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## Mix design factors of self-consolidating cement paste affecting the magnitude of variations in rheological properties induced by the addition time of PCE-superplasticizer



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#### HIGHLIGHTS

• Changing adding time of the superplasticizer changes the rheology of cement paste.

• The change in rheological properties strongly depends on the SP - VMA combination.

• Limestone filler reduces sensitivity of rheology to a change in SP adding time.

• Fly ash and silica increase sensitivity of viscosity to a change in SP adding time.

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### ABSTRACT

Robustness, which is defined as the capacity of a mixture to tolerate changes and variations in materials and procedures that are inevitable with production at any significant scale, is a key property to expand the use of self-consolidating concrete (SCC). Typically for robustness studies, the most investigated parameter is a variation in the water content. This paper evaluates the effect of the adding time of superplastizer (SP), which was either incorporated in the mixing water or delayed for 2.5 min, on the rheological properties of cement pastes with SCC consistency. Two different SCC mix design concepts, the powder-type (mixture 1) and VMA-type (mixture 2), were selected for this research. The results show that the selected powder-type mix design is more robust than the VMA-type to a change in addition time of SP. In the next step, different mix design parameters have been evaluated by varying the binder combination, the type of PCE-SP and the addition of viscosity-modifying agent (VMA) to determine which particular parameters cause the largest difference in rheological behavior due to a change in adding time of SP. The results have revealed that the limestone filler appears to have the most beneficial effect on the robustness of the cement pastes.

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#### 1. Introduction

Self-consolidating concrete (SCC) exhibits typically high fluidity and deformability, enabling the concrete to flow through congested reinforcement or in complex formworks causing difficult casting conditions [1]. The initial use of SCC in Japan in the late 1980s was intended to improve the construction speed, ease its placement, and eliminate the need for vibration, resulting in an increase in durability of concrete structures [2,3]. SCC is though much more sensitive to small changes in mix design and in the

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mixing procedure, compared to conventional vibrated concrete. Therefore, robustness is an important aspect when studying and optimizing rheological properties and workability of SCC. The robustness of concrete (or cement-based materials in general) is defined as the capacity of a mixture to retain its performance despite variations in the raw materials and mixing procedures that are inevitable when producing on any significant scale [4–8].

Variations in water content, type of superplasticizer (SP) or viscosity-modifying admixture (VMA), and different mixing procedures, such as the adding time of the SP, mixing duration and intensity, have an effect on the rheological properties and workability of SCC. The water content appears to be a key factor influencing the robustness of SCC. Therefore, many researchers [5–10] investigated robustness by varying the amount of water.

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SCC tolerating a change in water content of up to  $5-10 \text{ l/m}^3$ , while still meeting certain performance criteria, has been defined as a robust SCC in the European Guidelines for SCC [11]. Some researchers have reported more robustness of SCC with higher water-tocement ratio (w/c) [8,9]. On the other hand, sometimes it is reported that the robustness of SCC decreases when increasing w/c [10]. Usually, the presence of VMA increases the robustness of SCC [5,9,12–14], especially when focusing on stability, as the VMA is believed to counter moisture content variations in the sand [15]. However, some results show that SCC robustness decreases when employing some types of VMA [16]. Presence of supplementary cementitious materials or mineral fillers, such as fly ash (FA), silica fume (SF) and limestone filler (LF) have been reported to improve the robustness of SCC [17,18].

Mixing time [19-21] and the adding time of admixtures [22–27] also have significant effects on rheology and workability of SCC. Studies illustrated that, with increasing mixing energy, the slump flow increases up to an optimum mixing time, after which it decreases due to overmixing [19–21]. For superplasticizer, it has been shown that their adsorption (or consumption) on Portland clinker particles is mainly governed by the cement composition (especially the  $C_3A$  content), surface charge, the fineness, and the alkalinity of the cement [25-30]. Some researchers investigated the effect of the adding time of SP (simultaneous and delayed addition to water) [22,31–37]. By delaying the addition of the SP to the concrete, a lower amount of molecules get trapped in AFt, AFm, or early C-S-H phases formed at or shortly after initial contact between cement and water [35-37]. Therefore, the yield stress and plastic viscosity of cement paste at early age are lower for the same quantity of admixture in case of delayed addition. Furthermore, VMAs can substantially reduce the effectiveness of PCE, due to competitive adsorption, dependent on the nature of the VMA [38,39].

In this paper, the delayed addition of PCE SP is investigated. For a VMA-type mix design reported in a previous paper [40], it has been observed that delaying the addition of the SP in cement paste with SCC consistency has a larger effect on the yield stress and plastic viscosity than a change in water equivalent to  $10 \text{ l/m}^3$  in the corresponding concrete. This paper studies the sensitivity of cement paste mixtures to a delayed addition of SP, with varying types of supplementary cementitious materials or mineral fillers, water-to-powder ratio (w/p), presence or absence of VMA and with two different types of SP.

#### 2. Experimental program

#### 2.1. Materials

#### 2.1.1. Cement, supplementary cementitious materials and filler

ASTM C 150 type I/II Ordinary Portland cement (OPC, density ( $\rho$ ) = 3160 kg/m<sup>3</sup>) was used in this project. Silica fume (SF,  $\rho$  = 2300 kg/m<sup>3</sup>) and class C fly ash (FA,  $\rho$  = 2700 kg/m<sup>3</sup>) were utilized as supplementary cementitious materials (SCM), while limestone filler (LF,  $\rho$  = 2700 kg/m<sup>3</sup>), consisting of more than 98% CaCO<sub>3</sub>, was used as mineral filler. The cement, limestone filler, silica fume and class C fly ash are commercial products available on the local market.

#### 2.1.2. Chemical admixtures

Two different PCE-based SP were applied. SP 1, used in mix design 1, showed a larger decrease in slump flow with time but was more efficient compared to SP 2. The SP for mix design 2 (SP 2) had relatively long workability retention. Although the study on time-dependency of fresh properties of these mixtures is out of the scope of this paper, the mini-slump flow values at 60 min

after water addition were 210 mm for mix design 1, and 285 mm for mix design 2, showing the difference in mini-slump flow retention between both admixtures. Both SPs are commercial products from two different manufacturers. SP 1 and SP 2 had a density of 1.032 kg/l and 1.085 kg/l, respectively, while their solid contents were 26% and 42% respectively. For mix design 2, a VMA was used to assure stability. The VMA was selected from the same manufacturer as the superplasticizer to prevent compatibility issues. Similarly, for one mixture (2b-6, see further), SP 1 was combined with a different VMA, compatible with SP 1. No air-entraining agents were used in this research project.

#### 2.2. Rheological measurements

The Anton Paar MCR 302 Rheometer (Fig. 1) is a rheometer based on the principle of concentric rotating cylinders. The inner cylinder rotates at different velocities, while the outer cylinder remains stationary. The resulting torque is registered at the inner cylinder. Sandblasted cylinders were used for cement paste to minimize slip and prevent the formation of a lubricating layer. The sandblasted configuration has the following dimensions: the inner cylinder radius (R<sub>i</sub>) measures 13.33 mm, the outer cylinder radius (R<sub>o</sub>), 14.56 mm and the height (h) is 40.00 mm. The temperature of the sample in the rheometer is kept constant at 23 °C.

The following testing procedure was employed to each sample to determine the rheological properties of cement pastes with SCC consistency. At the start of each test, the cement paste is pre-sheared for 60 s at the maximum shear rate employed during the test, which is  $100 \text{ s}^{-1}$ . This time period was sufficient in most cases to assure equilibrium of the measured torque [41–43]. However, if equilibrium was not achieved, the corresponding data points were eliminated. After the pre-shearing period, the cement paste is subjected to a stepwise decrease in shear rate from 100 to  $10 \text{ s}^{-1}$  in 10 steps. The testing procedure is shown in Fig. 2.

The large majority of cement-based fresh pastes can be considered as Bingham materials (Eq. (1)) [41].

$$\tau = \tau_0 + \mu_p \cdot \dot{\gamma} \tag{1}$$

where  $\tau$  is the shear stress (Pa),  $\tau_0$  is the yield stress (Pa),  $\mu_p$  is the plastic viscosity (Pa s), and  $\dot{\gamma}$  is the shear rate (s<sup>-1</sup>). For some of the mixtures, the modified Bingham model was used because non-linear, shear-thickening rheological behavior was observed



Fig. 1. Anton Paar MCR 302 Rheometer.

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