: water to powder volume ratio (Vw/Vp); water to cement weight ratio (w/c); superplasticizer to powder weight ratio (Sp/p); sand to mortar volume ratio (Vs/Vm). The derived numerical models were used to find optimized solutions within the experimental domain, i.e. mixtures which exhibit both a spread flow of 260 mm and a flow time of 10 s, similar to values recommended by Okamura et al. [14].

The three main goals of the current study are: first, to assess how large the fluctuations on fresh mortar/paste properties can become when a new delivery of cement is used; second, to identify the constituents in cement which might have caused the fluctuations in mortar/paste properties and, third, to verify if the different test results obtained on pastes correlate with each other, in particular, empirical and rheological test results. To accomplish this, twelve SCC mortar/paste mixes (corresponding to four different Portland cement types, namely, CEM I 52.5R, CEM I 42.5 R, CEM II/A-L 42.5 R and CEM II/B-L 32.5 N, and three different levels of aggregate content) were characterized in the fresh, hardening and hardened states, for eight cement deliveries with different production dates. These mortars also incorporated limestone filler, CEN standardized sand and a polycarboxylate type superplasticizer. The testing programme included standard tests on cements, physical and chemical analysis of cements, empirical tests on SCC mortar and pastes, rheological tests on SCC pastes, experimental packing density and semi-adiabatic tests on SCC pastes.

2. Experimental programme

2.1. Materials characterization and mix proportions

The mortar and paste mixes investigated in this study were prepared with cement, a mineral addition (limestone filler), reference sand conforming to CEN EN 196-1:2006 and tap water. The chemical and physical properties of the different cement types used (first delivery) and limestone filler are presented in Table 1. The mean particle size of limestone filler was 4.5 μm. A commercially available polycarboxylate type superplasticizer (Sika Viscocrete 3000®) was used having a specific gravity of 1.05 and 18.5% solids content. Reference sand was a siliceous round grain natural sand (0.08–2 mm) with a specific gravity of 2.57 and an absorption value of 0.68% by mass.

The mix proportions of mortar and paste, for each cement type, were established based on the mixture parameter values presented in Table 2. Mortar mix proportions were adjusted to attain similar fresh properties (Dflow = 260 mm and Tfunnel = 10 s), for each cement type, in a previous study [13]. As can be observed in Table 2, in mortar mixtures exhibiting the same Vs/Vm and w/c ratios (see cases with $Vs/Vm = 0.5$ and $w/c = 0.45$), the Sp/p and Vw/Vp had to be adjusted when changing cement type to attain similar mortar fresh properties. Vw/Vp values did not change significantly but Sp/p varied considerably with cement type.

2.2. Mixing sequence and testing sequence

The mortar and paste mixes were prepared in the laboratory in 1.4 and 1.25 l batches, respectively, and mixed in a two-speed mixer complying to NP EN 196-1:2006. The mixing sequence consisted of mixing sand and powder materials (or only powders, in the case of pastes) with 0.81 of the mixing water during 60 s, stopping the

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