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Influence of Mixing Procedure on Robustness of Self-Consolidating Concrete

by

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16. Abstract

Self-Consolidating Concrete is, in the fresh state, more sensitive to small variations in the constituent elements and the mixing procedure compared to Conventional Vibrated Concrete. Several studies have been performed recently to identify robustness of SCC and to develop solutions to increase the robustness of SCC. Ghent University obtained a major research project from the Research Foundation in Flanders (FWO) to investigate fundamentally robustness of SCC and to identify potential solutions in the form of alternative materials to enhance robustness.

In the present research project, Missouri S&T extended the research at Ghent University by investigating the influence of the mixing procedure on the robustness of SCC. The project was split into four tasks. In a first task, the sequence of adding the constituent elements and mixing was investigated by measuring the rheological properties of cement pastes. In a second task, the combined influence of the most significant mix design and mixing procedure parameters were investigated, with particular attention to the mix design parameters which influence the robustness of the cement paste to a change in time of addition of the superplasticizer. In the third task, the results obtained on cement pastes were validated on concrete scale, with focus on the adding sequence of the aggregates and their initial moisture content. In the fourth task, the robustness of thixotropy and loss of workability was investigated on cement paste and concrete scale.

To enhance the application of SCC for the construction and repair of transportation infrastructure, two key concepts are of importance: quality control and consistency. The consistency refers to the mixing operations and transportation of SCC. It is recommended to keep the mixing procedure constant for every SCC produced. This includes the addition sequence of the materials, the mixing time, the mixing speed and the concrete volume (parameter not tested, but it is reflected in mixing energy). The quality control is not only necessary to determine the moisture content of the aggregates, but also for any of the other constituent elements used.

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Final Report

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1 Literature Review on Robustness of Self-Consolidating Concrete

1.1 Introduction

Self-Consolidating Concrete (SCC) is the concrete of the future: it is a highly fluid type of concrete which has the ability to flow under its own weight, fill the required space or formwork completely and produce a dense and adequately homogeneous material without a need for consolidation [1]. In order to combine such a high fluidity with a normal homogeneity, three approaches exist:

- Powder type SCC, in which a superplasticizer (SP) creates the right fluidity and a high amount of fines (typically 550 to 650 kg/m³) prevents the static segregation of the aggregates.
- Viscosity Modifying Admixtures (VMA) type SCC, in which superplasticizers disperse the cement particles to ensure the proper fluidity, but the segregation resistance is obtained by adding a VMA to the mixture. The typical powder content of a VMA type SCC is 350 to 450 kg/m³.
- Combination type SCC, in which intermediate powder content (450 to 550 kg/m³) is combined with the use of a VMA and superplasticizers in order to obtain a mix with the right fresh and hardened properties.

SCC properties no longer depend on the quality of the consolidation and the skills of the craftsmen, but instead, the properties depend more on the mix design, mixing procedure and casting process. SCC is being successfully utilized, mainly in the pre-cast industry. In some European countries, 100% of the pre-cast industry uses SCC technology. However, in the ready-mix industry, the application of SCC is slowed down due to a larger amount of variables that can play a significant role in the flowability of the concrete being delivered, which are mainly extended transportation time and exposure to high and low temperatures.

Another drawback of SCC, slowing down its practical implementation, is the robustness: the sensitivity of the (fresh) properties of the concrete to small variations in mix design, properties of the constituent elements or the mixing procedure. This research project investigates the robustness of SCC mixtures subjected to variations in mixing procedure and addition sequence of the materials. This report contains an extensive literature study on the robustness of SCC, the characteristics of the used materials and testing equipment, and the results on cement pastes with SCC consistency and on concrete.

1.2 Robustness of fresh SCC

1.2.1 Definition of Robustness

The robustness of concrete is the capacity of a mixture to tolerate changes and variations in materials and procedures that are inevitable when producing on any significant scale, and to retain its self-consolidating properties until placing [1]. The robustness of self-consolidating concrete depends on the mix design, the mixing procedure and the application of the mixture. Several practical definitions of the robustness coexist in literature:

a) The probability a mix fulfills the acceptance criteria for a certain application [2].

b) The coefficient of variation (C.O.V.) of a workability parameter when a certain mix design parameter is changed with a certain value [3]. In this definition, the robustness of a mix design increases as the C.O.V. of the workability parameter is smaller.

c) The intervals in which certain mixture composition parameters can vary and still a satisfying mix is produced [4, 5] (Figure 1).

d) The measured change in a workability parameter when a certain mix design parameter is changed with a certain value [4] (Figure 2). Using this type of definition, the robustness of a mix design increases as the measured change of the workability parameter is smaller.

e) The area in a workability box or rheograph surrounding the variations caused by changing a certain mix design parameter with a certain value [3, 4] (Figure 3). In this definition, the robustness of a mix design increases as the measured 'robustness area' is smaller.

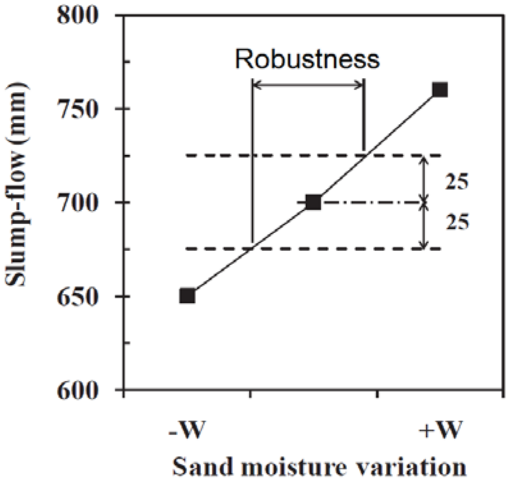

Figure 1. An example of robustness as defined by definition c. [4]

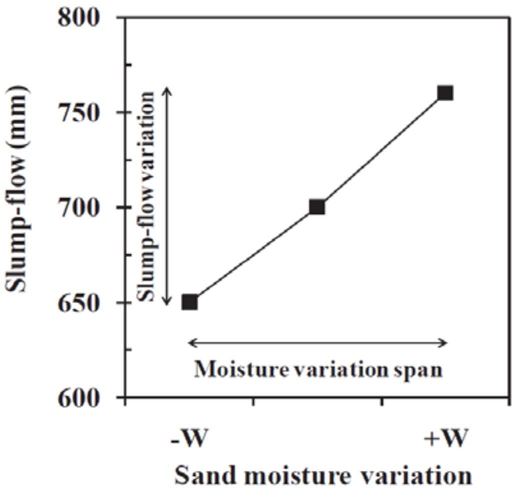

Figure 2. An example of robustness as defined by definition d. [4]

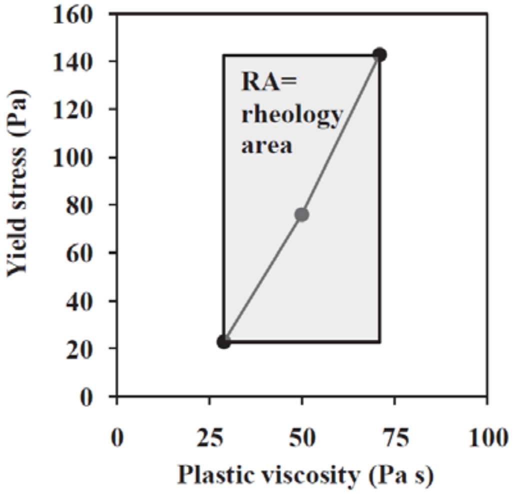

Figure 3. An example of robustness as defined by definition e. [4]

1.2.2 Material Proportions

Weighing Tolerances - Because measuring inaccuracies during weighing are inevitable during the production of concrete on an industrial scale, the standards ACI 117-90 [6] and EN 206-1 [7] provide restrictions on the allowed variations in mixture proportions, listed in Table 1. For powder-type SCC mixtures, Rigueira [8, 9] showed the fresh properties of SCC were mostly affected by the corresponding changes in the cement, water and admixture content. However, the amount of cement and admixture dosages are weighed very accurately in practice, thus most problems are caused by variations of the water content [8]. The EFNARC [10] guidelines on SCC recommend to ensure a mix design able to withstand changes of the water content up to 10 l/m³, which is about 5.5 % of the water weight of an average SCC mixture with 180 l/m³ water [11].

Table 1. Tolerances on material proportions according to ACI and EN.

Component	Limits ACI 117-90 [6]	Limits EN 206-1 [7]
Cementitious materials	1%	3%
Sand	2%	3%
Gravel	2%	3%
Water	3%	3%
Admixture	3%	5%

Variations in Water Content – As fluctuations in the water content dominate robustness, most studies on the robustness of SCC focus on changes in the fresh properties due to variations in the water content. This section summarizes which mix designs withstand best the variations in the water content, according to the literature.

As the amount of powder (cement + SCMs + fillers) in the mix design increases, its resistance to small variations in the water content increases [12-14]. According to Bonen et al. [13], the increase in robustness is caused by an increase in viscosity and density of the paste, but Jonasson et al. [12] and Nunes et al. [14] assume the increase in paste volume is responsible for an observed increase in robustness.

A small amount of VMA incorporated in the mix design increases the viscosity of the paste, and is reported to decrease the sensitivity to small variations in the water content [3, 4, 15-24]. The higher the water-to-powder ratio (w/p), the more the robustness can be increased using VMA, although the required dosage of VMA also increases [24]. In order to maintain the same fluidity, the dosage of superplasticizer needs to be adjusted when using VMA.

According to Li and Kwan [25], the water in fresh concrete can be divided into two parts: the filling water which fills the voids between the solid particles, and the excess water which forms a water film on the surface of the solid particles and contributes to the fluidity of the fresh concrete. As the amount of excess water in a mixture increases by a higher water-to-cement ratio or the use of a less water-demanding powder, the robustness of a mixture is reported to increase [19]. These observations could be explained using the Krieger-Dougherty model [26]: as the amount of excess water increases, the packing density decreases and hence the inclination of the Krieger-Dougherty curve decreases, which corresponds to a lower sensitivity to changes in the amount of excess water.

However, an increase in the water-to-cement ratio is also reported to cause a decrease in robustness [5], which contradicts the theory explained above. Billberg and Westerholm [19] used two different water-to-cement ratios in two powder-type SCC mix designs and Kwan and Ng [5] used three water-to-cement ratios in two powder-based SCC mix compositions.

In order to obtain an SCC mix composition with acceptable segregation resistance, a proper grading curve of the aggregates should be approached to determine the ratio of fine to coarse aggregates [1, 27]. Yet, the grading curve can be adjusted to obtain a higher robustness: an increase of the ratio of fine to coarse aggregates is reported to increase the robustness [5], a decrease of the maximum size of the coarse aggregates results in a higher robustness [13], the use of crushed aggregates instead of rounded aggregates decreases the robustness [12]. Both the specific surface area of the aggregates and the packing density all aggregates can potentially play an important role on the robustness of concrete.

Variations in SP Dosage – When part of the cement in a powder-based mixture is replaced by fly ash or silica fume, a higher resistance against changes in the superplasticizer dosage is observed by Kwan and Ng [28]. Because the density of fly ash and silica fume is lower than the density of cement, a replacement of cement decreases the paste density, and increases the paste volume of the mixture, which supports the hypothesis that the paste volume has a major impact on the robustness of a mixture [14]. However, the replacement of cement by puzzolanic additions also affects the water demand of the mixture.

1.2.3 Material Properties

Cement and Filler Properties - When the same SCC mix design is made with different cement deliveries, the produced mixtures will have large fluctuations in their fresh properties [29-31]. The usage of superplasticizers increases the sensitivity of the fresh properties to the cement properties [31]. According to Nunes et al. [30], the variations in fluidity are mainly caused by differences in cement fineness and the sulfate content of cement. These properties affect the adsorption equilibrium, and ratio of the adsorbed amount of superplasticizers to the surface area of the cement particles [32-35].

SP and VMA Properties - The use of a different superplasticizer or VMA type, or changing admixture producer, can have a major impact on the robustness of a mix composition [3, 13, 15, 17-19, 23, 24, 36, 37]. Polynaphthalene sulfonate (PNS) and polyphosphonic (PPh) based superplasticizers are reported to result in more robust SCC mixtures than polycarboxylate ether (PCE) based superplasticizers [13, 23, 37]. Many different products are referred to as VMA, however not all VMA types have the same influence on the robustness: some types of VMA are more efficient in improving the robustness than others [3, 17-19, 23, 24], and some types of VMA even decrease the robustness of the mix design [3]. Differences in the chemical purity, physical properties, and molecular sizes of superplasticizers and VMA's in between different deliveries probably also have a major impact on the fresh properties of SCC, but less information about this subject is available in literature.

Aggregate Properties - Because fluctuations in the fineness of sand are inevitable during the production of concrete, a good SCC mix design should be able to withstand such fluctuations. In addition to the deviations from the grading curve, small quantities of clay present in the sand have a major impact on the fluidity of SCC [38].

When the same SCC mix design is produced using air-dried sand (0.2% moisture content) or sand with a moisture content of 3% keeping the total amount of water added to the mixture the same, large differences in the slump flow and its evolution in time occur (Figure 4) [39].

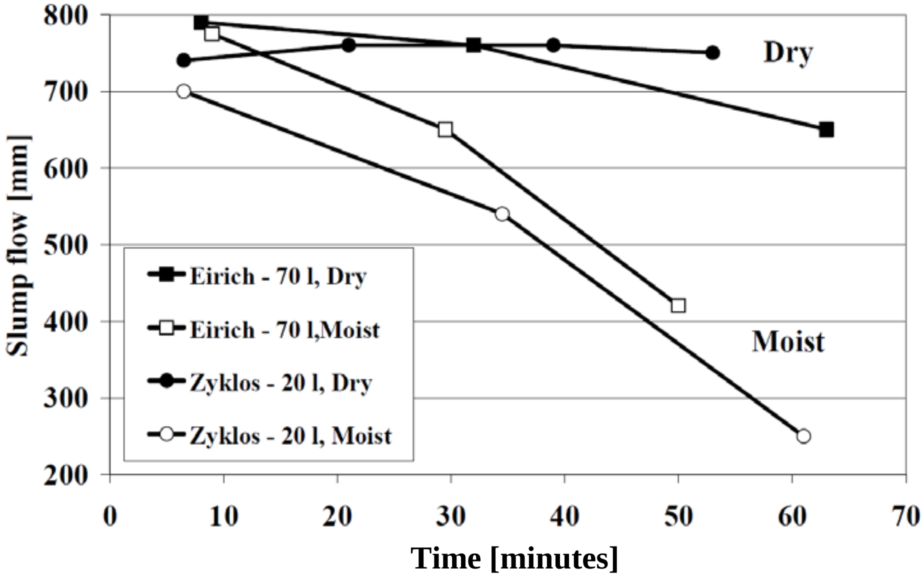

Figure 4. The influence of the moisture content of the aggregates on the fluidity evolution with time of fresh SCC. Independent of the mixer type, the moist aggregates appeared to cause a significant decrease in slump flow over time (in minutes, relative to the water adding time). [39]

1.2.4 Mixing Procedure

Besides a higher sensitivity of the workability and rheology to changes in material proportions and properties, SCC is also more sensitive to variations in the mixing procedure. In the following sections, the different mixing parameters influencing the workability and rheology of fresh concrete are described.

Mixing Time - During a mixing process in which first all dry constituents are premixed in the mixer before the water and superplasticizer is added, the fresh concrete passes three mixing stages according to Lowke [40, 41] (Figure 5):

- First, the water is dispersed and the fluid bonds between particles increase the power consumption. Clusters of powder, sand and water are formed inside the mixture [42].
- Once all particles are suspended in the liquid, the clusters are destroyed by the shear stresses in the mixing process [42] and the power consumption decreases asymptotically until a plateau is reached. The more coarse aggregates available, the faster the decrease of the power curve during this stage of the mixing process [43].
- When mixing continues after reaching the optimum dispersion of powder and superplasticizer, the concrete is overmixed. More clusters of cement and filler particles are breaking, resulting in an increase of the total fine particles surface and thus the water and superplasticizer demand [40, 41]. Other possible mechanisms for the decrease in flowability are the abrasion of first hydration products [40, 41] and the breaking of aggregates [40, 41, 44].

Based on measurements of the power consumption during the mixing process, the stabilization time is defined as the time at which the optimum dispersion is reached and the power curve reaches a horizontal asymptote (as illustrated in Figure 5 [40]). A more practical definition is the time at which the slope of an exponential decreasing curve, fitting the power consumption vs. time, reaches a value of -4 . 10^{-4} s^{-1} [45, 46] (Equation 1 and 2). Dils et al. [44] used the same practical definition, but with a slope of -6 . 10^{-4} s^{-1}.

$$\boldsymbol{P(t) = P_0 + P_1 * \exp\left(-\frac{t}{t_1}\right)} \qquad \text{(Eq. 1)}$$

$$\Rightarrow \boldsymbol{t_s = -t_1 * \ln\left(4 \cdot 10^{-4} * \frac{t_1}{P_1}\right)} \qquad \text{(Eq. 2)}$$

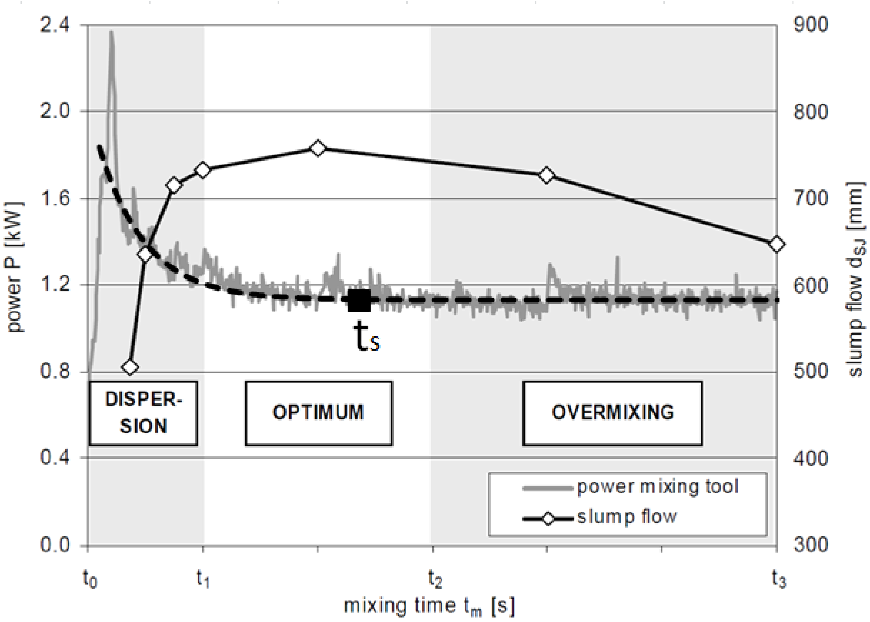

Figure 5. The mixing stages and stabilization time [40].

Mixer Type and Mixing Speed - Many different types of mixers exist [47, 48], and with the same ingredients, each type of mixer produces a different concrete [44]. The volume of concrete and the applied mixer speed or energy also influence the fresh properties of the produced concrete. The observed differences in fluidity for mixers operating at higher shear rates are caused by the increase in the rate of superplasticizer adsorption and powder dispersion [40, 41, 46, 49-51], shortening the dispersing phase and increasing the superplasticizer demand. According to Takada and Walraven [49], more fine air bubbles acting as a lubricant are mixed into the concrete at high shear rates. Dils et al. [44] explained a decrease of the flowability at high shear rates as a result of an increase in the water demand due to the breaking of aggregates.

As illustrated in Figure 6 [40], the effect of the mixing speed on the flowability of SCC mixtures with a fixed mixing time depends on the shift in stabilization time. Due to the higher forces in the mixture at high mixing speed, stronger clusters of particles can be broken and the maximum flowability of the mixture decreases [40, 41, 51] if the SP quantity remains constant. Because the kinetic energy in the mixed concrete increases with the square of the velocity and an increase of the coarse aggregates size and content [41], overmixing occurs more often in concretes made with coarser aggregates and intensive mixers.

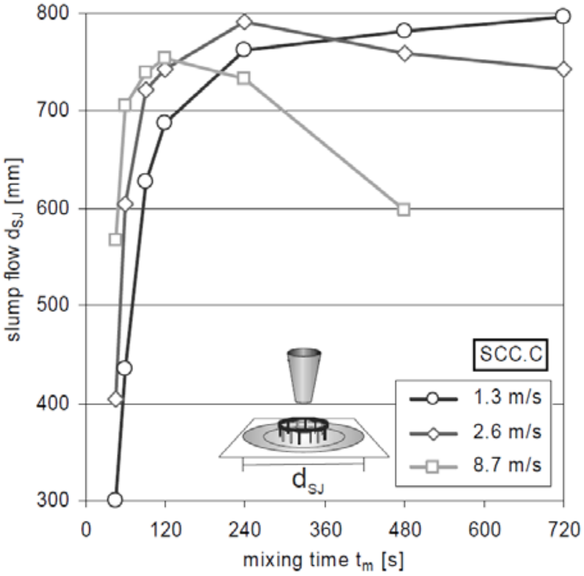

Figure 6. The influence of the mixing speed (expressed as the velocity of the rotor of the mixer) on the slump flow after mixing [40].

Mixing Sequence and Addition Time of the Superplasticizer - The mixing sequence and addition time of the superplasticizer are reported to have a major influence on the rheological behavior of the mixture. A delayed addition of the superplasticizer increases the fluidity [52], decreases the Visual Segregation Index [53], decreases the thixotropic behavior of a mixture [54], but has no significant influence on the hardened properties [52]. Differences between the delayed and direct addition of PCE type superplasticizer are larger when the dose of superplasticizer and the temperature of the mixture decrease [54]. When the superplasticizer is premixed in the water, part of the superplasticizer is included in the organic mineral phase on the cement surface during the first hydration peak by intercalation [55, 56], reducing the amount of superplasticizer available to disperse the cement particles. The delayed addition of superplasticizer eliminates this effect and thus creates a more fluid mixture.

1.2.5 Temperature

Last but not least, the temperature also influences the fresh behavior of the concrete [57]. Temperature changes affect the viscosity of the water in the concrete, the rate at which hydration reactions occur, and the sulfate dissolution rate – a competitive adsorbent with most superplasticizers [32, 33, 57, 58].

2 Research Needs and Goals of the Research Project

The practical application of Self-Consolidating Concrete (SCC) is limited due to large sensitivity of SCC properties to small variations in constituent elements proportions or properties. Robust mixtures have low sensitivity to those variations and errors, causing more stable properties of SCC. The major parameters influencing the fresh properties of SCC are found to be the water-to-powder volume ratio (V_w/V_p), with powder volume defined as the total volume of fines and the superplasticizer to powder volume ratio (SP/V_p).

Currently, the literature on the robustness of SCC mainly focuses on the variations in quantity of constituent elements (such as w/p) and the influence of the mixing energy on the properties of fresh concrete. The effectiveness of different chemical admixtures, depending on their addition time, is also documented, but less information is available on the influence of the addition sequence of constituent materials and mixing procedure on the rheological properties of SCC: yield stress and plastic viscosity.

This project aims to extend the knowledge on the influence of the mixing procedure and addition sequence of constituent elements on the rheological properties. Specific parameters investigated are:

- Addition time of the superplasticizer (with the mixing water or delayed)
- Mixing energy, by varying mixing time and mixing speed
- Addition sequence of aggregates

The influence of these three parameters is compared to the influence of a change in w/p equivalent to 10 l/m^3 of concrete and validated on different mix designs: powder type versus VMA type. The influence of these changes on the rheological parameters and their variation in time due to thixotropy and workability loss is monitored.

As main goals, the research team aims to better understand the mechanisms involved that affect robustness of SCC and to identify specific materials, mixing procedures and addition sequences that can enhance the robustness of SCC. As a result, good understanding and control of robustness will enhance practical applications of SCC.

3 Project Overview

This project involves collaboration between the Missouri University of Science and Technology and Ghent University in Belgium. The following tasks were executed to investigate the influence of mixing procedure and addition sequence of materials on the rheology of SCC:

3.1 Task 1: Robustness of Cement Pastes due to Changes in Mixing Procedure

In this task, the robustness of the mixing procedure is investigated on two types of mixtures: powder-type and VMA-type cement paste mixtures. Rheological measurements have been performed at 15 min. For verification, a mini-slump flow test was executed immediately after mixing.

Based on the reference mix designs and reference mixing procedure, the following parameters were investigated:

- Repeatability of mixture properties to have an indication on the intrinsic variations, enabling the identification of significant changes by other parameters.
- The amount of water: variations in water contents corresponding to a change of ± 5 and ± 10 l/m^3 in concrete. This enabled the research team to see if the changes in other parameters were significant compared to a change in water content.
- Different deliveries of cement from two different cement producers.
- The amount of SP: ± 5 and ± 10% compared to the reference value.
- Addition time of SP: 100% with the mixing water, 50% with mixing water+50% delayed, and 100% delayed addition.
- Influence of mixing speed: low, medium.
- Influence of mixing time: low, medium and high.

3.2 Task 2: Combining Variations in Constituent Elements and Addition Time of SP

Part A: The most important mix design and mixing procedure parameters that affect the robustness of SCC pastes are found to be the amount of water and the addition time of SP. In this task, the variation in the amount of water is combined with the variations in addition time of the SP or the mixing time to investigate their combined effect. This has been attempted to find out how the addition time of SP and the mixing time can enhance the robustness of SCC pastes to variations in water content.

Part B: In this part, a more detailed study on how to reduce the sensitivity of the plastic viscosity to a change in addition time of SP has been performed, by varying the following parameters:

- Binder composition: 100% Type I Portland cement, Class C fly ash, silica fume and limestone filler
- Two commercially available polycarboxylate-ether superplasticizers (SP) from two different manufacturers
- The addition of one commercially available viscosity-modifying agent (VMA), compatible with the used SP.

3.3 Task 3: Robustness of Mixing Procedure and Addition Sequence of Materials on Concrete

Task 3 partly served to validate on concrete scale the results obtained on the cement pastes, but also allowed the investigation of the addition sequence and initial conditions of the aggregates. All concretes were prepared in the Eirich intensive mixer, which allows controlling the speed of the rotor and the speed of the pan. The following parameters were investigated on concrete scale:

- Variations in water content of ± 10 l/m^3 compared to the reference
- Variations in mixing speed
- Variations in mixing time
- Different sequences of adding the materials, specifically focusing on the addition of the aggregates compared to the water, the cement and filler and the SP. The aggregates are below saturated-surface dry (SSD) condition, and the addition sequence influences the amount of water absorbed.

- Variation in initial moisture content of the fine aggregate
- Variation in the type of mixer, for which the same SCC mixture was reproduced in a drum mixer, but the amount of SP was varied to avoid severe segregation.

The investigations on concrete scale have only been carried out on the powder-type SCC mix design.

3.4 Task 4: Influence of Mixing Procedure on Thixotropy and Workability Loss

All results discussed in tasks 1 to 3 are obtained within 15 minutes after the contact between cement and water. This task investigates the influence of all parameters discussed above on the evolution of yield stress and plastic viscosity with time, up to 60 min for cement paste and 90 min for concrete. In addition, static yield stress measurements were performed on the concrete mixtures after different resting times to characterize the thixotropic properties of each concrete.

4 Materials and Testing Equipment

4.1 Characteristics of Materials

4.1.1 Cement

In all mixtures ASTM C 150 Types I or I/II Ordinary Portland Cements (OPC) were utilized. Cements from two different producers located in Missouri were incorporated in the cement pastes. The Type I cement from one producer showed adequate repeatability (see section 5), but between two deliveries, a significant change in SP demand was noticed. The research was further continued with a Type I/II cement from another producer. A change in delivery still led to variations in observed properties, but to a lesser extent. With a change in delivery, specific mixtures were repeated to eliminate variations caused by the cement delivery from the investigated parameters.

4.1.2 Supplementary Cementitious Materials and Fillers

Limestone filler, consisting of more than 98% $CaCO_3$, was used as mineral filler, while silica fume (SF) and class C fly ash (FA) were used as supplementary cementitious materials (SCM). The limestone filler, silica fume and class C fly ash are commercial products available in the Missouri market. The specific gravities of each material are reported in Table 2.

Table 2. Specific gravities of the SCMs and fillers.

	Specific gravity (-)
Limestone filler	2.7
Silica Fume	2.2
Class C fly ash	2.4

4.1.3 Chemical Admixtures

For all mixtures, a polycarboxylate ether (PCE)-based superplasticizer (SP) was used. One of the SPs (SP 2) had relatively long workability retention, while SP 1 was more efficient but showed a larger decrease in slump flow with time. Both SPs are commercial products from two different manufacturers.

Based on the choice of SP, some mixtures required the addition of a viscosity modifying agent (VMA) to assure stability. To avoid compatibility issues, the VMA was chosen from the same manufacturers as SP 2, and was only used in combination with SP 2. No air-entraining agents were used in this research project.

4.1.4 Aggregates

Missouri river sand and Missouri River pea gravel were utilitized in the concrete mixtures. The sand had a SSD-specific gravity of 2.64, and an absorption of 0.6%, a fineness modulus of 2.61 (according to ASTM C125), and it met the ASTM C33 requirements for sand. The pea gravel had a nominal maximum aggregate size of 9.5 mm (3/8"), a SSD-specific gravity of 2.56, an adsorption of 1.125%, a fineness modulus of 5.76, and its grain size distribution was in accordance with ASTM C33 Size Number 8. The pea gravel is a small, rounded aggregate. The choice for this aggregate was made based on availability, restriction of the maximum aggregate size in the ConTec rheometer (max. 15 mm) and the larger margin for stability due to its smaller size. The grain size distributions for the sand and the pea gravel are displayed in Figure 7.

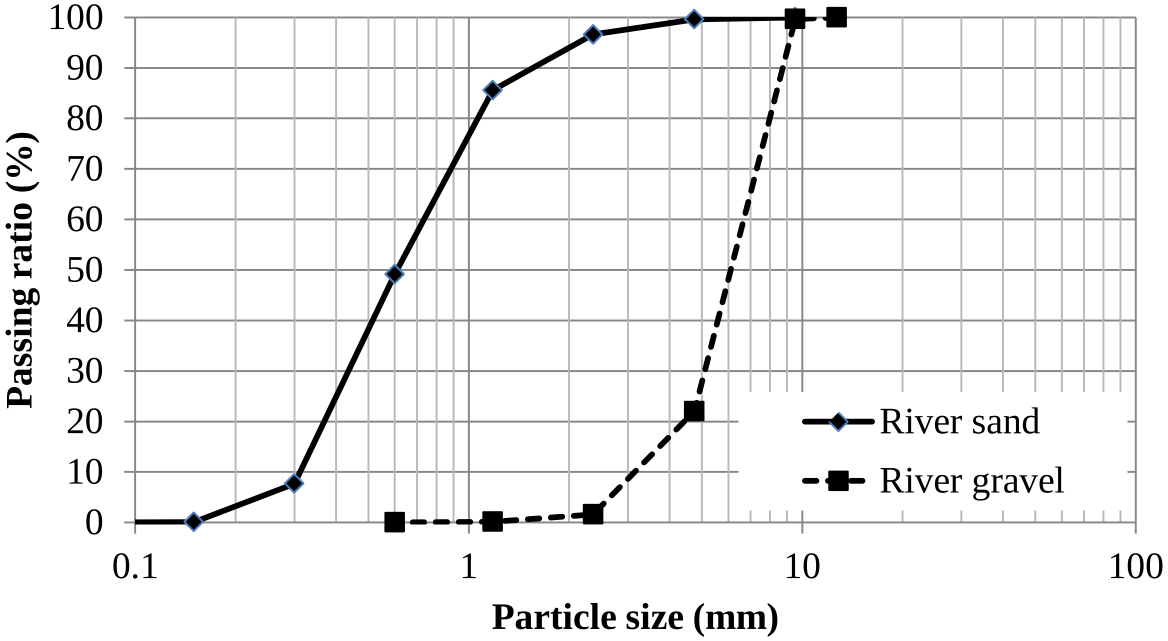

Figure 7. Grain size distribution of the sand and the coarse aggregates.

4.2 Workability Tests

4.2.1 (Mini-) Slump Flow

The simplest and most widely used test method for self-consolidating concrete is the slump flow test [59, 60]. The test is similar to the slump test for vibrated concrete, except that the cone is filled in one layer without rodding, and that the final spread diameter is measured, as an average of two measurements, instead of the slump. The slump flow is related to the yield stress of the concrete.

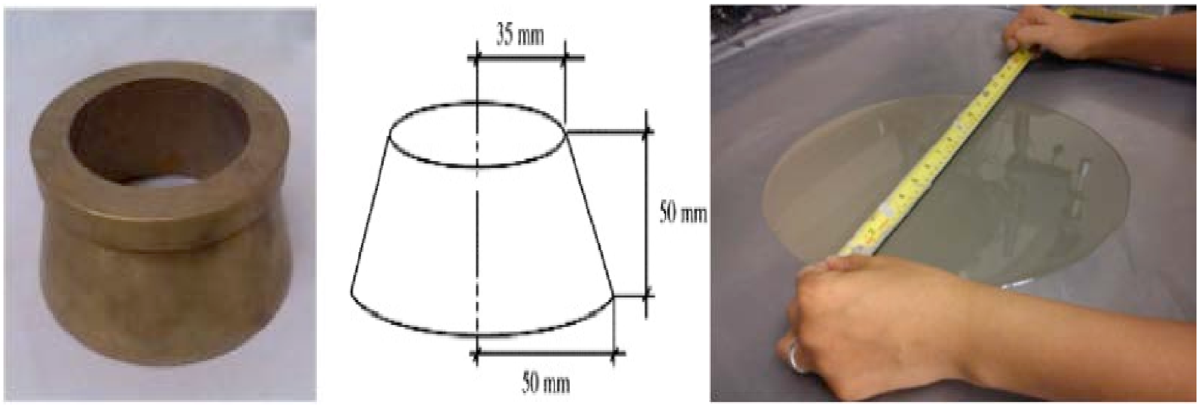

Figure 8. left) Dimension of mini slump cone, right) Determination of the slump flow as the average of two perpendicular measurements of the diameter.

The mini slump-flow is the equivalent test to measure the spread of mortar and cement paste. The mini slump cone has a diameter of 7 cm at the top, 10 cm at the bottom and a height of 5 cm. The mini slump test has only been used for cement paste with SCC consistency, so no external consolidation has been applied. The slump flow is defined as the average of two perpendicular measurements of the final diameter (Figure 8).

4.2.2 V-Funnel

The V-funnel flow time is the period a defined volume of SCC needs to flow through the V-Funnel apparatus and gives an indication of the filling ability of SCC provided that blocking and/or segregation do not take place. For SCC, the flow time of the V-funnel test is related to the plastic viscosity. The funnel is filled with about 12 liters of SCC, and once completely full, the bottom outlet is opened, allowing the concrete to flow out. The V-funnel flow time is the elapsed time (t) in seconds between the opening of the bottom outlet and the instant when the light becomes visible from the bottom, when observed from the top [61].

4.2.3 Sieve Stability

The resistance to static segregation of a fresh self-consolidating concrete mix can be evaluated with the sieve segregation test described in EN 12350-11 [62]. During the test, a sample of fresh concrete is at rest on a sieve and the amount of mortar separated from the mix is weighed and noted as the Sieve Segregation Index (S.S.I.) according to Equation 3 in which the following symbols are used.

m_{ps} = the mass of the receiver and the cement paste or mortar that has passed through the sieve

m_p = the mass of the receiver

m_c = the mass of the concrete poured at the sieve

$$S.S.I. = \frac{m_{ps} - m_p}{m_c} \cdot 100\% \qquad \text{(Eq. 3)}$$

Figure 9. The sieve stability test.

4.3 Rheometers

The influence of variations in constituent elements, mixing procedure and addition sequence of materials is mainly studied by means of rheology. Rheology is a more scientific way of characterizing the flow properties of cement paste, mortar and concrete. The science of rheology is widely spread over different research domains (polymers, food, suspensions, etc.) and it is widely applied in concrete science since the invention of SCC. In this section, the rheometers, testing procedure and data analysis are described.

4.3.1 Anton Paar MCR 302 for Cement Pastes

Description - The Anton Paar MCR 302 Rheometer (Figure 10) is a cement paste rheometer based on the principle of coaxially rotating cylinders. The inner cylinder rotates at different velocities, while the outer cylinder remains stationary. The resulting torque is registered at the inner cylinder.

Two sets of coaxial cylinders were used on cement-paste scale: the smooth cylinders and the sandblasted cylinders. The sandblasted cylinders have the advantage of reducing slip or the formation of a lubricating layer. Except for the cement pastes with lowered water contents, the smooth and sandblasted cylinders deliver similar results. For the smooth inner and outer cylinders, the inner cylinder (R_i) measures 13.385 mm, the outer cylinder (R_o), 14.562 mm and the height (h) is 40.001 mm. The sandblasted configuration has the following dimensions: inner cylinder (R_i) = 13.331 mm, outer cylinder (R_o) = 14.561 mm, and height (h) = 40.002 mm.

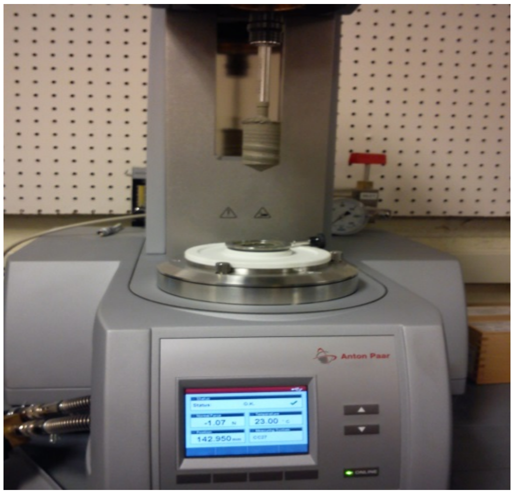

Figure 10. Anton Paar MCR 302 Rheometer.

Testing Procedure - The rheological properties of each cement paste are determined with the Anton Paar MCR 302 using the following testing procedure. 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 s^{-1}. This time period has been proven to be sufficient in most cases to eliminate the effect of thixotropy from the results. After the pre-shearing period, the cement paste is subjected to a stepwise decrease in shear rate from 100 to 2 s^{-1} in 11 steps. Each step takes 5 seconds. The testing procedure is shown in figure 11. However, the data point at 2 s^{-1} was in most cases eliminated from the series due to significant plug flow.

Data Treatment - The rheological properties of cement-based materials are usually characterized with the Bingham model:

$$\tau = \tau_0 + \mu_p \cdot \dot{\gamma} \qquad \text{(Eq. 4)}$$

For this equation, τ is the shear stress (Pa), τ_0 is the yield stress (Pa), μ_p is the plastic viscosity (Pa s), and $\dot{\gamma}$ is the shear rate (s^{-1}). The yield stress is the stress needed to start the flow. This means that applying a stress lower than the yield stress will not cause any flow in the material. The plastic viscosity is the resistance of the material to an increase in flow rate once the yield stress is exceeded. The yield stress and the plastic viscosity are the two Bingham parameters that characterize the flow properties of the studied materials. An example of a Bingham relationship can be found in Figure 12.

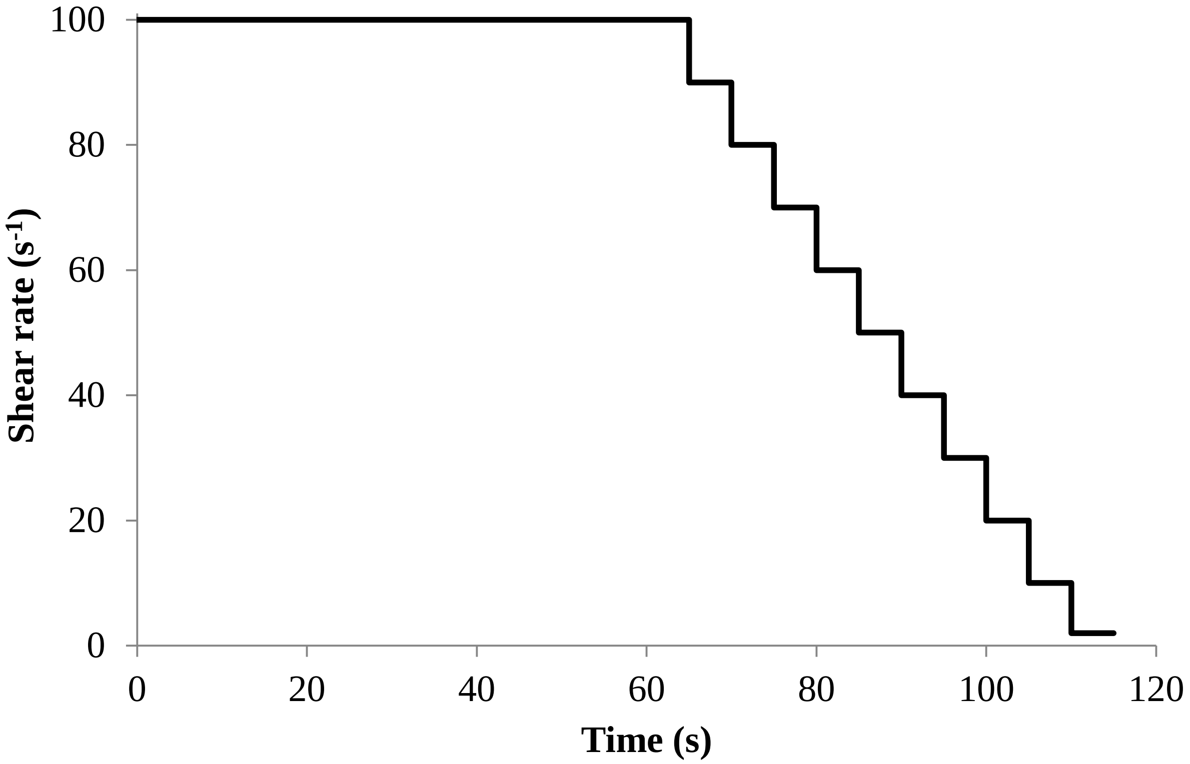

Figure 11. Testing procedure for cement pastes in the Anton Paar MCR 302 rheometer.

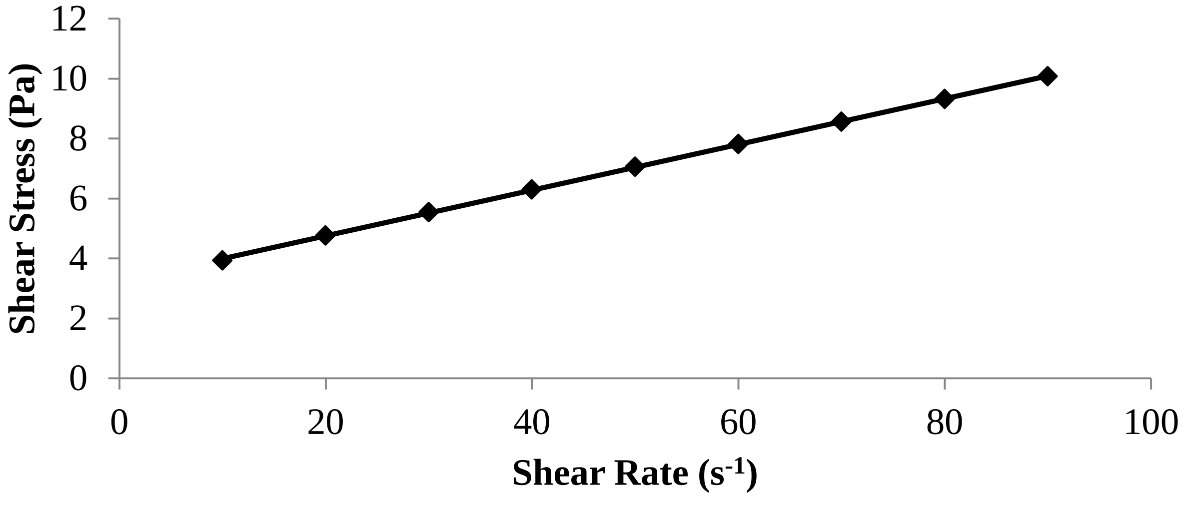

Figure 12. Example of Bingham behavior.

When the rheological measurement is performed with a coaxial cylinder rheometer, torque (T) and rotational velocity (N) are measured. Shear stress and shear rate data must be derived from the torque and rotational velocity data. When the torque is at equilibrium at each shear rate step, the rheological properties can be calculated by means of the Reiner-Riwlin equation. If the torque was not at equilibrium at a certain step, the respective data point was eliminated from the results. The Reiner-Riwlin equation transforms the parameters G and H (eq. 5), defining a linear relationship between torque (T) and rotational velocity (N), into the Bingham parameters (eqs. 6 and 7) [63]. This assumes a laminar, stable flow and no particle movements in the horizontal or vertical direction and all material in the entire gap must be sheared.

$$\boldsymbol{T = G + H.N} \qquad \text{(Eq. 5)}$$

$$\boldsymbol{\tau_0} = \frac{\boldsymbol{G}}{\boldsymbol{4\pi \cdot h}} \cdot \left(\frac{\boldsymbol{1}}{\boldsymbol{R_i}^2} - \frac{\boldsymbol{1}}{\boldsymbol{R_o}^2}\right) \cdot \frac{\boldsymbol{1}}{\ln\left(\frac{\boldsymbol{R_o}}{\boldsymbol{R_i}}\right)} \qquad \text{(Eq. 6)}$$

$$\mu = \frac{\boldsymbol{H}}{\boldsymbol{8\pi^2 \cdot h}} \cdot \left(\frac{\boldsymbol{1}}{\boldsymbol{R_i}^2} - \frac{\boldsymbol{1}}{\boldsymbol{R_o}^2}\right) \qquad \text{(Eq. 7)}$$

For most of the powder-type cement-pastes with SCC consistency, non-linear, shear-thickening rheological behavior has been observed (Fig.13), leading to the application of the modified Bingham model [64]:

$$\tau = \tau_0 + \mu \cdot \dot{\gamma} + c.\dot{\gamma}^2 \qquad \text{(Eq. 8)}$$

For this equation, τ is the shear stress (Pa), τ_0 is the Bingham yield stress (Pa), μ is linear term of modified Bingham model (Pa s), $\dot{\gamma}$ is the shear rate (s^{-1}) and c is second order term of modified Bingham model (Pa s^2).

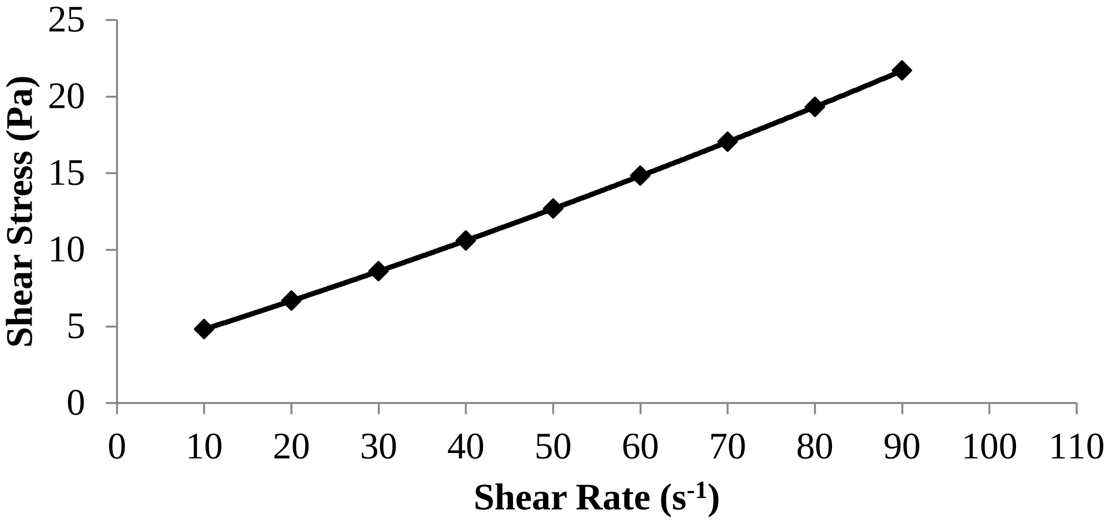

Figure 13. Example of shear-thickening behavior.

The shear-thickening phenomenon requires the application of the modified Bingham model, as the application of the Bingham model under-estimates the yield stress and can lead to apparent negative yield stresses which are physically impossible [65].

Similar to the Bingham model, the Reiner-Riwlin equation can be used to obtain the rheological properties: the intercept G, first order term H and second order term C can be transformed into yield stress (eq. 6), viscosity (eq. 7) (μ) and c-parameter (eq. 9).

$$\boldsymbol{c} = \frac{\boldsymbol{C}}{\boldsymbol{8\pi^3 \cdot h}} \cdot \left(\frac{\boldsymbol{1}}{\boldsymbol{R_i}^2} - \frac{\boldsymbol{1}}{\boldsymbol{R_o}^2}\right) \frac{(\boldsymbol{R_0} - \boldsymbol{R_i})}{(\boldsymbol{R_0} + \boldsymbol{R_i})} \qquad \text{(Eq. 9)}$$

One of the consequences of applying a non-linear rheological model is that the viscosity is dependent on the shear rate. To solve this problem, the reported viscosity values in this report are differential viscosity values, meaning they describe the inclination of the rheological curve at a fixed shear rate, which is chosen at 50 s^{-1}. For the Bingham model, the inclination of the line is the plastic viscosity (μ_p), while for the modified Bingham model, the inclination is μ + 100 c at a shear rate of 50 s^{-1}.

4.3.2 ConTec Viscometer 5 for Concrete

<u>Description</u> - The Contec viscometer 5 (Figure 14) is a wide gap concentric cylinder rheometer with an inner cylinder radius of 100 mm and an outer cylinder radius of 145 mm. In order to prevent wall slip, both the inner and outer cylinders are equipped with vertical ribs. The three dimensional bottom effect is avoided by the design of the inner cylinder, as it is split into two parts. Only the upper part of the inner cylinder registers torque, while the lower part is not connected to the torque sensor.

Figure 14. Contec Viscometer 5.

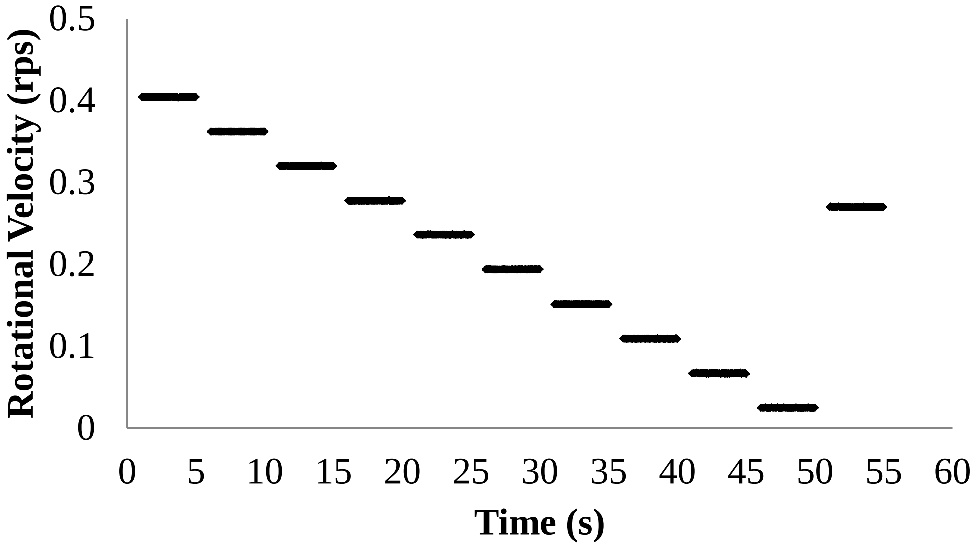

Figure 15. The applied rotational velocity profile in the Contec rheometer.

Testing Procedure - Immediately after mixing, a fixed volume of concrete is poured into the rheometer bucket and inserted into the rheometer. At 10 minutes after first contact of water and cement, the rheological properties were determined. The test started with a preshear period at a rotational velocity of 0.40 rps during 25 seconds, followed by a stepwise decreasing rotational velocity profile, from 0.4 to 0.025 rps in 10 steps of 5 s each, as given in Figure 15.

Data Treatment - After plotting the measured rotational velocity and torque values with time, a visual check of the data can verify if segregation occurred during the experiment and if all the rotational velocity steps are in equilibrium. When the torque measurement during a constant rotational velocity step was not in equilibrium, it was not considered in the analysis.

For all mixtures, the torque – rotational velocity diagram was linear and thus, the Bingham model (eq. 4) was applied. The dynamic yield stress and plastic viscosity were calculated assuming the Bingham model (eq. 4). When plug flow occurred [66], a plug flow correction was performed.

4.3.2 ICAR Rheometer for Concrete

Description - The ICAR rheometer (Figure 16) is a four-bladed vane rheometer with a vane radius of 63.5 mm and a vane height of 127 mm. The container, equipped with vertical ribs, has a radius of 143 mm. The ICAR was only used to perform static yield stress measurements. As the imposed rotational velocity was sufficiently low, different static yield stress measurements could be performed at different times assuming that the sample remained at rest the entire time.

Figure 16. ICAR rheometer.

Testing Procedure - During the static yield stress measurements, the torque of the vane necessary to obtain a constant rotational velocity of 0.001 rps was measured at 10, 20, 30, 40, 50, and 60 minutes after first contact of cement and water. Once a maximum peak torque was measured, the rotational velocity was stopped, allowing the buildup of an internal structure in the sample.

Data Treatment - A typical example of the torque measurement is given in Figure 17. The static yield stress $\tau_{0,s}$ is assumed to be the peak yield stress of the diagram, calculated assuming the concrete between the vanes behaves like a rigid cylinder and all the shearing happens across a cylindrical surface surrounding the vane [67]. The static yield stress is obtained by dividing the maximum torque by $2\pi R_i^2 h$. In order to avoid the influence of experimental scatter, the peak torque was calculated as the peak of a polynomial fit around the experimental data surrounding the peak torque.

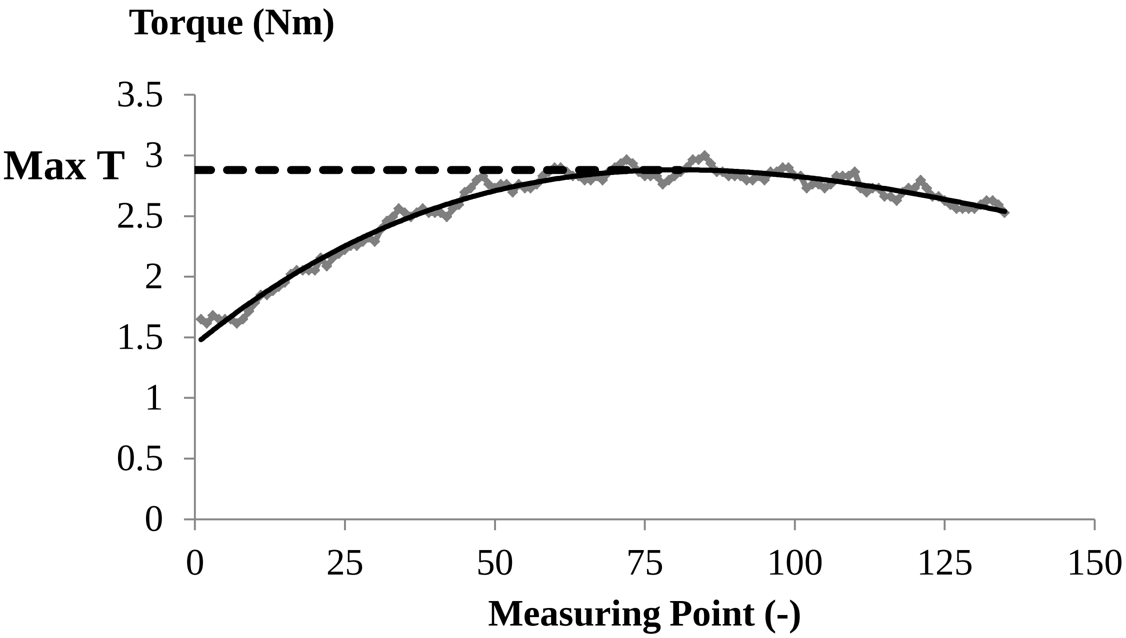

Figure 17. A typical example of a static yield stress measurement.

4.4 Mix Designs and Mixing Procedures

Self-consolidating cement paste and concrete are sensitive to small changes in the mixing procedure (e.g. an unnoticed change in addition time of SP or change in sand moisture content), especially compared to normal concretes. This project evaluates the influence of various mixing procedures on the robustness of self-consolidating cement paste and concrete, described below.

4.4.1 Cement Pastes

Various parameters, such as the amount of water, amount of superplasticizer (SP), addition time of SP, mixing time and mixing speed have been examined on the two different types of self-consolidating cement pastes (powder-type and VMA-type).

Reference Mixing Procedure – The preparation of the cement pastes was performed in a small Hobart mixer. All mixing occurred at the lowest speed available and the contact time between cement and water is taken as reference time (t_0). The reference mixing procedure consisted of homogenizing the dry materials (cement, SCM and/or fillers), mixing with water for 1 min, scraping the bowl of the mixer for 1 min and mixing for an additional 30 s. The SP is added (100% delayed) and the paste is mixed for 2 additional minutes, followed by some minor scraping (30s) and everything is homogenized during the final minute (see Table 3). The total mixing duration is 6.5 minutes.

This reference mixing procedure was employed to investigate:

- Repeatability of the mixing procedure and rheological results
- Influence of the water equivalent to a change of ± 5 and ± 10 l/m^3 of water in concrete
- Influence of different deliveries of cement of different manufacturers
- Influence of the amount of SP, changing with ± 5 and ± 10% compared to the reference value

Variations in Mixing Energy - The change in mixing energy of the cement pastes has been investigated in two ways: by changing the mixing speed and by changing the mixing time. The mixing speed was set at positions “1” and “2” of the Hobart mixer, corresponding to approximately 140 ± 5 rpm and 285 ± 10 rpm.

The second way to vary the mixing energy was to vary the mixing time. For the short mixing time, every mixing step longer than 30 s was divided by two, while for the long mixing time, all mixing steps in the reference procedure longer than 30 s were doubled. The details can be found in Table 3.

Table 3. Different mixing time a) Black as reference, b) Red as short mixing time and c) Blue as long mixing time.

Time	Duration	Action	Addition
-30 s	30 s	Mixing	Dry materials
0 s	1 min / 30 s / 2 min	Mixing	Water
1 min / 30 s / 2 min	1 min	Scraping	
2 min / 1.5min / 3 min	30 s	Mixing	
2.5 min / 2min / 3.5 min	2 min / 1 min / 4 min	Mixing	SP
4.5 min / 3 min / 7.5 min	30 s	Scraping	
5 min / 3.5 min / 8 min	1 min	Mixing	
6 min / 4.5 min / 9 min		Initial slump flow (except for the long mixing)	

Variations in Addition Time of SP - In this project, superplasticizer has been added to the mixtures in 3 different ways. All SP was delayed by 2 min (100% delayed), which was considered the reference, 100% with the mixing water, and 50% of SP with water, 50% delayed (2 min). The addition time of SP with the water was done by physically adding the SP to the mixing water before insertion into the mixture. In select cases, a fourth scenario was explored, by adding the SP just after the insertion of the water. The details of addition time of SP can be found in Table 4.

Table 4. Addition time of SP, a) Black as 100% Delayed (reference), b) Red as 50% SP with water and c) Blue as 100% SP with water.

Time	Duration	Action	Addition
30	30s	Mixing	Dry Materials
0 s	30s	Mixing	Water/ Water + ½ SP/ Water + All SP
1 min	1min	Scraping	
2 min	30s	Mixing	
2 min 30 s	2min	Mixing	All SP/ ½ SP/ - +VMA
4 min 30s	30 s	Scraping	
5 min	1 min	Mixing	
6 min	-	Initial slump flow	

Mix Designs - Four different self-consolidating cement pastes; reference mix designs 1, 2, 3 and 4, were investigated in this research project. Mix designs 1 and 3 are based on the powder-type approach, while mixtures 2 and 4 are based on the VMA-type approach, even though mixture 4 does not contain any VMA. The mix proportions for 1.5 liter of mixtures 1 to 4 are listed in Table 5. For each cement paste, the amount of SP was adjusted to obtain a mini-slump flow value between 320 and 340 mm (330 ± 10 mm) at 20 °C and 7 min after mixing.

Mixtures 1 and 3 were repeated with cement from different manufacturers (A and B), resulting in a change in the amount of SP to obtain the targeted mini-slump flow. The results for mixtures 2 and 4 discussed in task 1 are made with the cement from manufacturer B. All other results in this report, including results on mixture 2 discussed in other tasks are made with cement from manufacturer A, unless otherwise stated.

Further variations in the mix design were performed by changing the type of SP, adding or removing VMA or replacing cement by SCM or limestone filler or vice-versa. All replacements are based on a volumetric basis to keep the amount of water constant for each mixture. The SP dosage for each of the other mixtures was adapted to achieve the targeted mini-slump flow. The amount of SP for each mix design is presented in Table 6.

Table 5. Reference Mix Designs 1 to 4 (for 1.5 liter).

	Cement type I/II (g)	Filler or SCM (g)	Water (g)	SP1 (g)	SP2 (g)	VMA (g)	W/C	W/P	C/P
Mix Design 1	1210.8	*LF: 1210.8*	665.9	5.2	-	-	0.55	0.275	0.5
Mix Design 2	1443.3	SF: 50.4 + FA:499.3	759.4	-	9.690	1.250	0.53	0.38	0.724
Mix Design 3	1308.6	*LF: 981.5*	719.7	3.8	-	-	0.55	0.31	0.57
Mix Design 4	1443.3	SF:50.4 + FA:499.3	801.4	-	11.720	0	0.55	0.40	0.724

Table 6: Amount of SP and resulting mini-slump flow for different mix designs (See section 6).

	SP1 (g)	SP2 (g)	Mini Slump flow (mm)
Mix Design 1 (ref)	5.200	-	330
Mix Design 1 with SP 2	-	7.250	330
Mix Design 2	-	9.690	335
Mix Design 2 (New Cement delivery)	-	10.018	340
Mix Design 2 –SF -FA	-	12.080	345
Mix Design 2 -VMA	-	12.698	345
Mix Design 2 with SP1 -VMA	6.113	-	330
Mix Design 2 +LS –FA –SF -VMA	-	7.090	335

4.4.2 Concrete Mixtures

Reference Mixing Procedure – All concrete mixtures were produced in an intensive concrete mixer (Eirich), at a fixed concrete volume of 85 liter. The following mixing procedure is considered as the reference mixing procedure with a total mixing time of 3.5 minutes, starting after first contact between cement and water.

1) Mixing of the aggregates for 1 minute
2) Addition of cement and filler
3) Mixing for 1 minute
4) Addition of the water while mixing for 1 minute
5) Addition of the superplasticizer while mixing for 2.5 minutes.

Reference Mix Design – The reference mix design for the investigation on concrete is a powder-type mix design with a target slump flow of 700 mm and is displayed in Table 7.

Table 7. Mix design of reference concrete (units in kg/m^3).

Sand	1064
Pea Gravel	510
Type I/II Cement	300
Limestone Filler	300
Water	165
SP 2	4.15

The variations in mix design and mixing procedures are described in Task 3: Robustness of Mixing Procedure and Addition Sequence of Materials on Concrete.

5 Task 1: Robustness of Cement Pastes due to Changes in Mixing Procedure

As the first task in this research project, the responses of the rheological properties (yield stress and plastic viscosity) of cement pastes with SCC consistency are measured, revealing the effect of changes in the amount of water and SP, variations in the addition time of SP and mixing energy and different cement deliveries. The robustness of the four reference mixtures, presented in the section above, is evaluated. However, the first task was to identify the repeatability of producing the cement pastes and the reproduction of the rheological properties.

5.1 Repeatability of Rheological Measurements on Cement Pastes

Before starting the variations in constituent elements and measuring the responses of the rheological properties, confidence intervals were established based on the repetitive production of the reference cement pastes and the measurement of the rheological properties. For each mix design, a 90% confidence interval was established for yield stress and plastic viscosity, based on 4 or 5 repetitions. Examples for the 90% confidence intervals can be seen in Figures 18 to 21 for mixtures 3 and 4, respectively. The shown confidence intervals in these figures include the variation of rheological properties with time. The results do not only show the repeatability of the initial measurements (at 15 min), but also show that the evolution of the rheological properties with time is quite repeatable. A summary of the confidence interval for all mixtures, measured at 15 min, is shown in table 8.

Table 8. Average values of yield stress and plastic viscosity for reference mix designs 1 to 4 and corresponding 90% confidence interval based on 4 or 5 measurements.

	Mix Design 1	Mix Design 2	Mix Design 3	Mix Design 4
Average Plastic viscosity (Pa s)	0.435	0.317	0.328	0.353
+90% confidence limit (Pa s)	0.443	0.332	0.332	0.374
-90% confidence limit (Pa s)	0.426	0.301	0.323	0.332
Average Yield stress (Pa)	3.63	1.600	2.64	2.23
+90% confidence limit (Pa)	4.10	2.367	2.80	2.82
-90% confidence limit (Pa)	3.15	0.991	2.48	1.64

Repeatability can give a first indication on the robustness of the mixtures. The variations in constituent elements, mix design and mixing procedure are very small, but the larger the confidence interval, the less robust the mixture. As can be deducted from Table 2, mix design 3 appears to be the most robust mixture to errors in material properties, mixing procedure and measurements, as the differences between the average value and the confidence interval boundaries are 5 mPa s for the viscosity and 0.16 Pa for the yield stress. Mixture 1 is slightly less robust, mixtures 2 and 4 have larger confidence intervals. It should however be noted that mixtures 1 and 3 were produced with cement from manufacturer A, and mixtures 2 and 4 with cement from manufacturer B. A more detailed discussion on the influence of the cement deliveries is provided in section 5.3.

The defined 90% confidence intervals include the errors due to small variations in constituent material properties, mixing procedure, sampling as well as the rheological measurements. If due to an induced variation in mix design or mixing procedure the rheological properties are outside the confidence interval, the probability is large that the variation is significant.

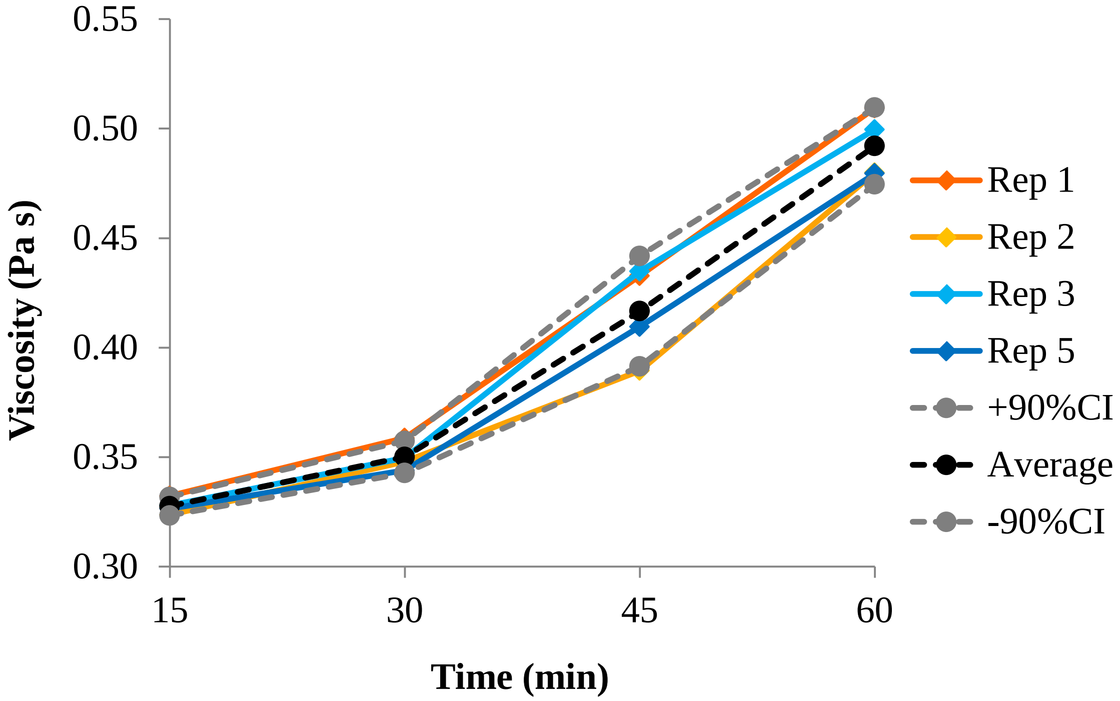

Figure 18. Viscosity of mix design 3 at different times with confidence intervals.

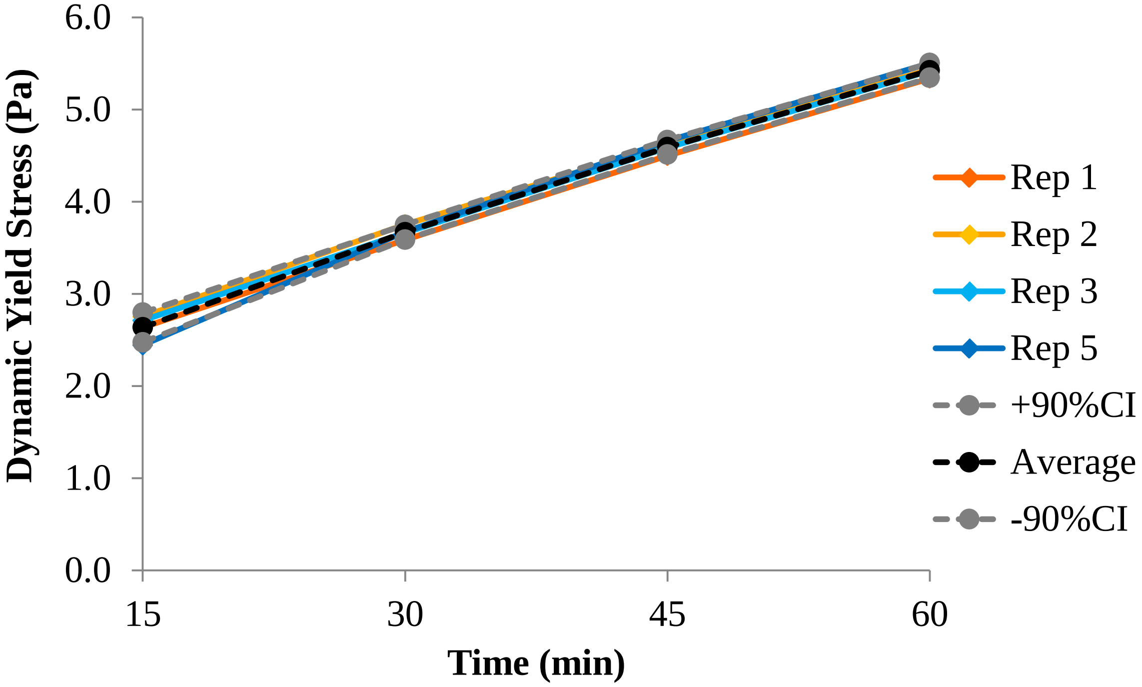

Figure 19. Yield stress of mix design 3 at different times with confidence intervals.

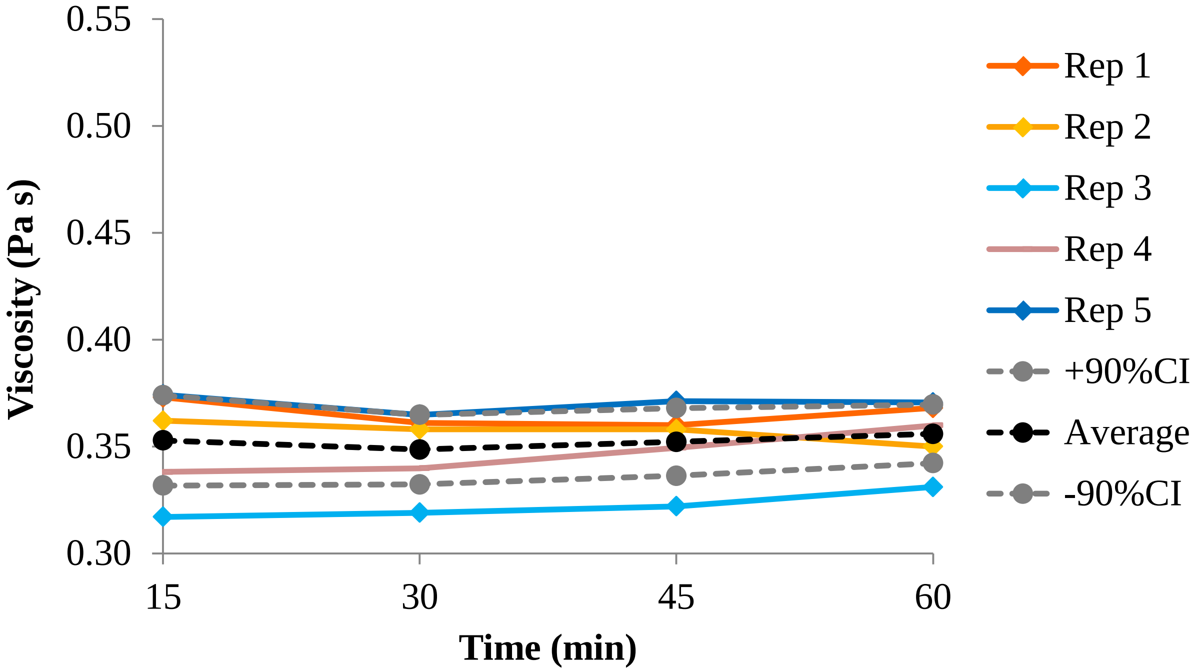

Figure 20. Viscosity of mix design 4 at different times with confidence intervals.

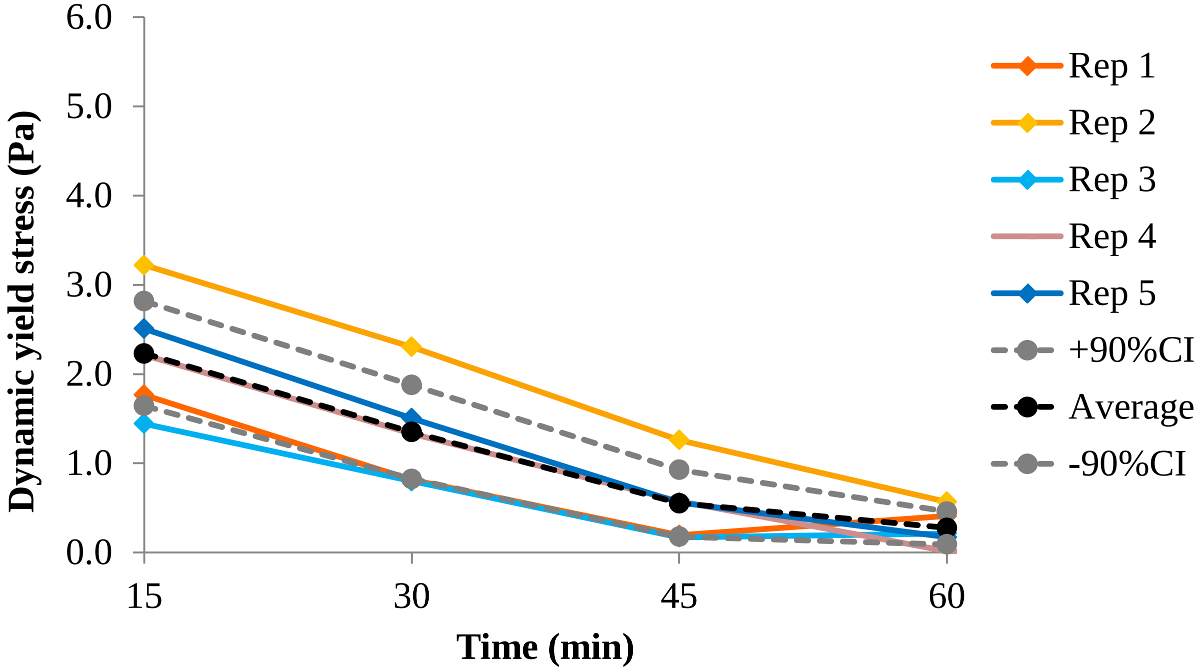

Figure 21. Yield stress of mix design 4 at different times with confidence intervals.

5.2 Robustness to Variations in Water Content

The variations in plastic viscosity and (dynamic) yield stress due to a change in the quantity of water are illustrated in Figs. 22 and 23 for mix designs 1 and 3. The induced variations of water in the cement pastes correspond to changes of ± 5 l and ± 10 l per cubic meter of concrete. As can be seen in Figs. 22 and 23, the changes in water content have a significant influence on the rheological properties of the mixtures. Defining robustness as the inclination of the response to a change in mix design, as shown in Figure 2, then mix design 1 is clearly less robust than mix design 3 (with cement from manufacturer A), especially for the plastic viscosity measurements. This behavior is in accordance with the literature review, as mixtures with lower w/p are more sensitive to a change in water content than mixtures with a higher w/p.

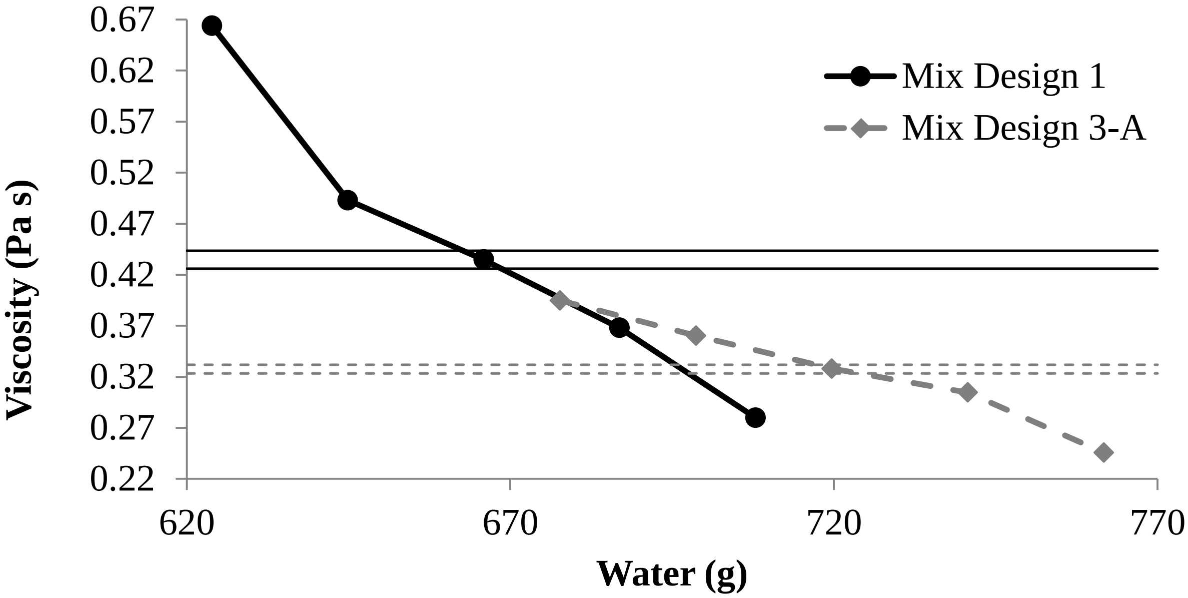

Figure 22. Influence of the amount of water added to the mixture on the viscosity. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

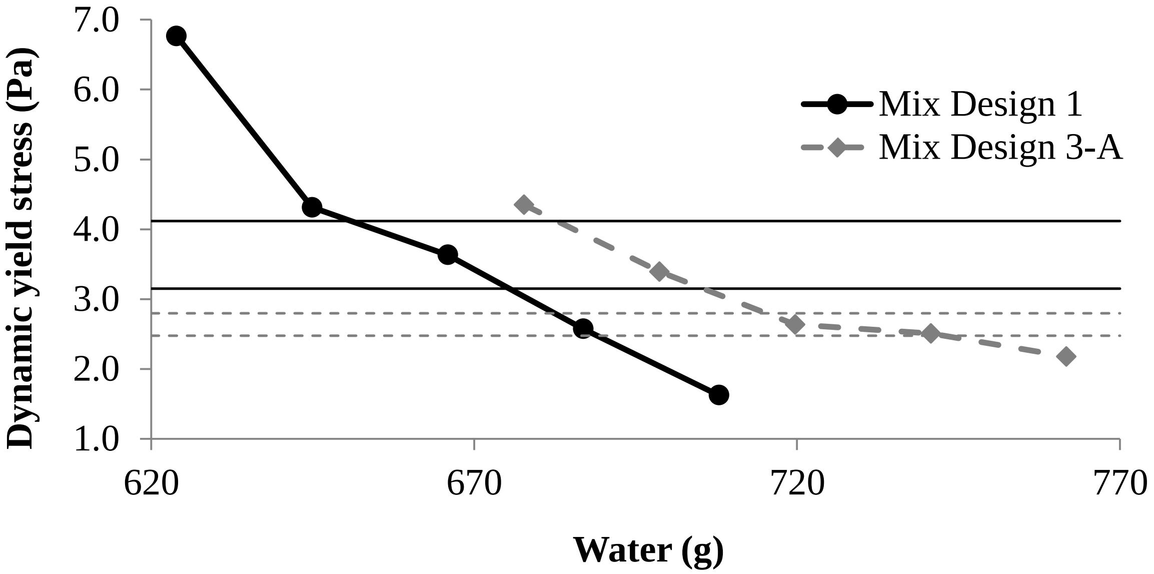

Figure 23. Influence of the amount of water added to the mixture on the yield stress. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

5.3 Robustness to Variations in Cement Deliveries

As mentioned in section 5.1, a significant difference was found between the confidence intervals of mixtures 1 and 3 on the one hand, and mixtures 2 and 4 on the other hand. There are two main differences between these two groups of mixtures: the first group are powder-type SCC cement pastes, while the second group are VMA-type SCC cement pastes. The second difference is the cement manufacturer. The powder-type pastes were made with cement from manufacturer A, while the VMA-type pastes were made with cement from manufacturer B. To investigate the influence of the cement delivery, the confidence intervals for plastic viscosity and yield stress of mixtures 1 and 3 were repeated with cement from manufacturer B. The results for mixture 3 can be found in Figs. 24 and 25. While for mixture 3, the confidence interval for the plastic viscosity was only slightly affected, the repeatability of the yield stress measurements was far more precise for the pastes with cement from manufacturer A, compared to that from manufacturer B. In a similar fashion (not shown), mixture 1 showed a significantly larger confidence interval for the plastic viscosity: the difference between the average and the 90% limit is 9 mPa s for mixture 1-A, while it is 33 mPa s for mixture 1-B. However, the yield stress repeatability was similar for both mixtures, most probably due to the relatively large confidence interval of mixture 1-A.

It can thus be concluded that the cement from manufacturer A allows for more robustness than cement from manufacturer B. The causes for this behavior were not investigated, but most probably, the variation in physical and/or chemical properties of cement B was larger than for cement A. In section 6.2.1, it is shown that a change in delivery of cement (from manufacturer A) entrains some differences, necessitating the repetition of the reference mixtures. For a change in cement delivery from producer B, repeating reference mixture 2 resulted in a mini-slump flow larger than 400 mm, which is significantly larger than the target 330 mm and compromising the stability of the cement paste. As a consequence, the cement from manufacturer B was no longer used for the further investigations in this research project.

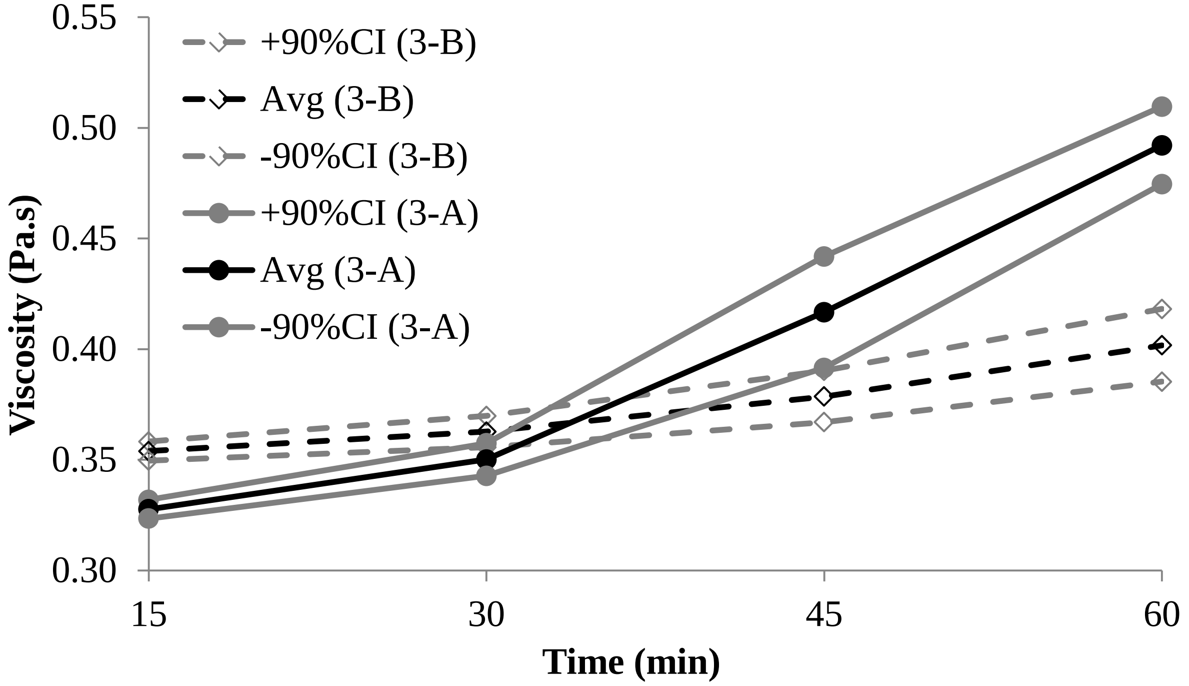

Figure 24. Confidence intervals for the viscosity of reference mixture 3, produced with cement from manufacturers A and B.

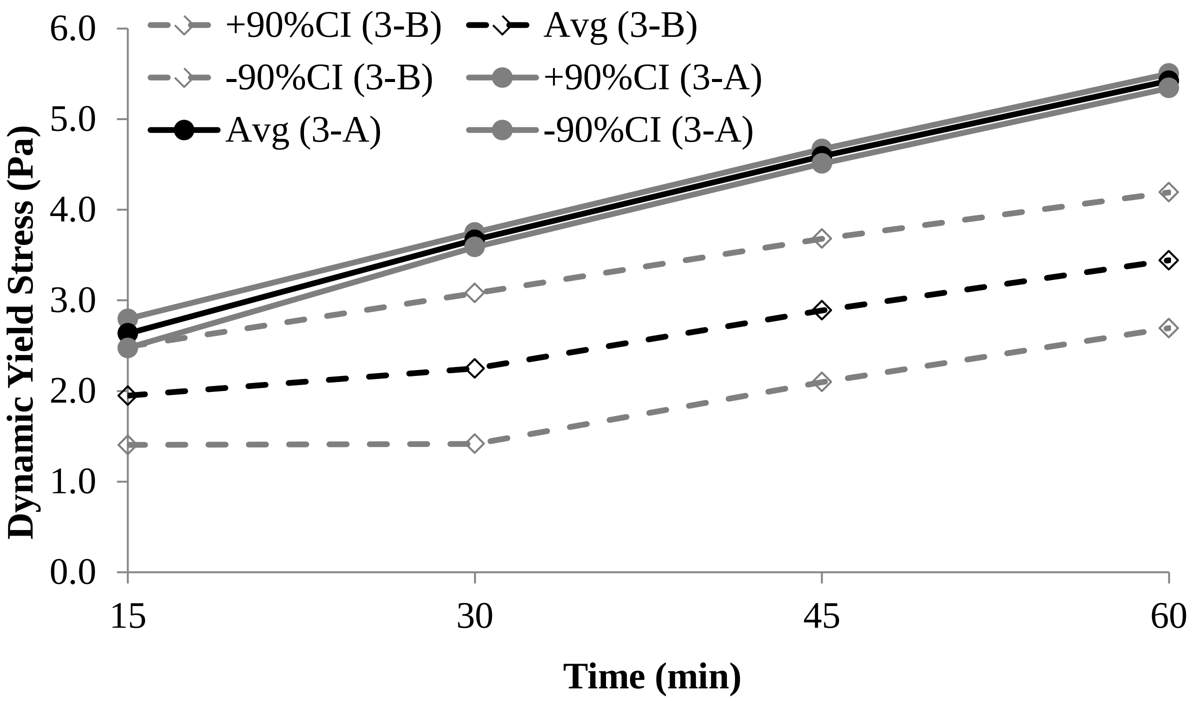

Fig. 25. Confidence intervals for the yield stress of reference mixture 3, produced with cement from manufacturers A and B.

5.4 Robustness to variations in amount of SP

Figures 26 and 27 show the rheological behavior as a consequence of variations in the amount of superplasticizer added. The variation in amount of SP is defined as ± 5% and ± 10% compared to the reference value. As can be seen in Figures 26 and 27, both the yield stress and plastic viscosity show a significant decrease with increasing SP dosage. The decrease in yield stress is expected, but the decrease in plastic viscosity is larger than what is anticipated. Based on the yield stress results, it can be concluded that mixture 3 is more robust than mixture 1, due to the higher w/p.

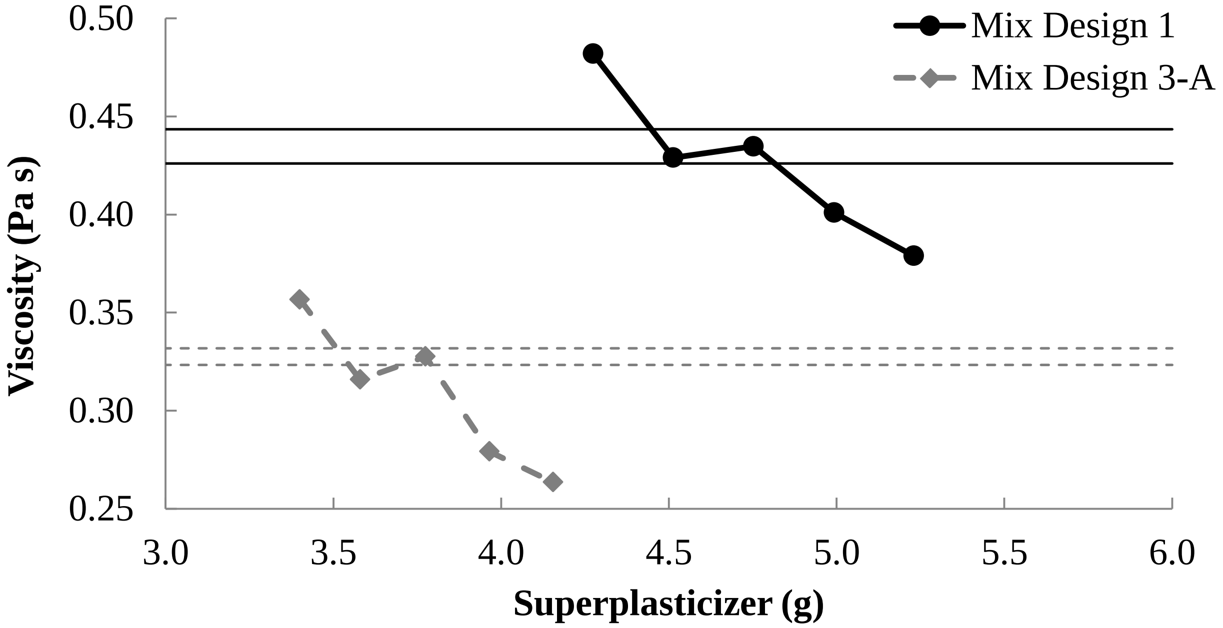

Figure 26. Influence of the amount of SP added to the mixture on the viscosity. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

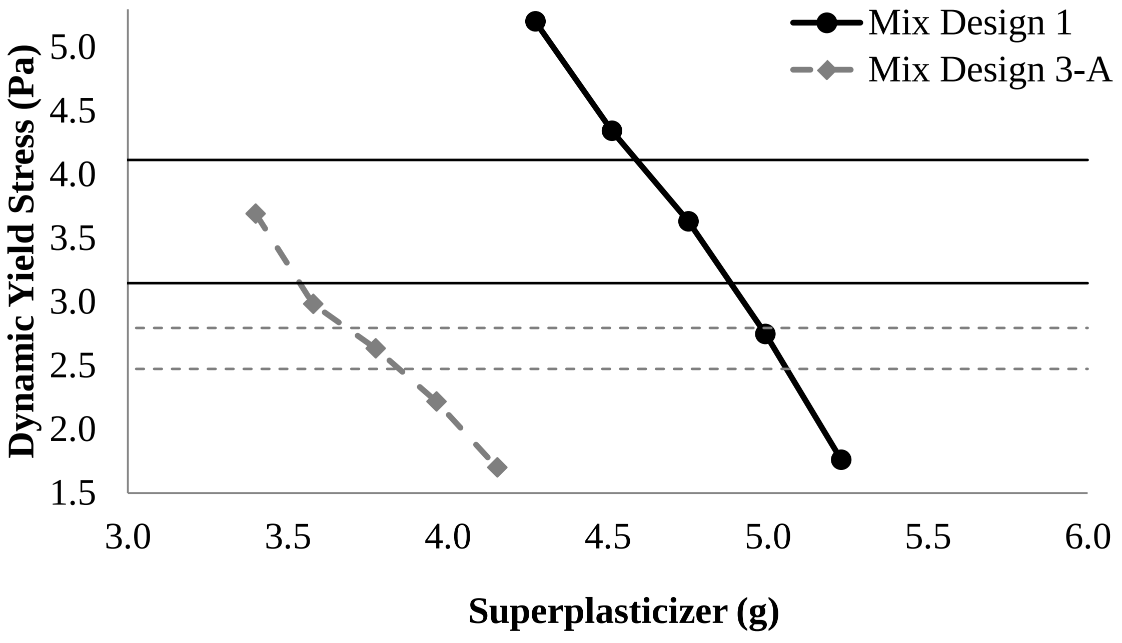

Figure 27. Influence of the amount of SP added to the mixture on the yield stress. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

5.5 Robustness to Variations in the Addition Time of the SP

The adding time of the superplasticizer was investigated by adding the entire quantity of SP to the mixture 2 minutes after cement-water contact (100% delayed), or adding the SP in the mixing water before incorporating it in the cement paste (100% with water). An intermediate scenario (50/50) is also investigated. Changing the addition time of the SP from 100% delayed (reference) to 100% with water mainly increases the plastic viscosity of the mixtures (Fig. 28), somewhat surprisingly. The yield stress, which is the parameter expected to vary more significantly, is not so much affected for mixture 1, and even remains in the confidence interval for mixture 3 (Fig. 29). The 50/50 case was anticipated to deliver results between the 100% with water and the 100% delayed, but over the course of the project, it has been observed that the 50/50 case is a lot more complex and does not easily follow all trends. An example can be seen in Fig. 29, showing different trends for mixtures 1 and 3. Consequently, the 50/50 case will not be extensively discussed, but it can be argued that it is not the most robust solution.

The above results are obtained on powder-type SCC cement pastes, and it will be shown in section 6.1 that for the VMA-type SCC cement pastes, the yield stress is significantly affected. A detailed discussion on the addition time of SP and the influence of different constituent elements can be found in section 6.2.

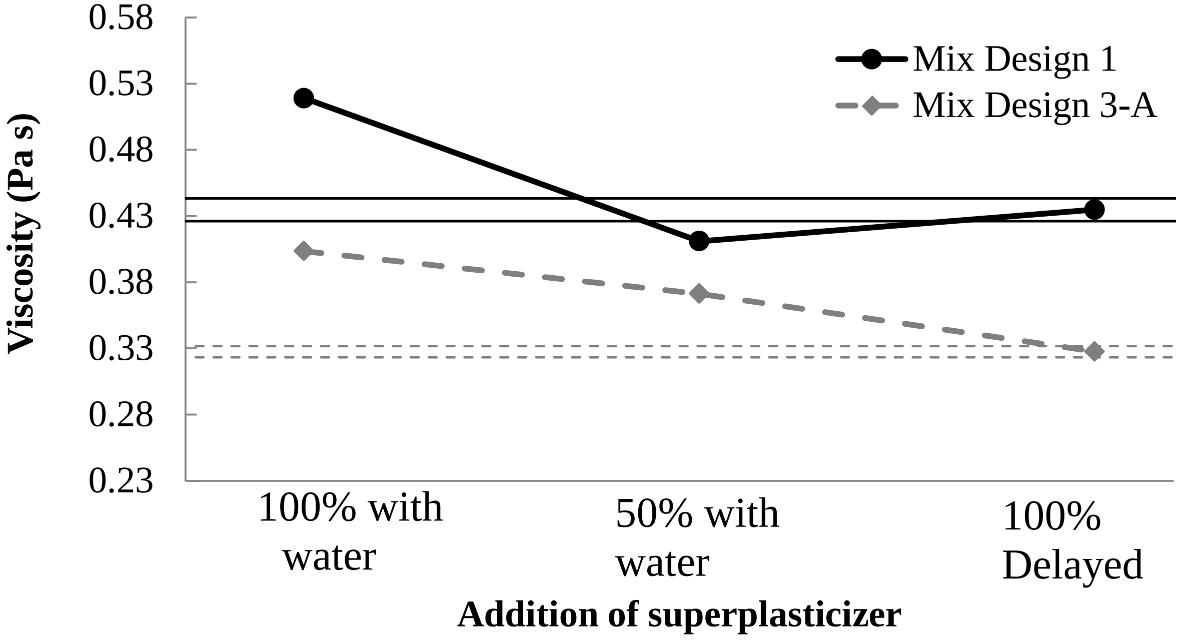

Figure 28. Influence of the addition time of SP added to the mixture on the viscosity. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

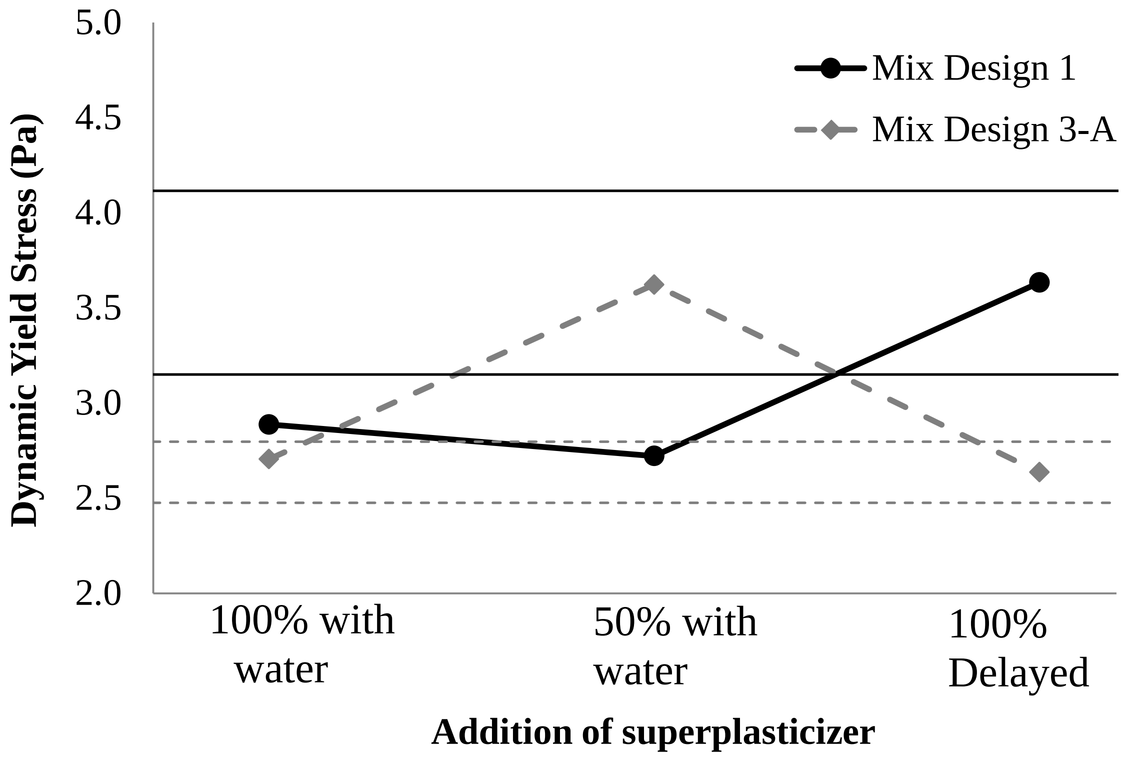

Figure 29. Influence of the addition time of SP added to the mixture on the yield stress. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

5.6 Robustness to Variations in Mixing Energy: Mixing Time and Mixing Speed

The rheological properties were evaluated to determine the influence of mixing time and speed. Mix designs 1 and 3 were prepared at two different mixing speeds (1 (=ref) and 2) and the time of mixing was short, medium or long, as described in Table 4. The influence of mixing time is shown in Figs. 30 and 31, showing that an increase in mixing time makes the cement pastes more fluid (decrease in yield stress and plastic viscosity with increasing mixing time). Referring to the literature described in the first section, the mixtures are not overmixed and the stabilization time t_s (eq. 4) is most likely not reached in the reference mixing procedure.

Figures 32 and 33 confirm the above observation by increasing mixing speed instead of mixing time. Apart from the apparent independence of the yield stress of mixture 1 to a change in mixing speed, the same conclusion stands. More interesting is the larger sensitivity of mixture 3 to a change in mixing energy, compared to mixture 1. For the above described properties, mixture 1 was the least robust. A possible argument for this observation could be that due to the lower water content in mixture 1, the shear rate in the water between the cement and limestone filler particles is larger, enhancing the dispersion action of the SP and increasing the mixing efficiency. Furthermore, significantly larger stabilization times are expected when producing cement pastes, compared to mortars and concretes, due to the absence of sand and coarse aggregates to “grind” the agglomerated fine particles.

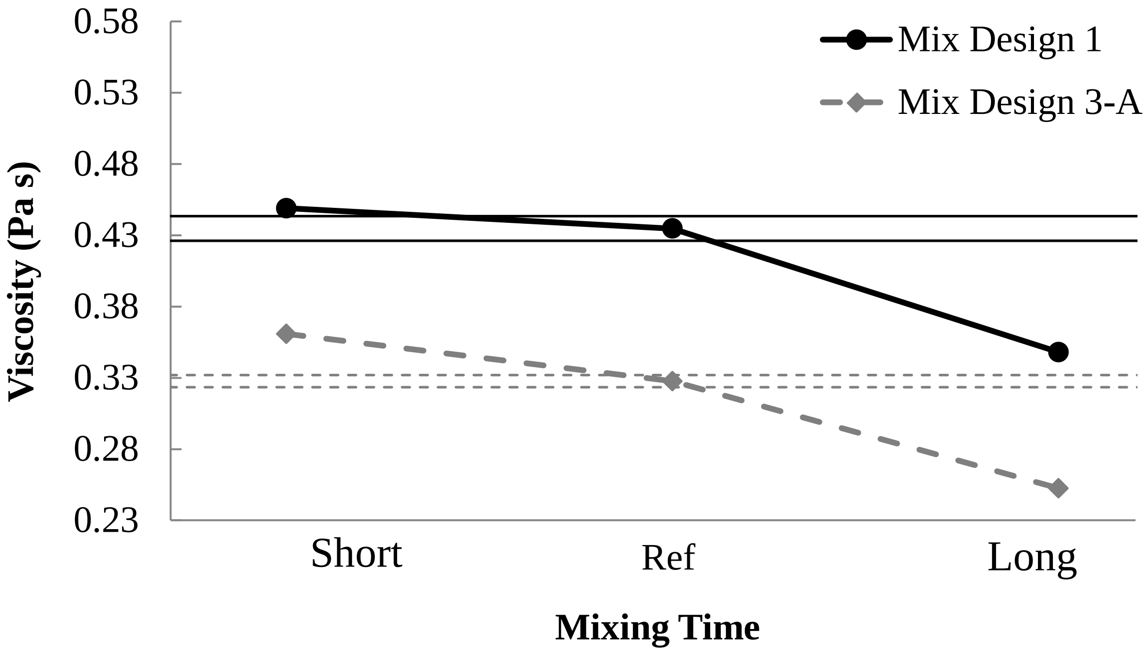

Figure 30. Influence of the mixing time on the viscosity. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

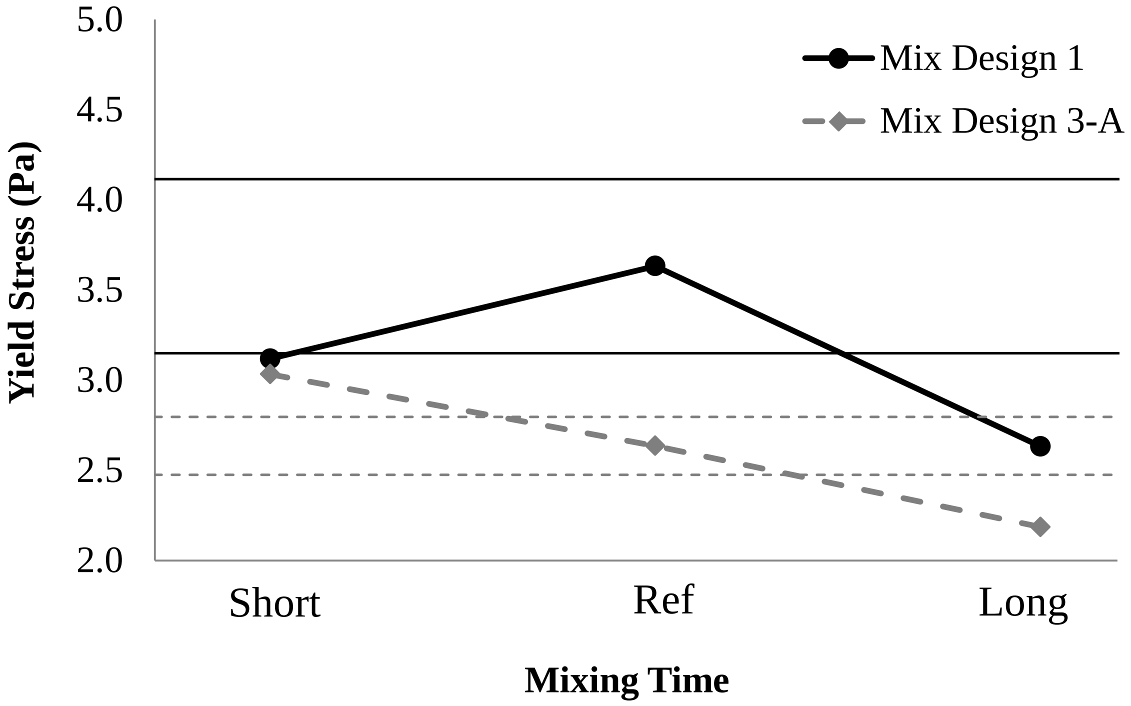

Figure 31. Influence of the mixing time on the yield stress. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

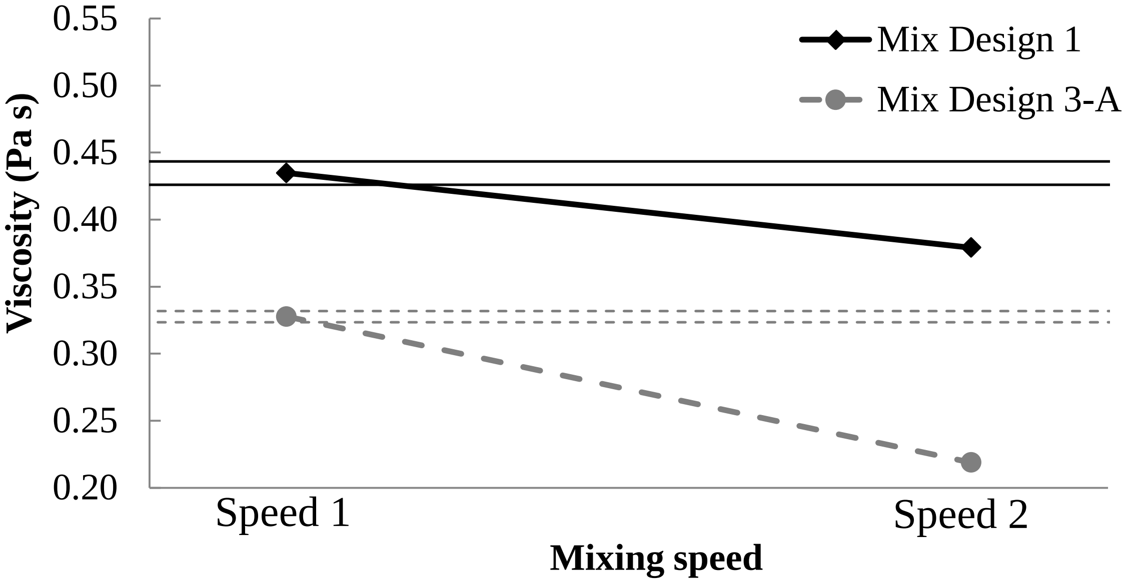

Figure 32. Influence of the mixing speed on the viscosity. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

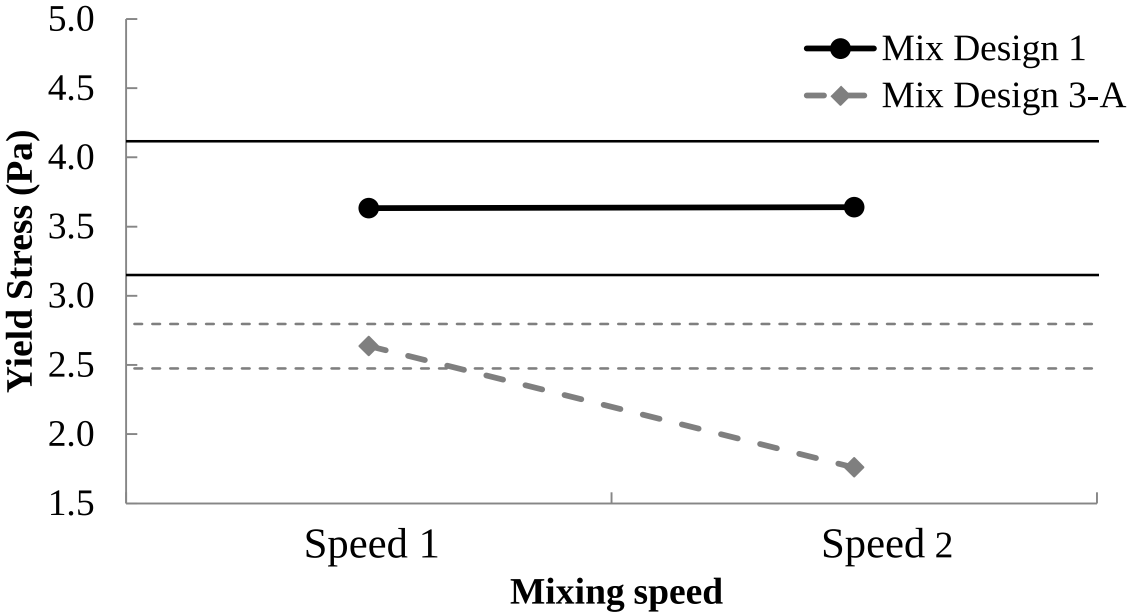

Figure 33. Influence of the mixing speed on the viscosity. Black/solid = mixture 1, grey/dashed = mixture 3. The horizontal lines represent the confidence interval for the respective reference mixtures.

5.7 Summary

Comparing mixtures 1 and 3, mixture 3 appears to be the most robust mixture of the two, due to its smaller 90% confidence intervals and its lower sensitivity to variations in water content and SP content. However, the differences between both mixtures in robustness to a change in adding time of the SP are not significant and mixture 1 appears more robust to changes in mixing speed and mixing time. Furthermore, the robustness of the mixtures, in terms of repeatability, depends significantly on the cement deliveries.

Figures 34 and 35 show, for mixture 3, the evolution of the plastic viscosity and yield stress with the induced changes, where the central point indicates the reference mixture. It can be seen the amount of water, addition time of the SP (especially for viscosity) and the mixing speed have major influence on the variations in the mixture. The amount of SP added and the mixing time have minor influence. Unsuccessful attempts have been undertaken to induce a 3rd mixing speed and as a consequence, the mixing time combined with the addition time of the SP were withheld as parameters for the next task, in combination with variations in the water content.

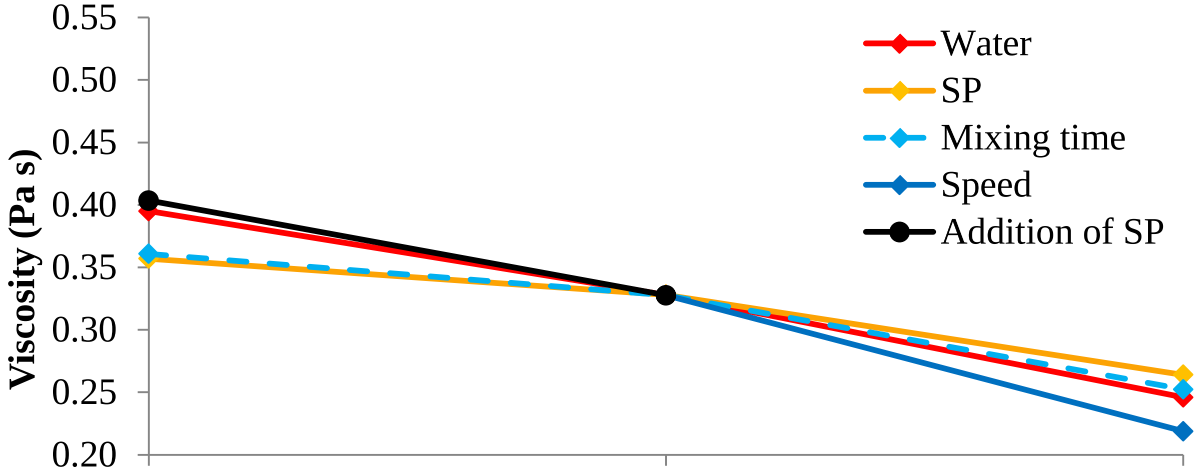

Figure 34. Summary of variations in viscosity due to induced changes. The center point represents the reference mixture (ref. water, ref. amount of SP, delayed addition, slow mixing speed and medium mixing time). The change in water corresponds to -10 and +10 l/m^3 (in the corresponding concrete), the change in SP is ± 10%.

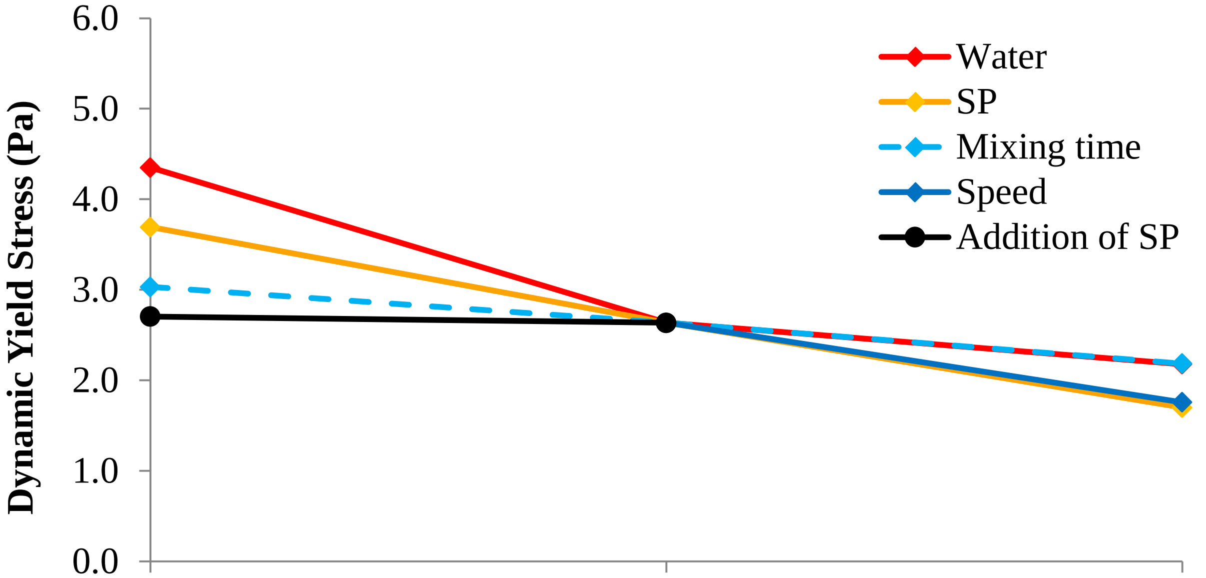

Figure 35. Summary of variations in yield stress due to induced changes. The center point represents the reference mixture (ref. water, ref. amount of SP, delayed addition, slow mixing speed and medium mixing time). The change in water corresponds to -10 and +10 l/m^3 (in the corresponding concrete), the change in SP is ± 10%.

6 Task 2: Combining Variations in Constituent Elements and Addition Time of SP

6.1 Combination of Variation in Water with Addition Time of SP and Mixing Time

In the first part of task 2, the combined influence of the previously selected factors influencing robustness is investigated. These parameters are: the amount of water, addition time of SP and mixing time. In this part, the addition time of the SP and the mixing time are combined with the variations in water content. The addition time of SP is 100% with water, 100% delayed and the 50/50 case (Table 4), while the mixing times are short, medium and long, according to Table 3. The variations in water amount in the cement pastes correspond to the variations of ± 10 l/m^3 in concrete. Although the main parts of the results in task 1 were obtained on powder-type SCC cement pastes, the VMA-type SCC cement pastes are included in task 2. The reference mixtures are mixture 1 (least robust powder-type mixture) and mixture 2 (VMA-type mixture containing VMA).

The plastic viscosity and dynamic yield stress values of both mixtures subjected to variations in the addition time of SP and in the amount of water are illustrated at Figs. 36 and 37, respectively. For the powder-type SCC cement paste (Mix design 1 displayed on the left side of both figures), the sensitivity to a change in water content is significantly larger than the sensitivity to a change in addition time of the SP. It also appears that changing the addition time of SP does not largely affect the robustness of the investigated powder-type cement paste to variations in water content. For the VMA-type SCC cement paste (displayed on the right), the conclusions are opposite. It appears that plastic viscosity and dynamic yield stress undergo more significant variations due to a change in addition time of SP than due to a variation in water content corresponding to 10 l/m^3 in concrete. With increasing water content, the robustness to the addition time of SP appears to increase, but still remains significant. By adding the SP delayed relative to the water addition, the robustness of the mixtures to a variation in water content appears to increase. Comparing both mixtures, it can be concluded that mixture 1 is more sensitive to a change in water content compared to mixture 2, but mixture 2 is significantly more sensitive to a change in addition time of the SP, compared to mixture 1. A more detailed investigation on the influence of the constituent materials on this behavior is described in section 6.2.

Figures 38 and 39 depict, respectively, the plastic viscosity and the yield stress for mixtures 1 and 2 subjected to a variation in water content and a change in mixing time. Similarly to what is observed in Figs. 36 and 37, mixture 1 is more sensitive to a change in water content than mixture 2 and the variation in mixing duration does not significantly influence the robustness. It can also be observed that the change in water content is more significant than the change in mixing time for both mixtures, although the yield stress data show some strange results for mixture 2 with increased water content. The results are also in agreement with section 5.6, showing that for the reference (medium) mixing duration, the stabilization time is not yet reached.

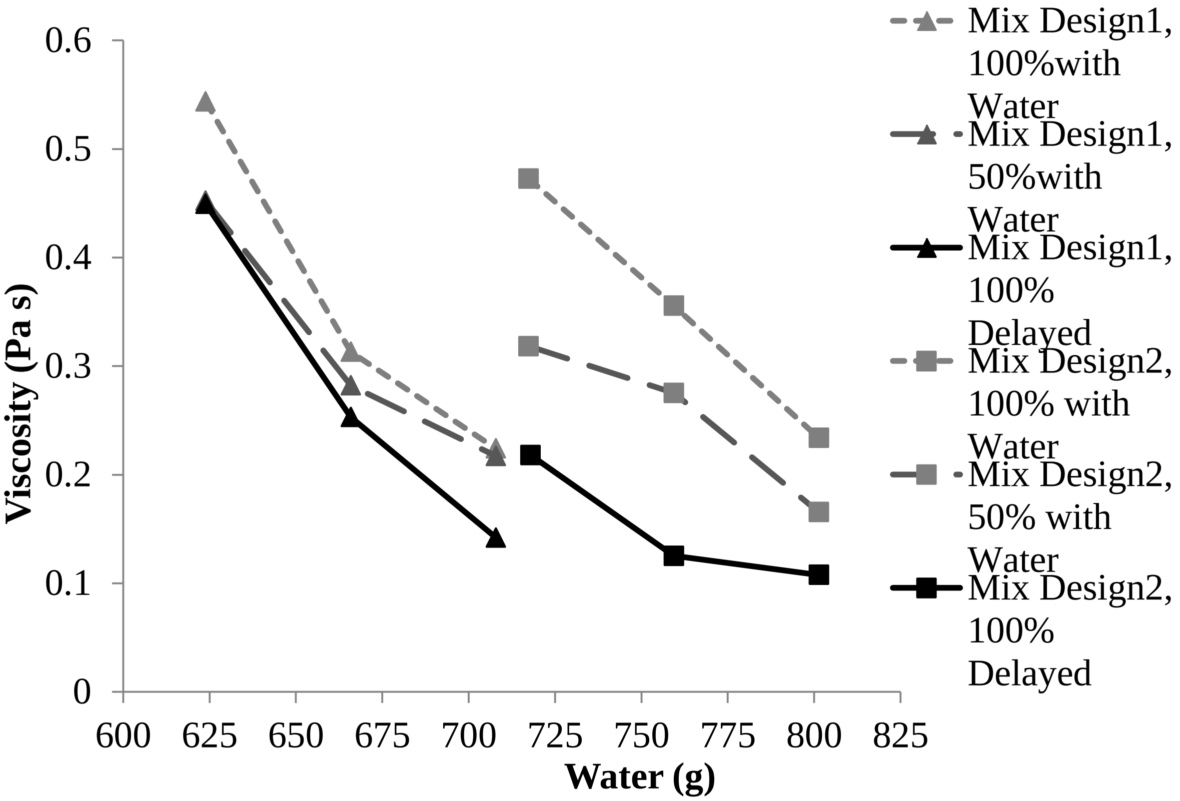

Figure 36. Viscosity of mix designs 1 and 2 at different addition time of SP-water.

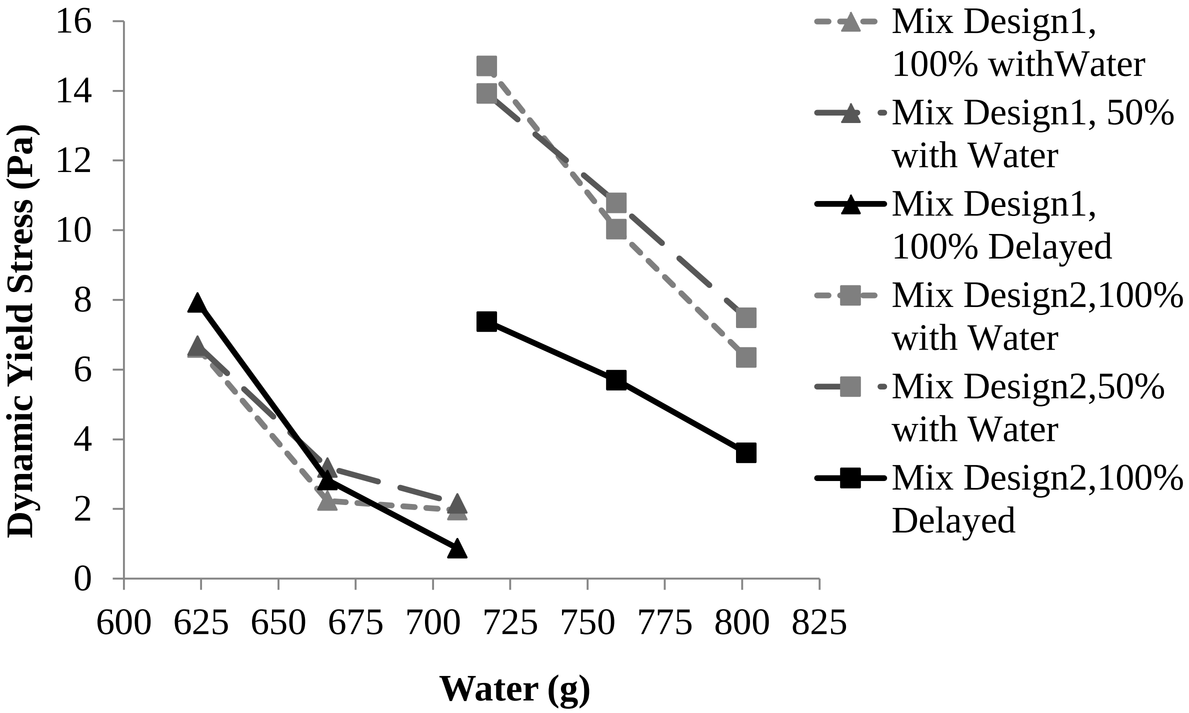

Figure 37. Yield stress of mix designs 1 and 2 at different addition time of SP-water.

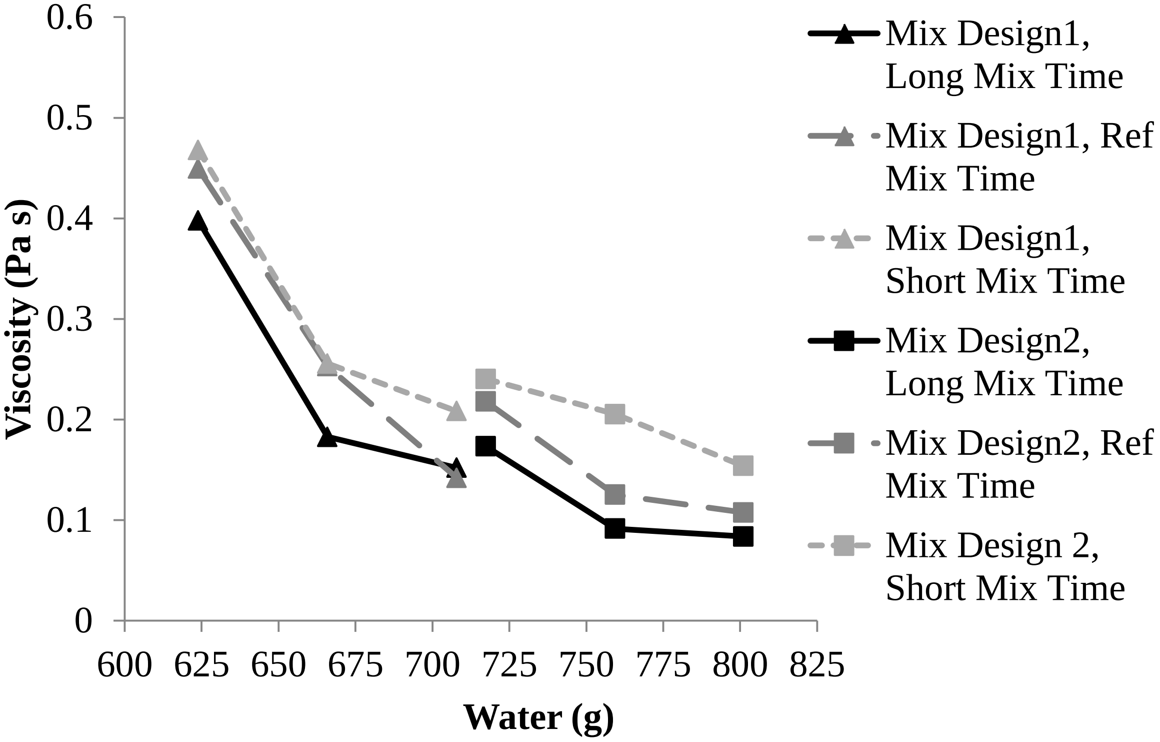

Figure 38. Viscosity of mix design 1 and 2 at different mixing time-water.

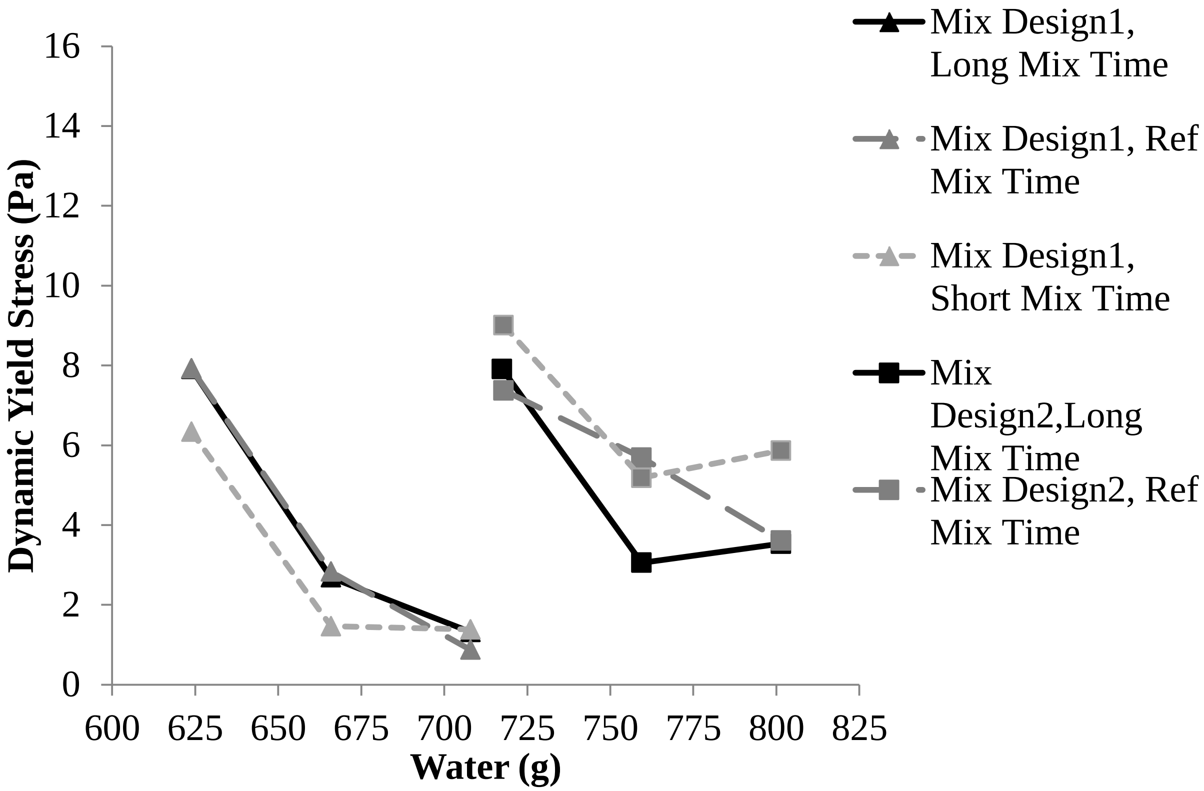

Figure 39. Yield stress of mix design 1 and 2 at different mixing time-water.

6.2 Investigation of Critical Constituent Elements Increasing Robustness of Cement Pastes to a Variation in Addition Time of SP

Based on the results in Figs. 36-39, it can be concluded that the two most significant factors affecting robustness are the amount of water and the addition time of the SP. Especially for the VMA-type SCC cement pastes, the addition time of the SP has a significant influence on yield stress and plastic viscosity and can even alter the robustness of the mixture to a variation in water. In the next section, it is investigated which mix design parameter causes the largest difference in behavior between mixtures 1 and 2. The parameters investigated are the presence of VMA, the different SP types used, the presence of silica fume and fly ash and the influence of the limestone filler. For all mixtures, the dosage of the SP is adopted to reach the target mini-slump flow. The SP dosages and mini-slump flows can be found in Table 6. The research is especially focused on the plastic viscosity of the mixtures, as it shows a significantly larger sensitivity to the adding time of the SP than expected, and the 90% confidence intervals for the viscosity are smaller than those for the yield stress.

6.2.1 Change in cement delivery

Figures 40 and 41 show the response of plastic viscosity and yield stress for reference mixture 2. The difference between the reported results is a different delivery in cement, produced by the same manufacturer. As can be seen, the robustness of the cement paste to a variation in addition time of SP has significantly improved. It can thus be concluded that one of the critical mix design parameters for robustness is the interaction between cement and the chemical admixtures. The study on the other mix design parameters is conducted with the second delivery of the Type I/II cement, displayed on the right of Figures 40 and 41.

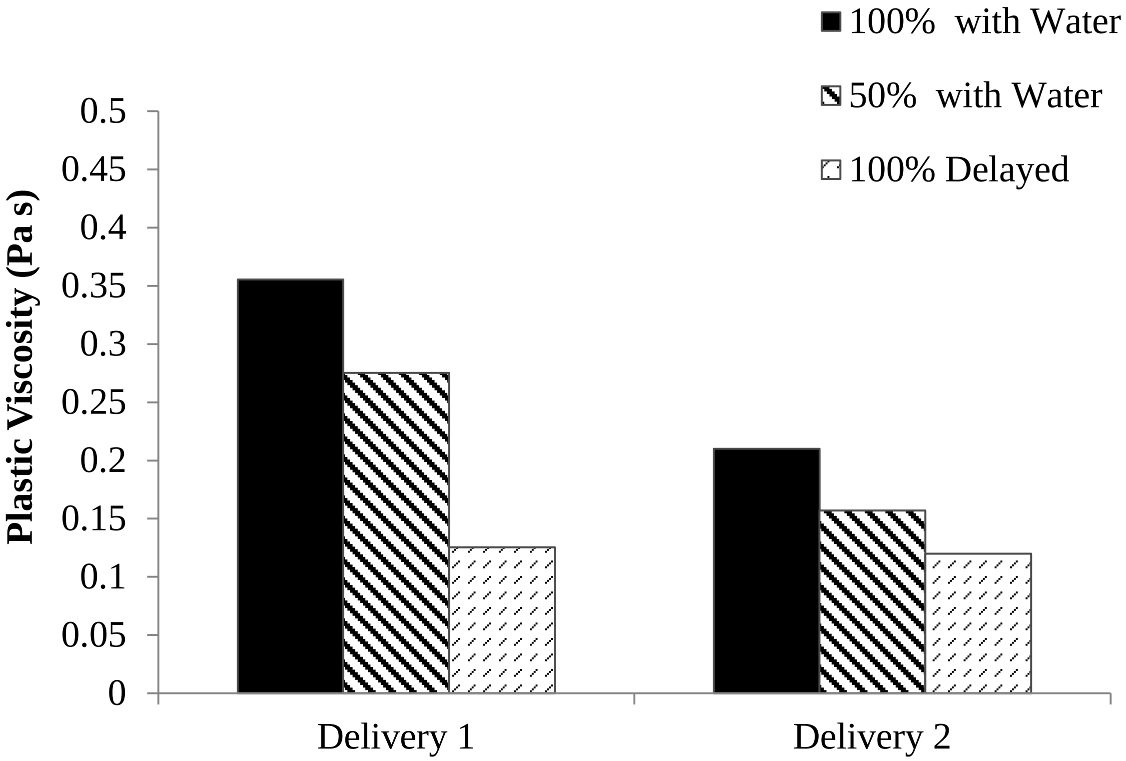

Figure 40. Influence of a change in delivery of cement from the same manufacturer on the response of plastic viscosity to a variation in adding time of the SP.

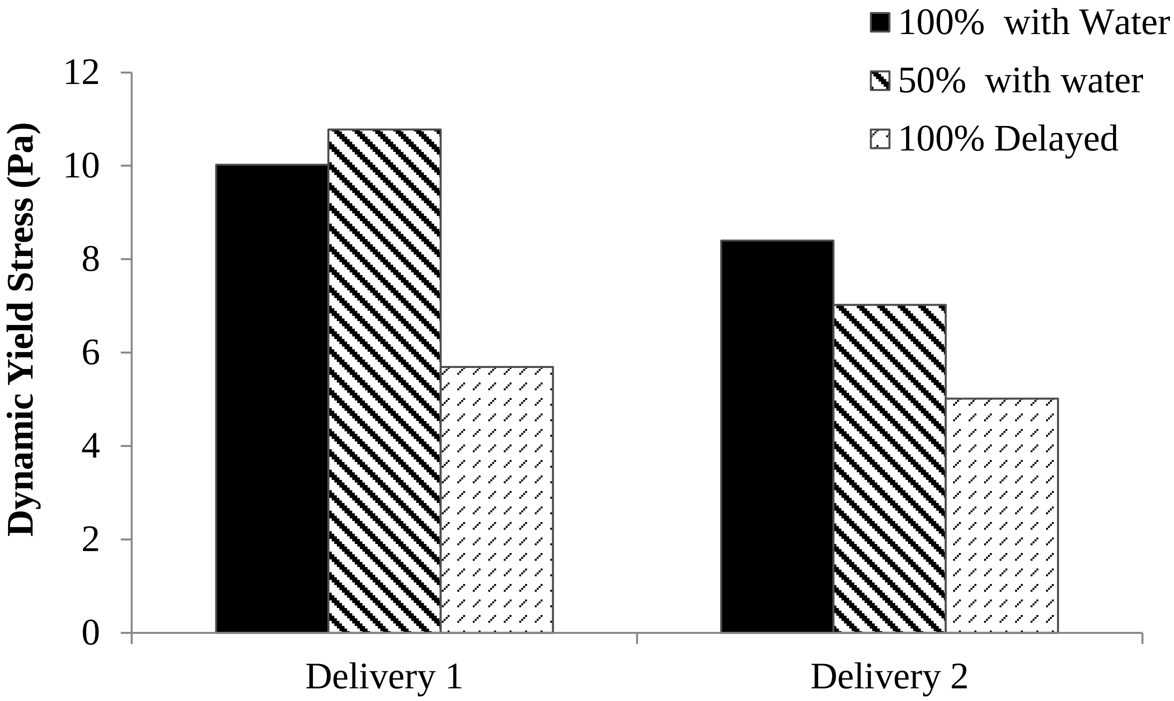

Figure 41. Influence of a change in delivery of cement from the same manufacturer on the response of yield stress to a variation in adding time of the SP.

6.2.2 Presence of VMA

Starting for mix design 2, the influence of different constituent elements is analyzed step by step to discover the most critical parameter influencing the response due to the delayed addition. The first parameter investigated is the presence of the viscosity modifying agent (VMA). The results for plastic viscosity and yield stress are respectively shown in Figs. 42 and 43. The left part of the figures show the response to a change in adding time of the SP of the mixtures with the VMA, while the right side is exactly the same mix design, but without VMA and a modified dosage of SP to obtain the same mini-slump flow for the mixtures with the 100% delayed addition. It should also be noted that the VMA was added 2.5 min after mixing, after the SP in all cases, regardless of the adding time of the SP. When focusing in Fig. 42 on the full black bar (100% with water) and the dark grey bar (100% delayed), it can be observed that the presence of VMA does not significantly affect viscosity, but more importantly, neither the change of viscosity with the change in adding time of SP. However, the presence of VMA does significantly alter the yield stress behavior when the cement paste is subjected to a variation in adding time of SP (Fig. 43). Focusing on the plastic viscosity, it can thus be concluded that the VMA does not influence the robustness of the mixture subjected to a change in adding time of the SP.

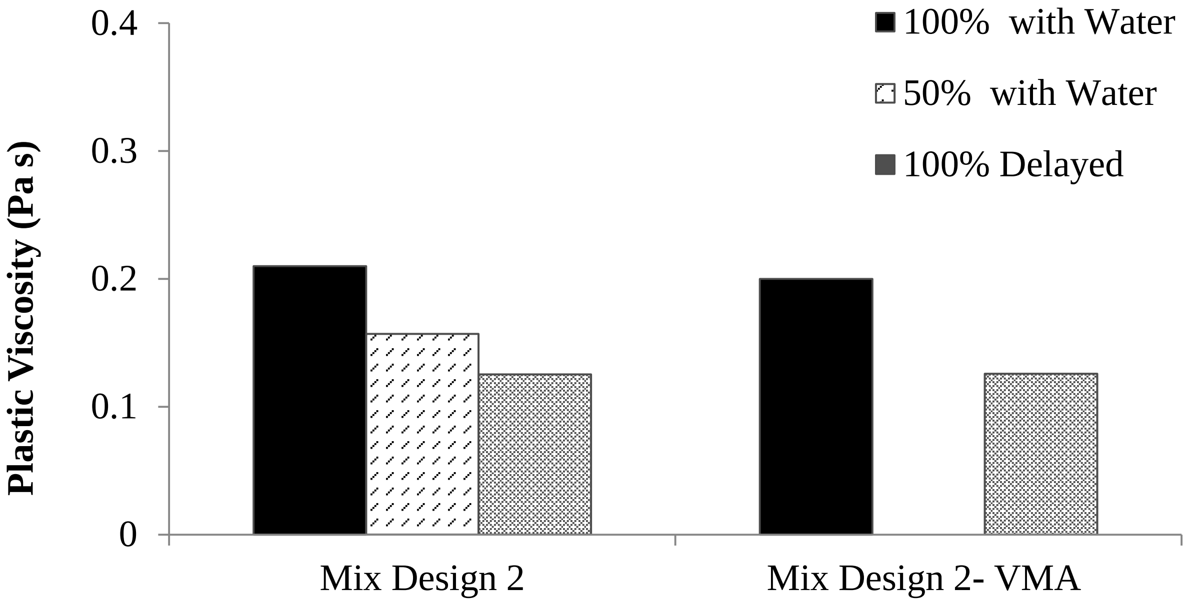

Figure 42. Influence of the presence of VMA on Plastic Viscosity: Left: Ref mix design 2 with VMA, Right: Ref mix design 2 without VMA.

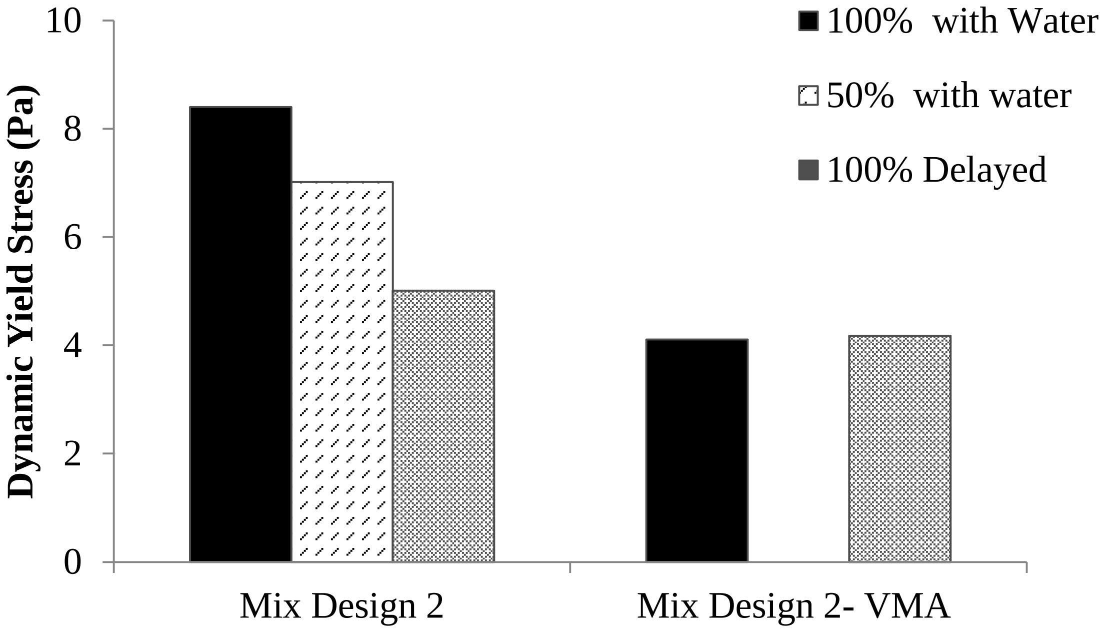

Figure 43. Influence of the presence of VMA on Dynamic Yield Stress. Left: Ref mix design 2 with VMA, Right: Ref mix design 2 without VMA.

6.2.3 Influence SP Type

Reference mixtures 1 (powder-type) and 2 (VMA-type) were produced with SP from different manufacturers. SP 1, used for mixture 1 is a PCE with relatively short workability retention, while SP 2 used in mixture 2 is a PCE with long workability retention. Although no details on the molecular structures are known, it is most probable that the chemical molecules are different [68]. To investigate the influence of the SP type, mixture 2 was reproduced with SP 1, adjusting the dosage to obtain the target mini-slump flow after mixing for the mixtures with the delayed addition of SP. The VMA was omitted to avoid any potential compatibility problems between SP 1 and the VMA (which is from the same manufacturer of SP 2). Figure 44 shows the results for the plastic viscosity, in which it can be seen that both mixtures have approximately equal robustness to a variation in addition time of the SP. As a result, the differences observed in Fig. 36 are probably not the consequence of the type of SP. Concerning the yield stress (Fig. 45), the mixture with SP 2 seems even more robust than the mixture with SP 1, supporting the above statement.

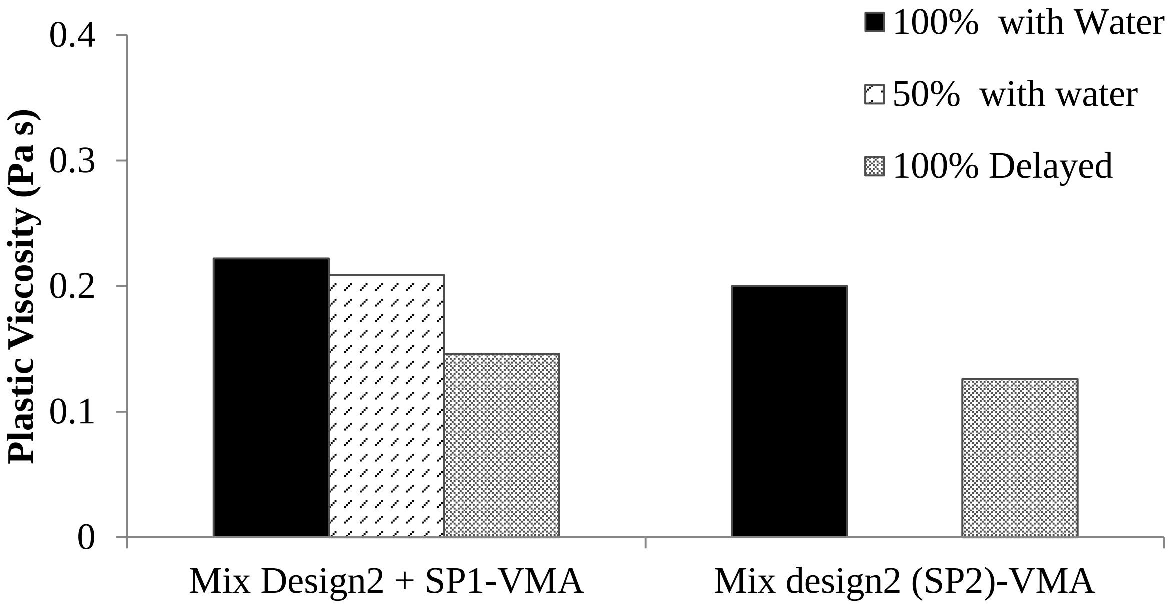

Figure 44. Influence of the type of SP on Plastic Viscosity. Left: Ref mix design 2 without VMA with SP 1, Right: Ref mix design 2 without VMA, with SP 2.

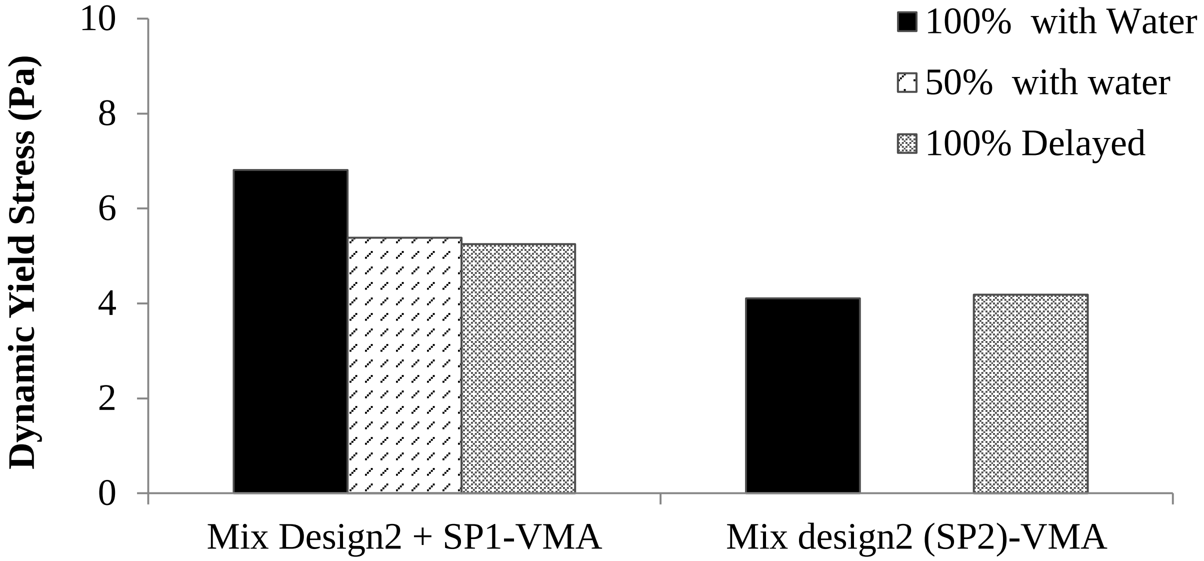

Figure 45. Influence of the type of SP on Dynamic Yield Stress. Left: Ref mix design 2 without VMA with SP 1, Right: Ref mix design 2 without VMA, with SP 2.

6.2.4 Binder Combinations

The changes in plastic viscosity and yield stress due to a change in adding time of SP are shown in Fig. 46 and 47 respectively for mixture 2 (left) and mixture 2 without fly ash and silica fume (right). The results on the right are thus obtained on mixtures with 100% cement as powder material and the replacement of fly ash and silica fume was done by volume to keep the water content in the mixtures constant. The dosage of SP is adjusted for the mixtures with the delayed addition to obtain the target mini-slump flow. Both mixtures contained an equal dosage of VMA. As can be observed in Fig. 46, the viscosity of the mixture without silica fume and fly ash is significantly higher than the reference mixture, because of the absence of SF and FA. However, the difference between viscosity at 100% SP with water and 100% delayed is approximately equal (0.0847 and 0.0850 Pa s), illustrating that the presence of SF and FA does not affect the robustness of cement paste. A similar conclusion on the robustness of the cement pastes to the variation in adding time of SP, with and without SF and FA, can be drawn from Fig. 47 showing the yield stresses.

As the presence of fly ash and silica fume does not significantly affect robustness, the influence of the replacement of cement (16%) by limestone filler can be directly compared to the reference mixture. However, no VMA was added to the mixture with limestone filler, so the comparison is done with the reference mixture without VMA, as discussed in section 6.3.1. Figure 48 shows the plastic viscosity evolution as a function of the adding time of SP for the VMA-type reference mixture without VMA (left) and the same mixture in which all SF and FA is replaced by cement and 16% of cement is replaced by limestone filler. It can be clearly observed that for these mixtures, the robustness is enhanced by the addition of the limestone filler.

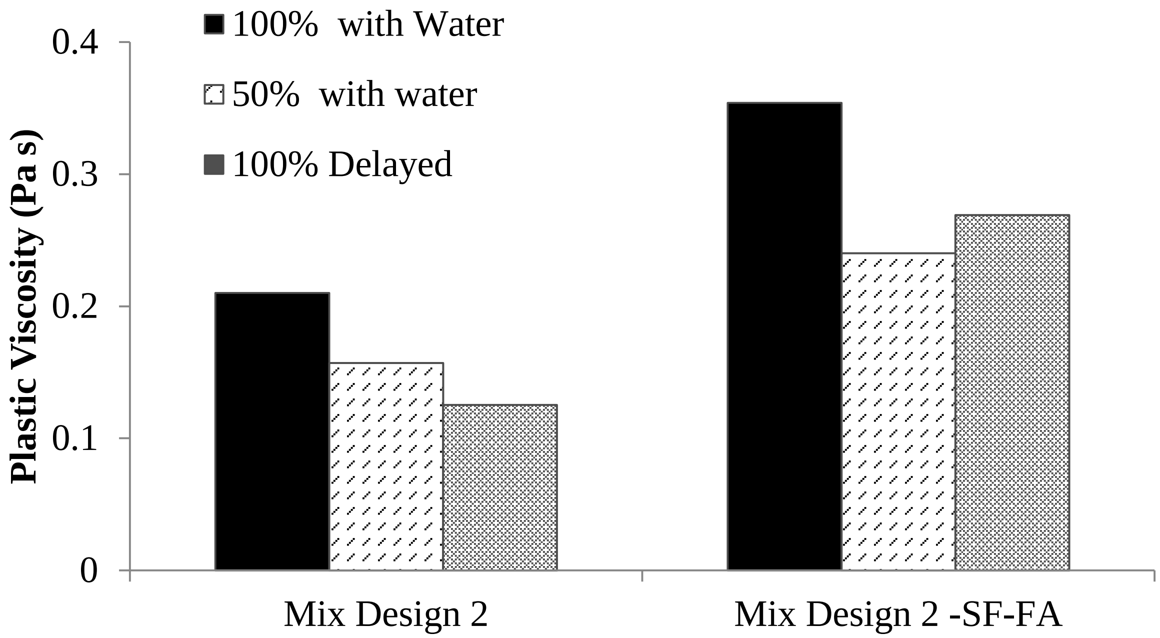

Figure 46. Influence of presence of Silica Fume and Fly Ash on Plastic Viscosity. Left: Ref mix design 2, Right: Ref mix design 2 in which all silica fume and fly ash are replaced by cement (by vol.).

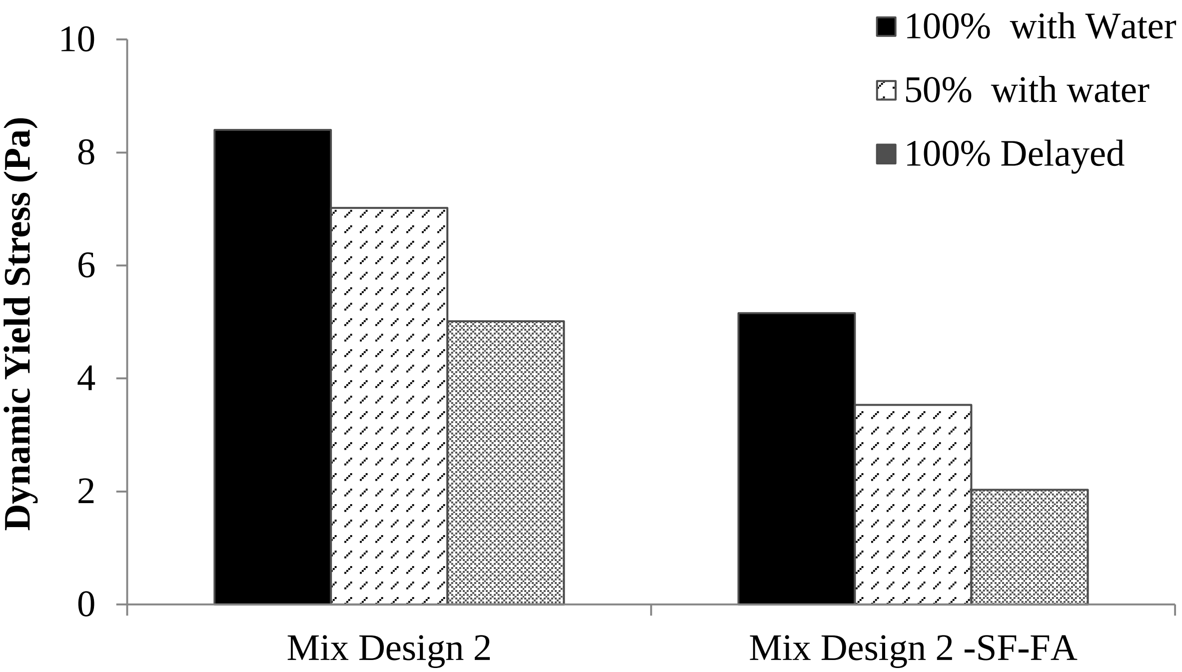

Figure 47. Influence of presence of Silica Fume and Fly Ash on Dynamic Yield Stress. Left: Ref mix design 2, Right: Ref mix design 2 in which all silica fume and fly ash are replaced by cement (by vol.).

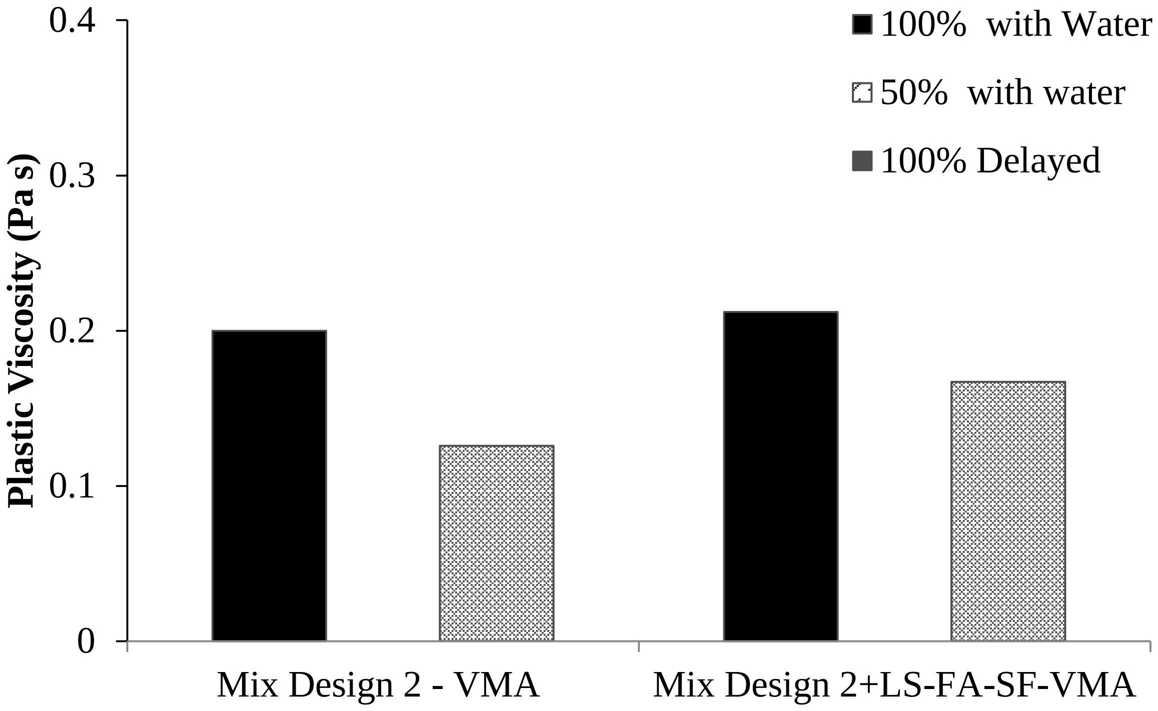

Figure 48. Influence of presence of Limestone on Plastic Viscosity. Left: Ref mix design 2 without VMA, Right: Ref mix design 2 without VMA in which all silica fume and fly ash are replaced by cement (by vol.), and 16 % of the cement is replaced by limestone filler.

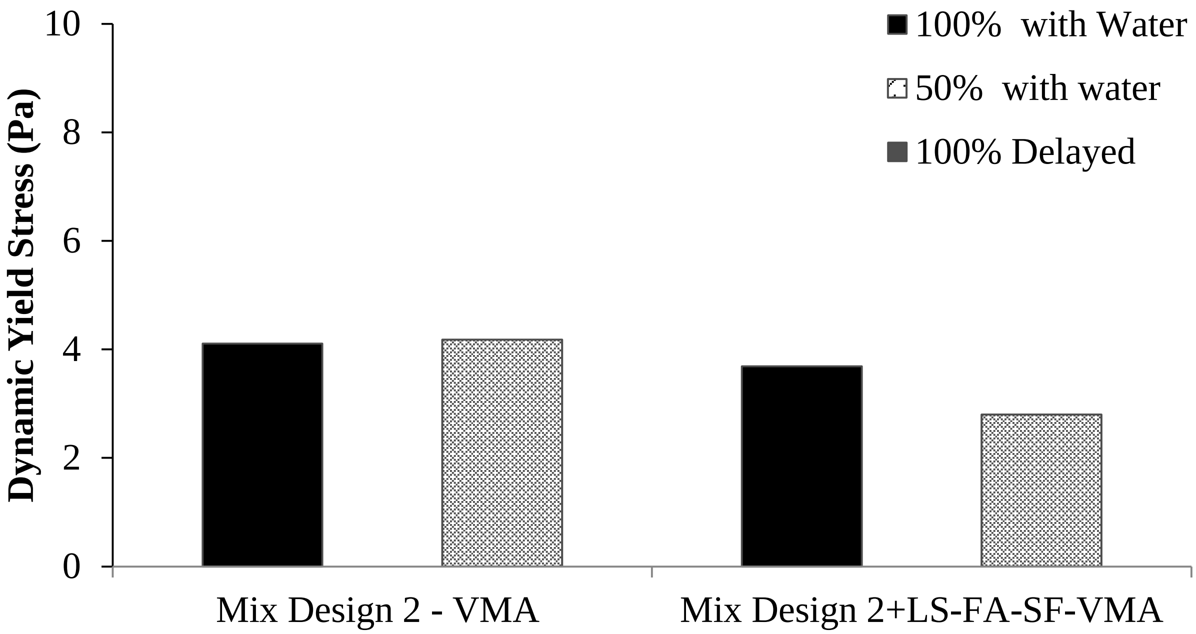

Figure 49. Influence of presence of Limestone on Dynamic Yield Stress. Left: Ref mix design 2 without VMA, Right: Ref mix design 2 without VMA in which all silica fume and fly ash are replaced by cement (by vol.), and 16 % of the cement is replaced by limestone filler.

6.2.3 Summary

Concerning the robustness of the powder-type SCC cement pastes, it appeared that the mixtures show most sensitivity to a change in water content and that a change in mixing time and adding time of the SP did not significantly affect the robustness of the mixture. For the VMA-type SCC cement pastes studied, the adding time of the SP significantly affected the rheological properties, in combination with the amount of water added. It even appeared that the adding time of the SP had an as large or even larger effect on the rheological properties compared to the studied variations in water content. Studying more in detail the VMA-type mixtures, the study revealed that the interaction between cement and admixtures, altered by cement deliveries, and the presence of limestone filler act as main mix design parameters influencing the robustness of the cement pastes. When especially focusing on the plastic viscosity of the mixtures, the presence of the VMA, a change in the commercial SP product and the presence of silica fume and fly ash affected the robustness to a significantly lesser extent.

7 Task 3: Robustness of Mixing Procedure and Addition Sequence of Materials on Concrete

7.1 Induced Variations on Concrete Scale

In this part of the experimental program, the influence of small variations in the water content, differences in mixing speed, mixing time, the addition sequence of the aggregates, and different moisture contents of the sand are studied. The variations of the fresh properties immediately after mixing are discussed in this section, while in the next chapter, the variations on thixotropy and workability loss will be shown.

The reference mix design is a powder-type SCC. The target slump flow measured at 20 minutes after contact between water and cement of the reference mixture is 700 mm to study the influence of variations in the mixing time, the addition sequence of aggregates, and the moisture content of the sand, and 600 mm to study the influence of variations in the water content. The details on the mix design can be found in section 4.4.2. The following subsections describe the induced variations to the concrete mix design or mixing procedure, focusing on the mixing energy (directly monitored by the Eirich mixer) and the stabilization time.

Every mixture was produced in an intensive mixer registering the power consumption during mixing. Based on the measured power consumption curve, the stabilization time can be calculated as the moment at which the power curve reaches a horizontal asymptote (an exponential fit of the power consumption curve reaches a slope of -4.10^{-4} kW/s).

7.1.1 Variations in Water Content

The water content is varied by ± 10 l/m^3 of concrete relative to the reference mixture, reflecting typical robustness studies. Figure 50 shows the mixing energy over time for the mixtures which are logically ranked according to the water content, as a small increase in water content reduces power consumption. The figure also shows the stabilization time of the mixes, which is only slightly affected by the variation in water content.

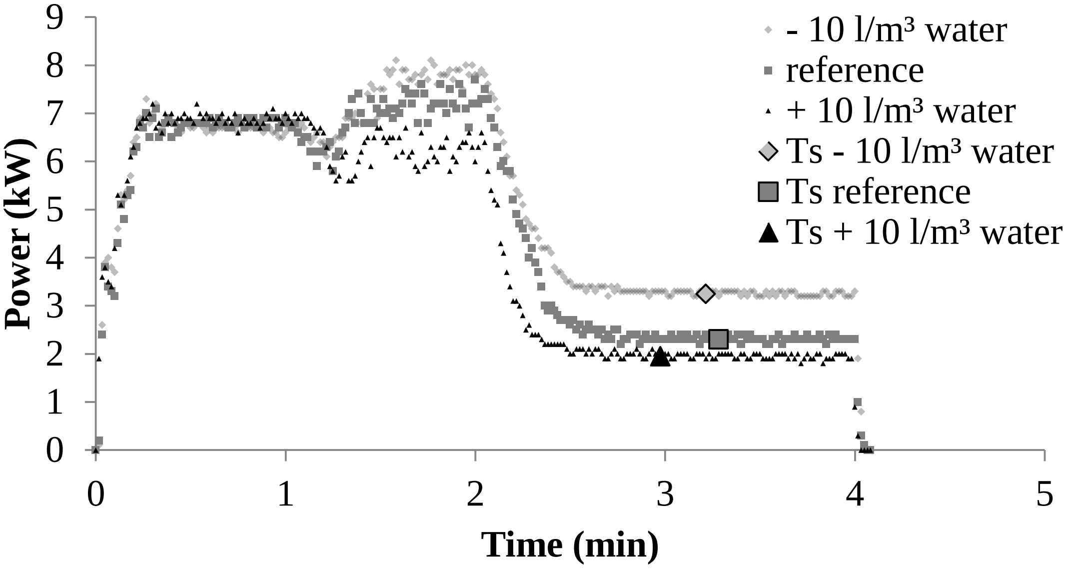

Figure 50. The influence of a variation in the water content on the stabilization time.

7.1.2 Variations in Mixing Speed

Employing the same mixing procedure and identical mix design, the influence of the mixing speed can be investigated. The mixing speeds are labeled as low, medium (ref) and high, corresponding to a rotor speed of 3.5, 7.0, and 13.7 m/s and a pan speed of respectively 8.7, 17.0, and 26.0 rpm. The variation of mixing speed resulted in significantly different fresh properties. The mixing energy and stabilization times are shown for the three procedures in Figure 51, indicating a clear difference in stabilization time. Although the difference in stabilization time between the mixture at high speed and reference speed is not large (Figure 51), the mixture at high speed is overmixed, judging by the slump flow of 400 mm (see further).

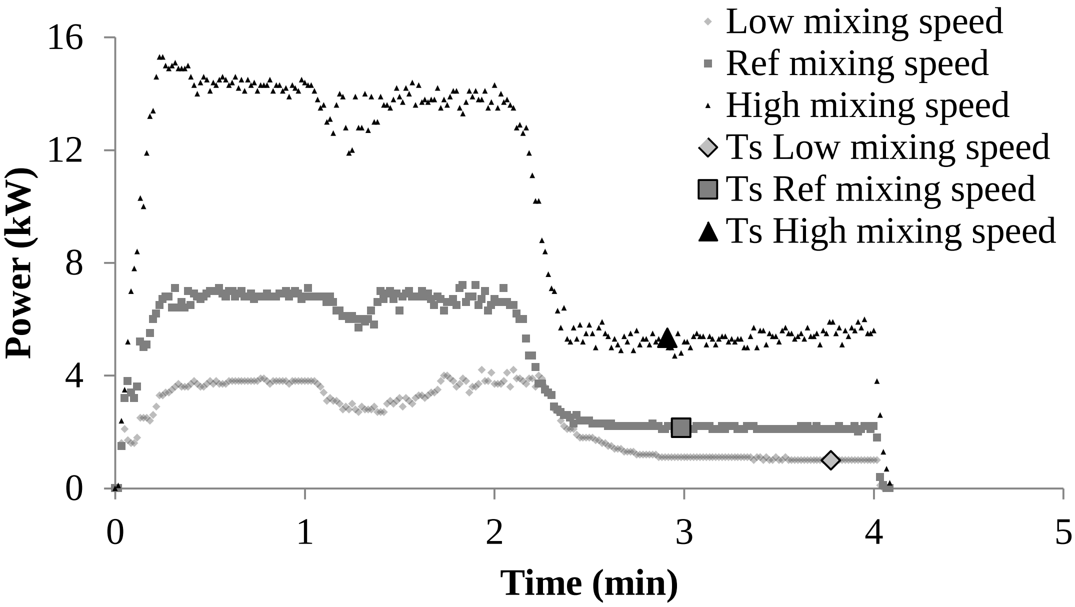

Figure 51. Influence of the mixing speed on the stabilization time

Table 9. Mixing procedures in drum and intensive mixer.

	Drum mixer	**Intensive mixer**
Aggregates	30 sec	30 sec
Half of water	30 sec	30 sec
Cement and filler	1 min	1 min
Scraping	1.5 min	-
Half of water	30 min	1 min
VMA and SP	2 min	2 min
Scraping	30 sec	-
Mixing	2.5 min	-
Total mixing time after contact cement and water	**8 min**	**4 min**

In addition, the reference powder-type mix design was reproduced in a drum mixer, in which the mixing process is provoked by gravity. In order to obtain equal slump flow as in the Eirich mixer, the SP dosage in the drum mixer was significantly reduced: from 3.88 kg/m^3 in the Eirich mixer to 2.58 kg/m^3 in the drum mixer. The lower SP dosage reflects the lower mixing energy of the drum mixer, despite the longer mixing time (Table 9), and thus the lower capacity of dispersing the formed clusters of cement particles. As small cement particles (or clusters) are responsible for the yield stress, dispersing more of them by mixing requires more SP to cover the larger surface area and prevent them from re-coagulating. The viscosity of the mixture in the high-shear mixer is expected to be lower than the viscosity of the drum mixer, as entrapped water from within the clusters becomes available for flow.

7.1.3 Variations in Mixing Time

The reference mixture was reproduced three times varying in mixing time (2.5 min, 3.5 min, and 4.5 min) according to the mixing procedures summarized in Table 10. The mixing energies with fitted power curves are shown in Figure 52 to Figure 54, for the short, medium and long mixing time, respectively. As can be seen, the mixing time does, logically, not affect the stabilization time significantly, as for all three procedures, the stabilization time is around 2.25 min.

Table 10. The mixing procedures, varying in mixing time.

Short mixing time	**Medium mixing time**	**Long mixing time**
Aggregates 1 min	Aggregates 1 min	Aggregates 1 min
Cement and filler 1 min	Cement and filler 1 min	Cement and filler 1 min
Water 40s	Water 1 min	Water 1 min
SP 110 s	SP 2.5 min	SP 3.5 min
Mixing time: 2.5 min	**Mixing time: 3.5 min**	**Mixing time: 4.5 min**

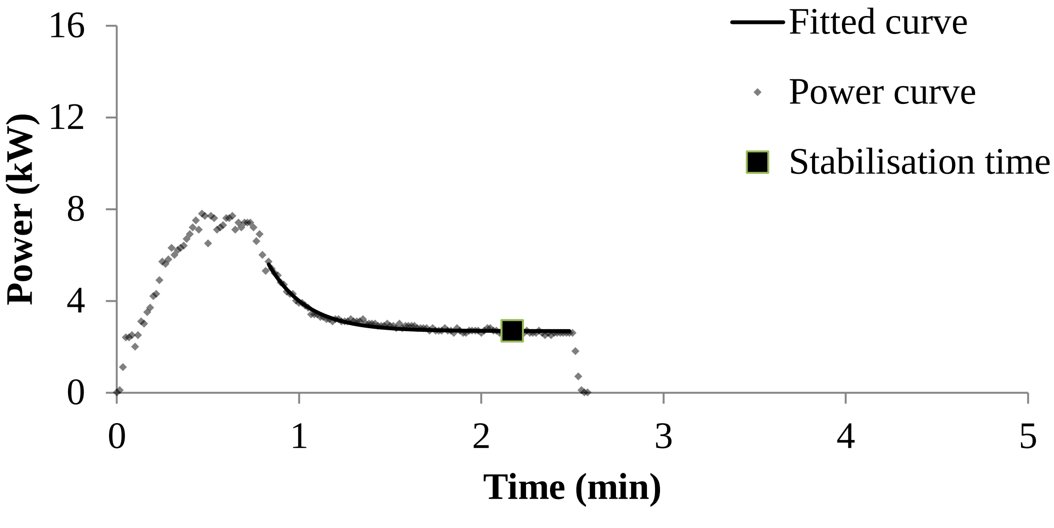

Figure 52. The power curve with a short mixing time.

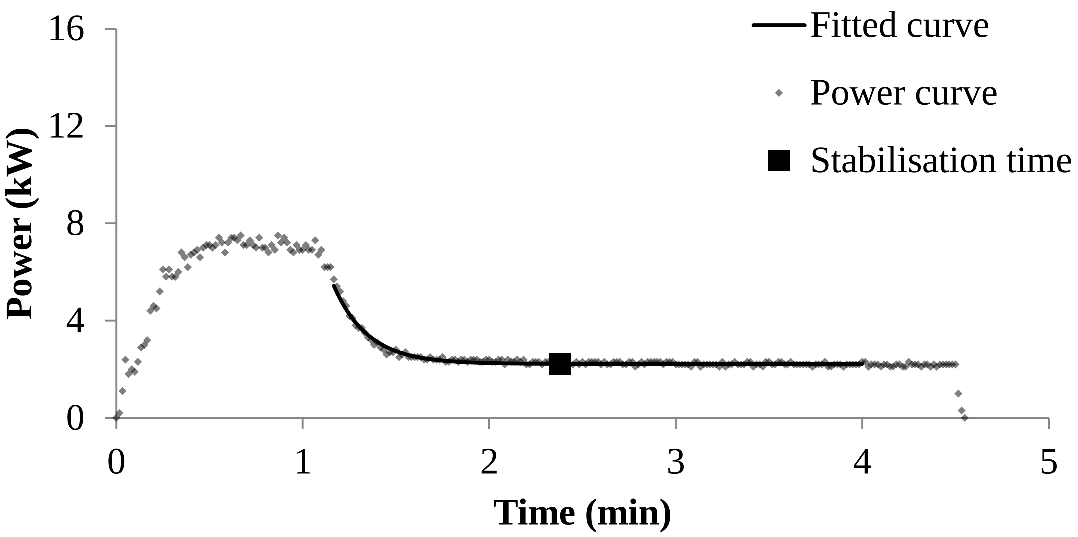

Figure 53. The power curve with a long mixing time.

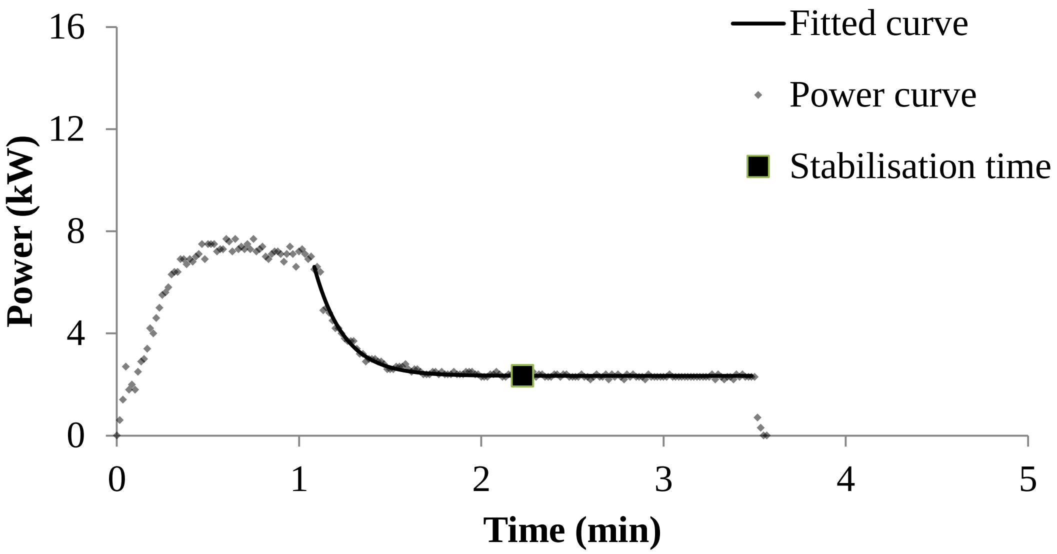

Figure 54. The power curve with a medium mixing time.

7.1.4 Variations in Addition Sequence of the Aggregates

The influence of four different addition sequences of the aggregates, cement, filler, and water on the workability and rheology was studied in this experimental program. The four addition sequences are summarized in Table 11. All aggregates were air-dry when introduced into the mixer.

Table 11. The different mixing procedures, varying in addition sequence.

SCC1	**SCC2**	**SCC3**	**SCC4**
Aggregates 1 min	Aggregates 30 sec	Cement and filler 1 min	Cement and filler 1 min
	Half of water 30 sec		
Cement and filler 1 min	Cement and filler 1 min	All water 1 min	All water 1 min
All water 1 min	Half of water 1 min	SP 1 min	Aggregates 1 min
SP 2.5 min	SP 2 min	Aggregates 2.5 min	SP 2.5 min

7.1.5 Variations in Initial Moisture Content of the Aggregates

As the absorption of water by the aggregates seemed to play a major role on the rheological behavior of SCC, the initial moisture content of the sand could also influence the rheological behavior. For two identical mixtures, using the same mixing sequence, the water and air-dry aggregates were added together in the mixing pan in one mixture, while for the other mixture, all water was poured over the sand and soaked for one night to ensure full absorption by the aggregates. In a similar experiment in which the influence of the moisture content on the slump flow evolution was examined, described in [39], large differences in the slump flow evolution were observed when sand with a moisture content of 3% instead of 0.2% was used in SCC, keeping the total amount of water in the mixture constant. Any evaporation of water from the buckets with sand was prevented by closing them with a plastic cover. In order to maximize the effect of soaking, the following mixing procedure was applied:

- Aggregates and water were mixed for 30 seconds.
- Cement and filler were added and mixed for 1 minute.
- Superplasticizer was added and mixed for 2.5 minutes

7.2 Observations on Workability and Rheological Properties

For each mixture, the rheological parameters were measured at 10 minutes after contact between cement and water, and the slump flow, V-funnel flow time and Sieve Segregation Index were determined at 20 minutes.

7.2.1 Variations in Water Content

As shown in Table 12, a variation of ± 10 l/m^3 water (± 6% of the total water content) in the mix composition of SCC (with target slump flow of 600 mm) has a significant impact on the workability parameters. The mixture with the lowest water content had a slump flow of 465 mm and thus could not even be considered as SCC. The effect of water content on the rheological parameters indicates that the variation in water content results in a significant change in yield stress and viscosity of the mixtures. Both the yield stress and the plastic viscosity decrease with the same order of magnitude as the water content increases. Despite the change in water content, all mixtures resisted segregation (SSI < 15%), most probably due to the low slump flow of the reference mixture and the small aggregates employed.

Table 12. The influence of variations in the water content on the fresh properties of SCC.

	- 10 l/m³ water	Reference	+ 10 l/m³ water
Slump flow (mm)	465	595	700
V-funnel time (s)	12.5	5.1	4.5
Sieve Segregation Index (%)	0.1	2.3	9.1
Dynamic yield stress (Pa)	85	36	20
Plastic viscosity (Pa s)	52	31	17

7.2.2 Variations in Mixing Speed

Varying the mixing speed has a significant influence on the workability and rheology of the concrete, as shown in Table 13. A significant increase in yield stress can be observed with increasing mixing speed, while the plastic viscosity only significantly increased from the intermediate to the very high speed. Increasing mixing speed increases the dispersion of the cement particles during mixing. As the quantity of SP was not varied, the amount of SP relative to the surface of cement particles decreases, resulting in a lower yield stress, and eventually, in a higher plastic viscosity. However, the mixing speed was very high and amplifies the observations, compared to standard mixing speeds.

Table 13. The influence of mixing speed on the fresh properties of SCC.

	Low speed	Intermediate speed	High speed
Slump flow (mm)	800	690	405
V-funnel time (s)	3.6	4.3	8.8
Sieve Segregation Index (%)	8.5	5.2	0.0
Dynamic yield stress (Pa)	13	21	161
Plastic viscosity (Pa s)	26	25	31

Table 14 shows the influence of the mixer type on the fresh properties. At first glance, no difference in yield stress and slump flow is observed, but the quantity of SP was changed. If the same quantity of SP would have been used in the drum mixer, as was used in the Eirich mixer, the SCC from the drum mixer would have shown severe segregation, invalidating all measurements. Instead, the decrease in SP demand to obtain the same slump flow reflects the decrease in yield stress due to the lower mixing energy. However, the plastic viscosity increases when switching from the Eirich mixer to the drum mixer, indicating a lower dispersion of cement particles in the drum mixer. These results are in agreement with the decreased SP-demand. Note that the concrete mixtures discussed in this section were made with a different delivery of cement compared to the mixtures described above and those described in the next sections, necessitating the repetition of the reference mixtures with adjusted SP dosage.

Table 14. The influence of mixer type on the fresh properties of SCC.

	Powder based SCC Drum mixer	Powder based SCC Intensive mixer
Superplasticizer dosage (kg/m³)	2.58	3.88
Slump flow (mm)	710	690
V-funnel time (s)	4.0	4.3
Sieve Segregation Index (%)	4.9	5.2
Dynamic yield stress (Pa)	21	21
Plastic viscosity (Pa s)	32	25

7.2.3 Variations in Mixing Time

The studied influence of the mixing time appears not to have a major influence on the workability and rheology of the mixtures. Slump flow, yield stress and sieve segregation index all appear approximately constant. A slight decrease in plastic viscosity can be observed with increasing mixing time, which is confirmed by a minor decrease in V-Funnel flow time. The potential cause for this behavior could be the additional dispersion of clusters of fine particles due to the extended mixing time. However, this action must be minor as the adsorption and dispersion action of the superplasticizer is not affected (constant yield stress). Extending the mixing time beyond the studied interval will most likely increase yield stress and plastic viscosity due to overmixing, similar to the influence of increasing the mixing speed.

Table 15. The influence of the mixing time on the fresh properties of SCC.

	Short mixing time (2.5 min)	Intermediate mixing time (3.5 min)	Long mixing time (4.5 min)
Slump flow (mm)	760	705	730
V-funnel time (s)	4.0	3.9	3.6
Sieve Segregation Index (%)	6.9	6.0	7.5
Dynamic yield stress (Pa)	17	16	16
Plastic viscosity (Pa s)	38	27	22

7.2.4 Variations in Addition Sequence of the Aggregates

Table 16 summarizes the influence of the addition sequence on the fresh concrete. The following three cases can be distinguished:

- The mixture in which the aggregates are premixed with part of the water, before the cement and filler are added in the mixer, has the lowest fluidity: a lower slump flow, a slightly higher V-funnel time, the lowest Sieve Segregation Index, and a higher yield stress and plastic viscosity.
- The mixtures in which the cement, filler and water are first mixed together, before the aggregates and superplasticizer are added to the mixer have the highest fluidity: a higher slump flow, a slightly lower V-funnel time, the lowest yield stresses, and a low plastic viscosity. Both mixtures also have a slightly higher Sieve Segregation Index.
- When the aggregates, cement and filler are first mixed together, an intermediate mix with a slump flow, V-funnel time, Sieve Segregation Index, yield stress and plastic viscosity in between the two other cases is produced.

Table 16. The influence of the addition sequence on the fresh properties of SCC.

	Aggregates ½ Water Cement ½ Water SP	Aggregates Cement Water SP	Cement Water SP Aggregates	Cement Water Aggregates SP
Slump flow (mm)	680	705	775	760
V-funnel time (s)	4.6	3.9	3.8	3.8
Sieve Segregation Index (%)	2.9	6.0	6.6	7.9
Dynamic yield stress (Pa)	27	16	9	13
Plastic viscosity (Pa s)	32	27	29	26

A possible explanation for these observations is the absorption of part of the water by the aggregates, as the aggregates were air-dry and attempt to reach fully saturated condition. When the water and cement are first mixed together, a paste is formed before any contact with the aggregates. Because the absorption of water by the aggregates is reduced or even prevented, more water is available in the paste, causing the paste, and thus the concrete, to be more fluid. When the cement and aggregates are mixed together before the water is added to the mixer, the absorption of water by the aggregates is in between the two above cases, resulting in a concrete with an intermediate fluidity.

7.2.5 Variations in Initial Moisture Content of the Aggregates

As shown in Table 17, the soaked aggregates in which more water is absorbed by the aggregates, results in a mixture with a lower slump flow and higher yield stress, but no different V-funnel time, sieve segregation index, and plastic viscosity. Probably, the absorption of water into the pores of the air-dry aggregates during the 30 sec of premixing is not fast enough to reach full saturation before the addition of the cement and filler to the mixer, and thus more water is available in the paste when air-dry aggregates are used instead of soaked aggregates. Compared to the four addition sequences from the previous section, the V-Funnel and viscosity values are in the range of the case where the aggregates and half of the water was mixed and are significantly higher than the case where aggregates were added in a cement paste.

Table 17. The influence of the moisture content of the aggregates on the fresh properties of SCC.

	Air-dry aggregates	Soaked aggregates
Slump flow (mm)	760	690
V-funnel time (s)	5.1	4.5
Sieve Segregation Index (%)	3.1	4.6
Dynamic yield stress (Pa)	15	19
Plastic viscosity (Pa s)	37	34

7.3 Ranking of Influential Parameters

The influence of all studied parameters on the slump flow and V-funnel time is illustrated in Figure 55, globally showing a more important effect of the variation in water content than the other parameters. A more detailed graph of the other parameters is shown in Figure 56, not considering the influence of the water content. All V-funnel time measurements are between 3.5 and 5 seconds and thus have a repeatability of 0.6225 – 1.155 [10], making it impossible to draw any significant conclusions from the V-funnel time variations.

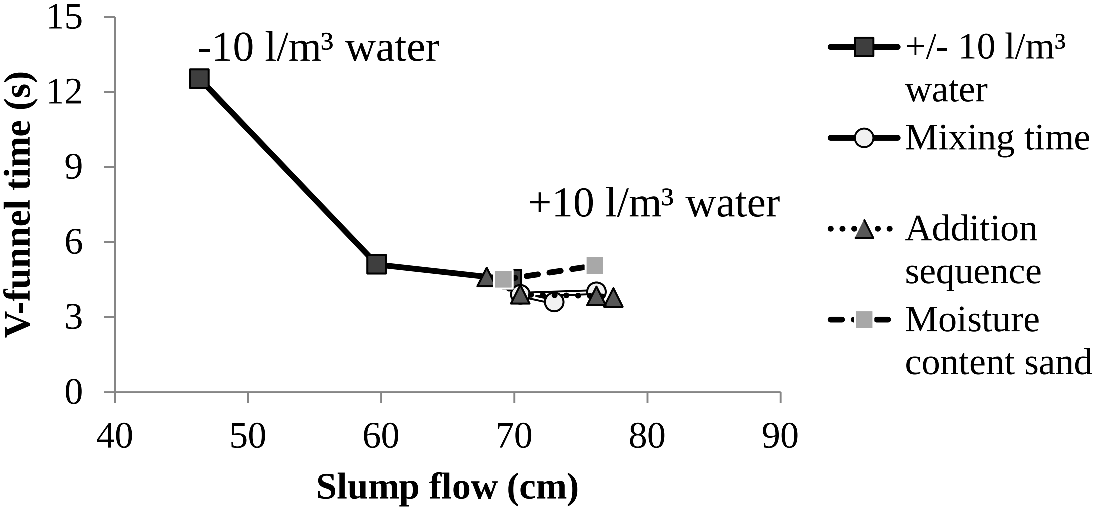

Figure 55. Ranking of the parameters – Slump flow and V-funnel time (a).

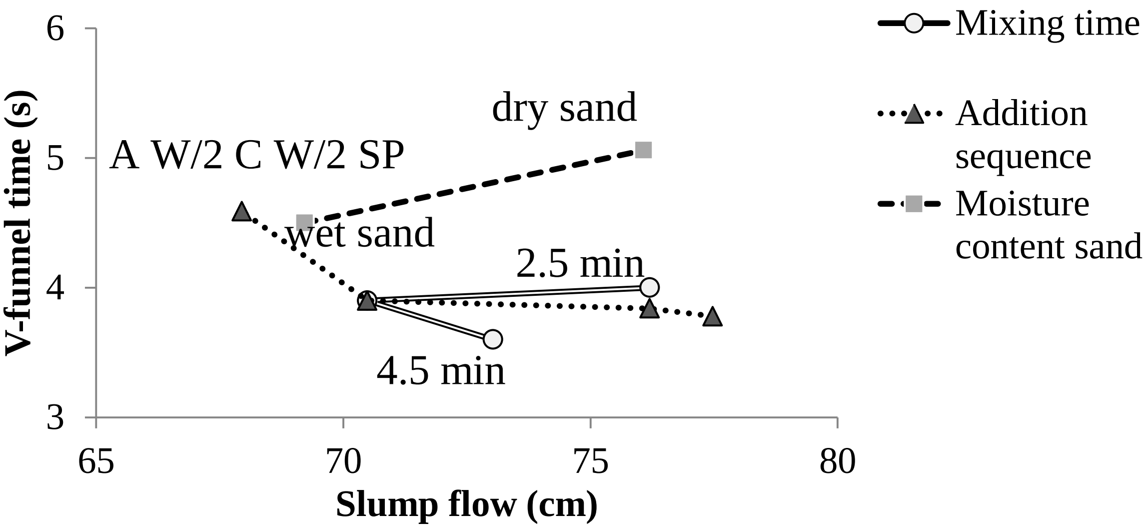

Figure 56. Ranking of the parameters – Slump flow and V-funnel time (b).

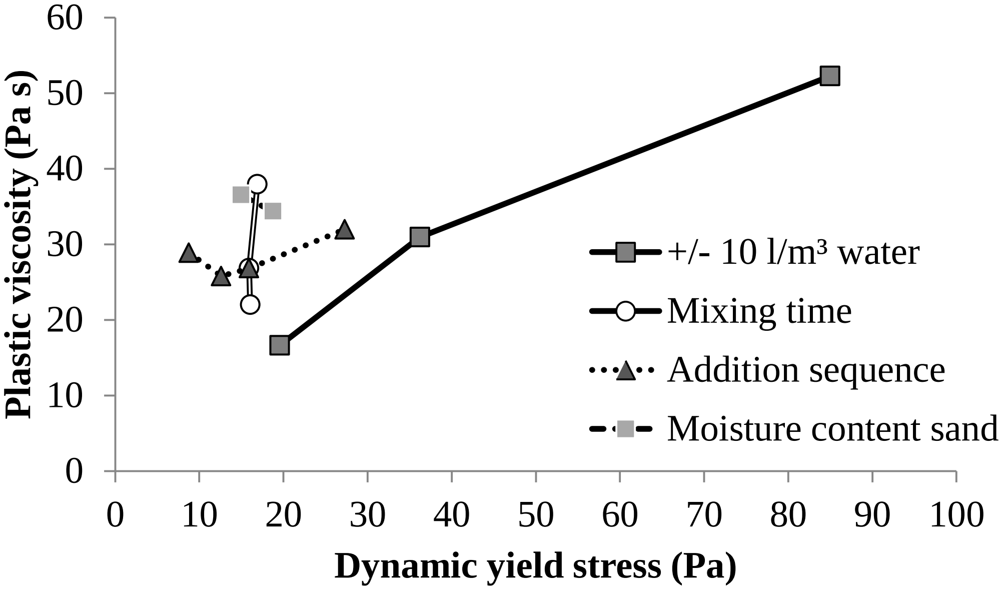

Figure 57. Ranking of the parameters – Yield stress and Plastic Viscosity (a).

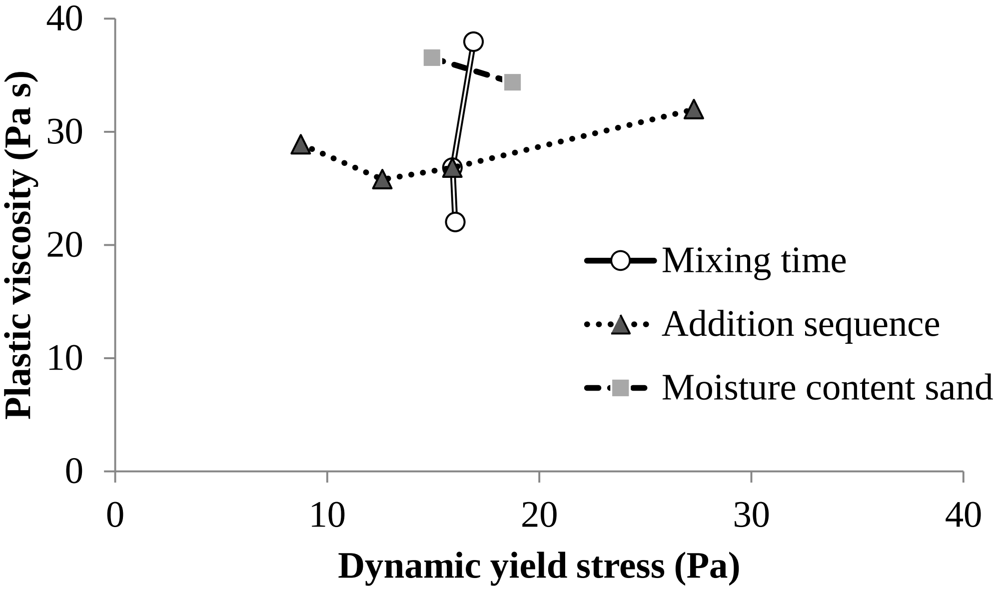

Figure 58. Ranking of the parameters – Yield stress and Plastic Viscosity (b).

Due to the variability of the rheological measurements, it is more difficult to assess whether the impact of a certain mixing parameter is significant or not. However, from Figure 57, it can be clearly deducted that variations in the water content are more significant than the other induced variations. The mixing speed is not included in the analysis, due to the disproportional variation in mixing speed compared to practical procedures. The addition sequence and moisture content mainly affect the dynamic yield stress of the produced mixture (Figure 58). The mixing time is the only parameter which causes significant differences in the plastic viscosity.

7.4 Summary

Variations in water content of 10 l/m^3 and the investigated mixing speed have the largest influence on workability and rheological properties of the studied powder-type SCC. The mixing time, in the investigated interval, and the addition sequence of all materials also influence the rheological properties, but to a lesser extent than the water content and mixing speed. Concerning the addition sequence of the materials, interesting results were obtained on the absorption of mixing water by the aggregates. If the aggregates, which have a moisture content lower than SSD, are added to cement paste, the cement paste adsorbs onto the aggregate surface and the aggregates absorb a significantly lower amount of water than when they are premixed with the mixing water. This leads to a more fluid SCC in the first case (aggregates in paste) than in the second case (aggregates premixed with water). More interestingly, the research team is not certain whether the aggregates reach full saturation when being briefly premixed with water before the cement is added. Attempts to study the rate of absorption of the aggregates were unsuccessful. However, if the aggregates are below SSD condition, it is anticipated that they will not fully absorb the "correction" amount of added water, leading to a more fluid mixture. This anticipation is in agreement with Neville's assumption [69]:

"Normally it is assumed that at the time of setting of concrete the aggregate is in a saturated and surface-dry condition. It the aggregate is batched in a dry condition, it is assumed that sufficient water will be absorbed from the mix to bring the aggregate to a saturated condition, and this absorbed water is not included in the net or effective mixing water. It is possible, however, that when dry aggregates is used the particles become quickly coated with cement paste which prevents further ingress of water necessary for saturation. This is particularly so with coarse aggregate, where water has further to travel from the surface of the particle. As a result, the effective water/cement ratio is higher than would be the case had full absorption of water by the aggregate been possible.

This effect is significant mainly in rich mixes where rapid coating of aggregate can take place; in lean, wet mixes the saturation of aggregates proceeds undisturbed. In practical cases the actual behavior of the mix is affected also by the order of feeding the ingredients into the mixer." A.M. Neville [69].

The opposite case, when aggregates are above SSD condition has not been studied, but its effects are expected to be less significant, as long as the moisture content is precisely controlled and adequately corrected.

8 Task 4: Influence of Mixing Procedure on Thixotropy and Workability Loss

In the three previously described tasks, the influence of specific constituent elements or mixing procedure on the initial rheological properties (max. 15 min after the addition of the water) was studied. In this task, the “robustness” of the evolution of the rheological properties with time is discussed, meaning that the influence of several of the influencing parameters described in the previous sections is determined. The evolution in time of rheological properties of cement-based materials is influenced by two different phenomena: thixotropy, which is a reversible dispersion and coagulation process and workability loss, which is the consequence of the chemical reactions occurring in the dormant period of the hydration process. Thixotropic properties were only measured on concrete scale, as a second rheometer is necessary to keep the sample undisturbed between measurements.

8.1 Workability Loss of Cement Pastes

8.1.1 Parameters Describing Workability Loss

The average rate of change of the rheological properties (plastic viscosity and dynamic yield stress) over 1 hour has been calculated. The method of calculation is as follows for plastic viscosity. First the change of rheological properties at 30, 45 and 60 minutes has been obtained by taking the difference between the newly measured plastic viscosity and the previous value, divided by the time lapse (equations 10-12). Finally, the average rate of change of the plastic viscosity was determined by calculating the average of the three previously obtained values (eq. 13). This method was preferred to reduce the influence on the rate of increase with time caused by small errors in the individual measurements. The rate of increase with time of the dynamic yield stress has been calculated using the same method.

At 30 mins: $$\Delta\mu_p / \Delta t_{30} = \frac{\mu_{p,30} - \mu_{p,15}}{15} \quad \text{(Eq. 10)}$$

At 45 mins: $$\Delta\mu_p / \Delta t_{45} = \frac{\mu_{p,30} - \mu_{p,15}}{15} \quad \text{(Eq. 11)}$$

At 60 mins: $$\Delta\mu_p / \Delta t_{60} = \frac{\mu_{p,30} - \mu_{p,15}}{15} \quad \text{(Eq. 12)}$$

$$\Delta\mu_p / \Delta t = \frac{\Delta\mu_p / \Delta t_{30} + \Delta\mu_p / \Delta t_{45} + \Delta\mu_p / \Delta t_{60}}{3} \quad \text{(Eq. 13)}$$

8.1.2 Influence of the type of SP

The two different SPs utilized in this research project were commercial products from two different manufacturers. One product: SP 2, is specifically designed to extended the workability window beyond 1 hour, while the other product, SP 1, is more efficient, but has a shorter retention of workability. The specific influence of the different working action of the two SP can be clearly seen in Figures 59 and 60, where each SP was employed in both reference mixtures 1 and 2. The specific characteristics concerning workability retention of each SP are clearly reflected in Figures 59 and 60, as mixtures with SP 2 have clearly a slower increase in viscosity than the mixtures with SP 1. Furthermore, the yield stress data show that SP 2 causes in average a decrease in yield stress over the hour, while SP 1 demonstrates a clear increase over time. The observation on the cement pastes with SP 2 are in agreement with previous experiences with this SP, showing in most cases an increase in SCC slump flow over the first hour.

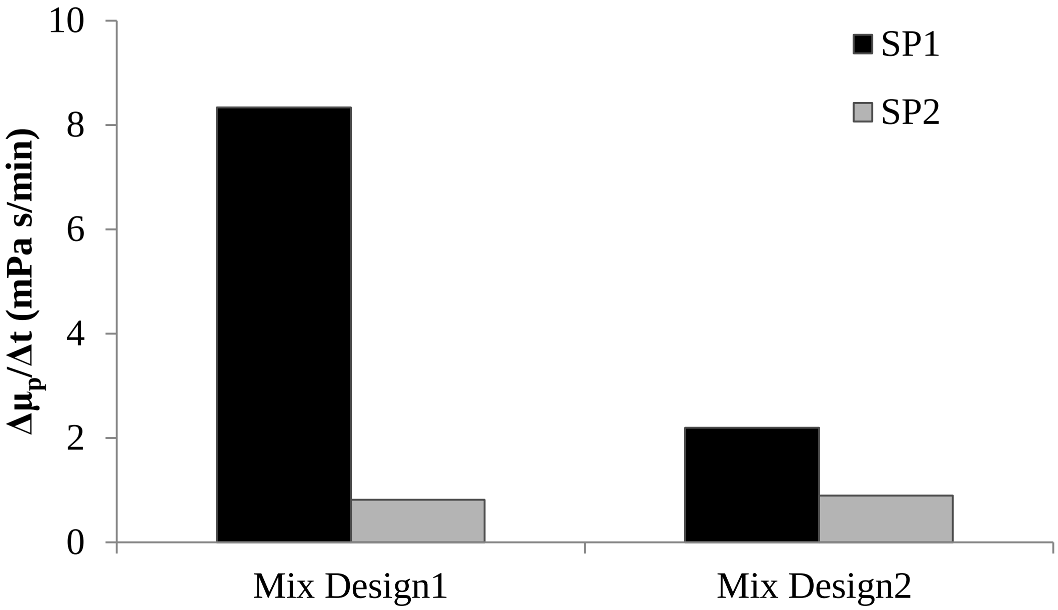

Figure 59. Rate of change of plastic viscosity with time (in mPa s/min) for reference mixtures 1 and 2, each produced with SP 1 and SP 2.

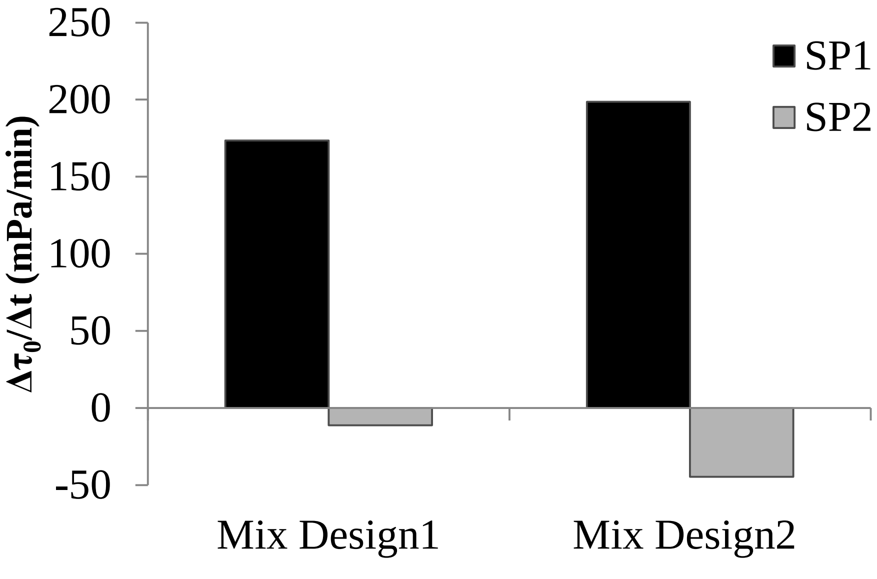

Figure 60. Rate of change of yield stress with time (in mPa/min) for reference mixtures 1 and 2, each produced with SP 1 and SP 2.

8.1.3 Repeatability

The 90% confidence limits for the rate of increase of yield stress or plastic viscosity were determined in the same way as described in section 5.1. The results are summarized in Table 18 and will be used to determine the significance of changes in mix design or mixing procedure.

Table 18. 90% confidence intervals for change of plastic viscosity and yield stress with time for reference mixtures 1, 2 and 3.

	Mix Design 1	Mix Design 2	Mix Design 3
Average Plastic viscosity increase with time (mPa s/min)	6.73	1.20	3.65
+90% confidence limit	7.02	1.25	4.24
-90% confidence limit	6.45	1.14	3.06
Average Yield stress increase with time (mPa/min)	106	9.11	61.9
+90% confidence limit	121	17.38	65.5
-90% confidence limit	92	0.84	58.4

8.1.4 Influence of Amount of SP Added

For reference mixtures 1 and 3, the variation in workability loss with a change in the amount of SP (SP 1) (± 10%) is displayed in Figures 61 and 62 for plastic viscosity and yield stress respectively. It should be noted that the units of the vertical axes are mPa s/min and mPa/min respectively, to enhance clarity of the graphs. It can be seen from Figure 61 that a reduction in the amount of SP enhanced the loss of workability, both for mixtures 1 and 3, while for mixture 3, a reduction in the amount of SP 1 added caused a slower increase in yield stress (Fig. 62).

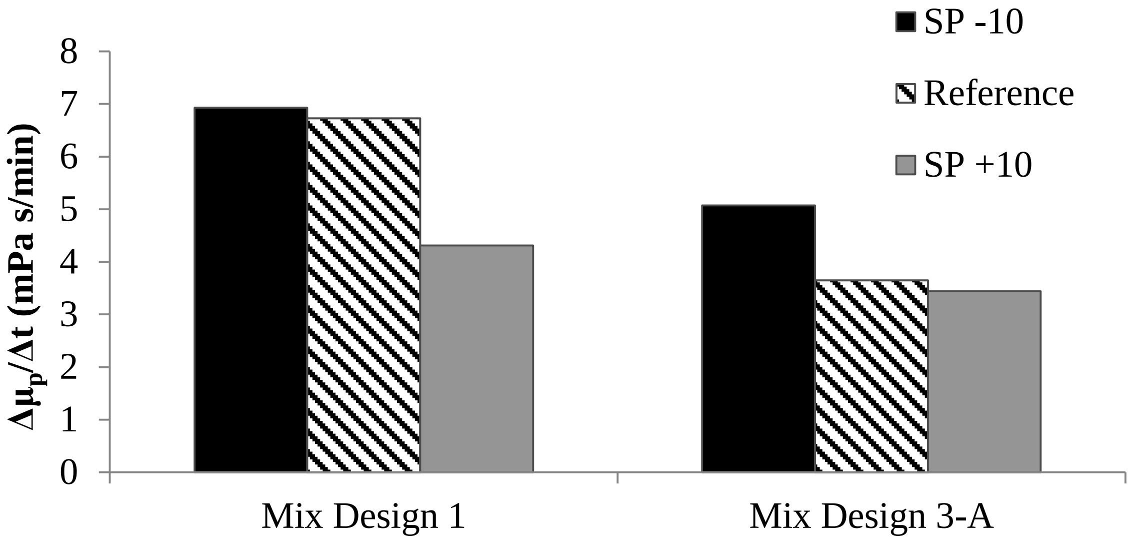

Figure 61. The rate of change of plastic viscosity (μ_p) with time, expressed in mPa s/min, as a function of the amount of SP (SP 1) added.

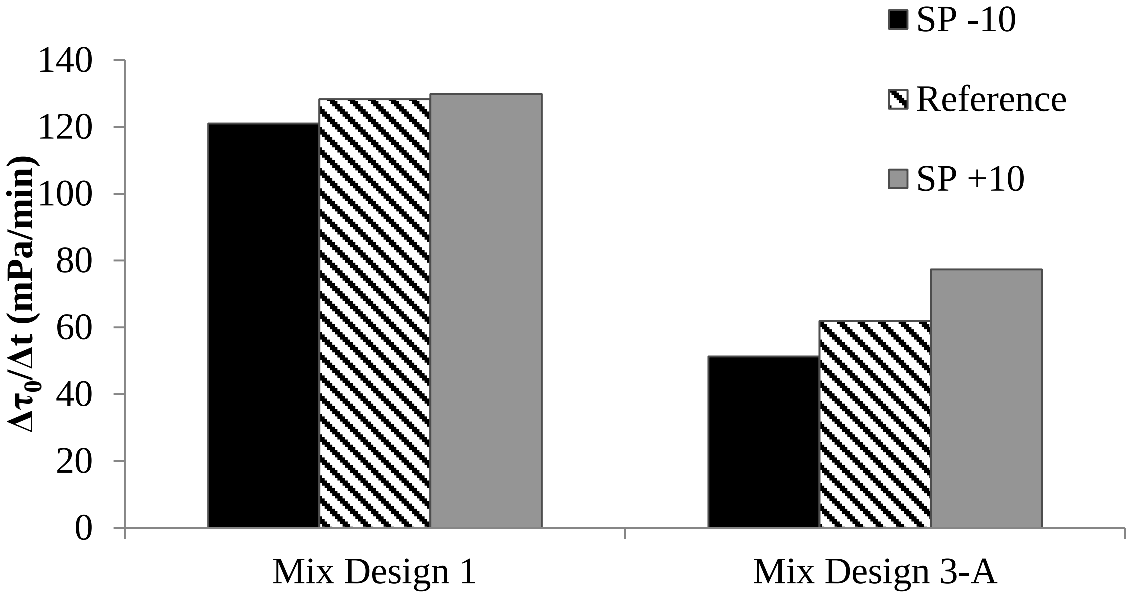

Figure 62. The rate of change of yield stress (τ_0) with time, expressed in mPa/min, as a function of the amount of SP (SP 1) added.

8.1.5 Influence of Mixing Energy

For reference mixtures 1 and 3, the influence of the mixing energy on the workability loss has been determined by varying the mixing time and mixing speed. The specific duration of each step with varying mixing time can be found in Table 3, while the mixing speed was changed from step 1 (ref) to step 2 in the Hobart mixer. Figures 63 to 66 show the influence of mixing time and mixing speed on the rate of change of plastic viscosity and yield stress for mixtures 1 and 3. The mixing time appears to mainly affect the yield stress for both mixtures, showing an increase in rate of increase of the yield stress with increasing mixing time. The increase of plastic viscosity with time is somewhat influenced but does not show any specific correlation with the mixing time. When changing the mixing speed, the increase of yield stress with time appears unaffected, while the rate of change of plastic viscosity with time is different when comparing the mixtures. While the increase in plastic viscosity with time was slowed for mixture 3 when mixing at the higher speed, mixture 1 has viscosity variations close to the size of the confidence interval. Further research is needed to explain the different behavior, although the observed changes are close to the 90% confidence intervals, showing they may be less significant compared to other parameters.

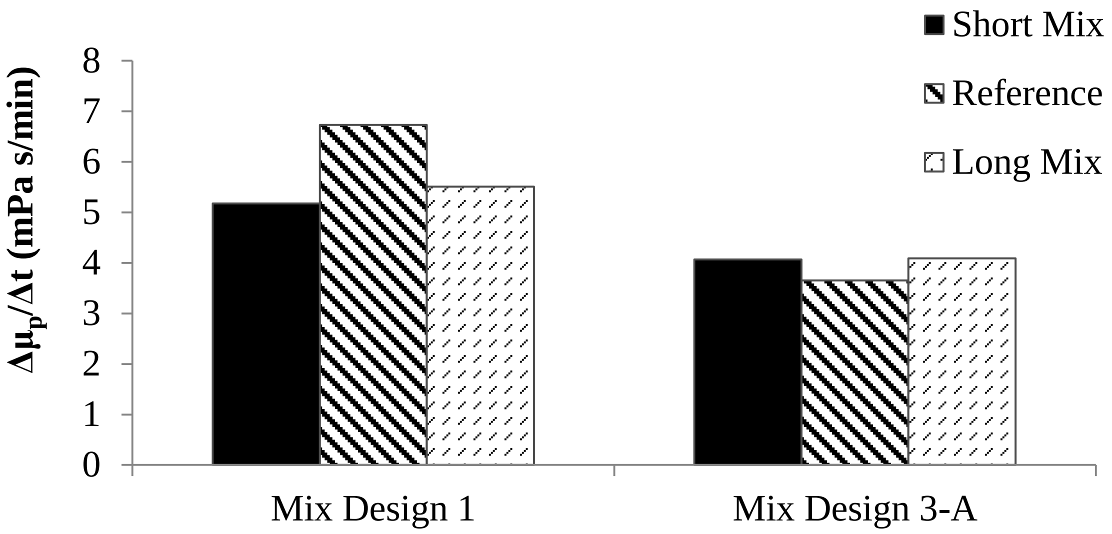

Figure 63. The rate of change of plastic viscosity with time, as a function of the mixing time (short, medium (ref) or long).

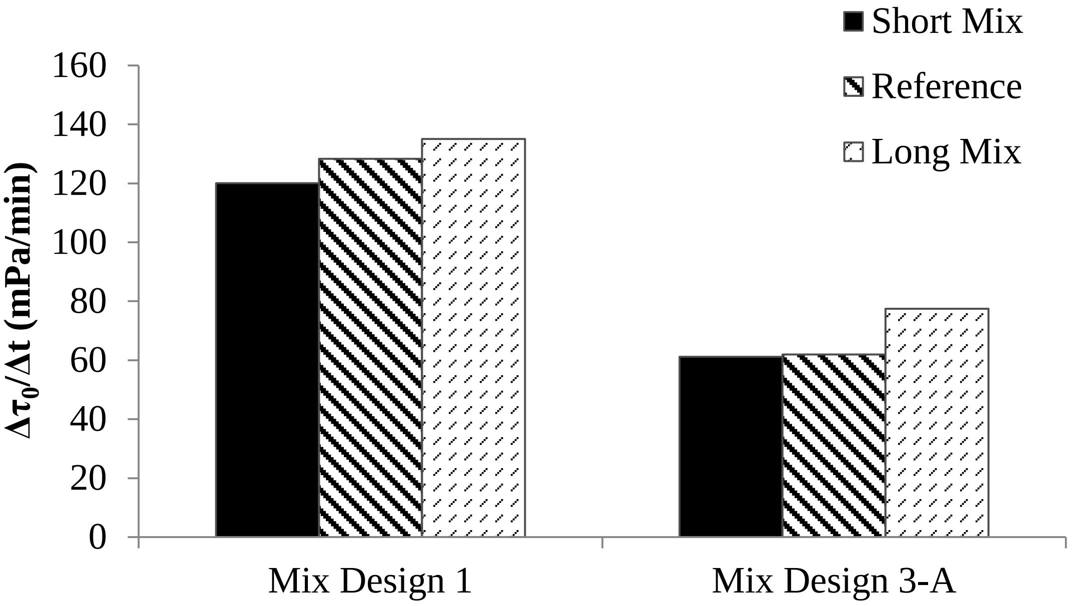

Figure 64. The rate of change of yield stress with time, as a function of the mixing time (short, medium (ref) or long).

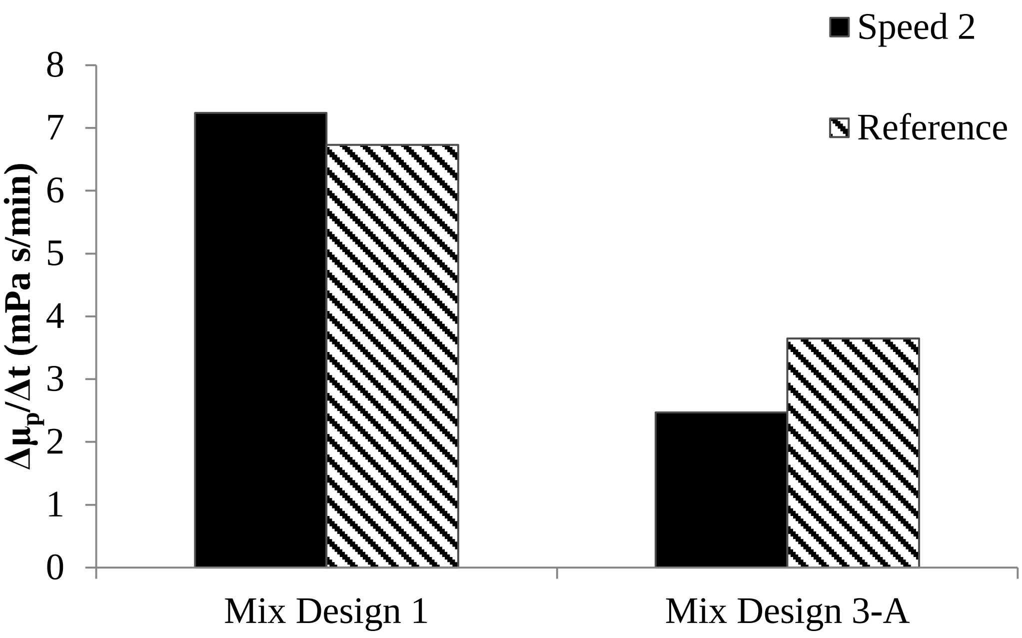

Figure 65. The rate of change of plastic viscosity with time, as a function of the mixing speed (reference: speed 1, or high: speed 2).

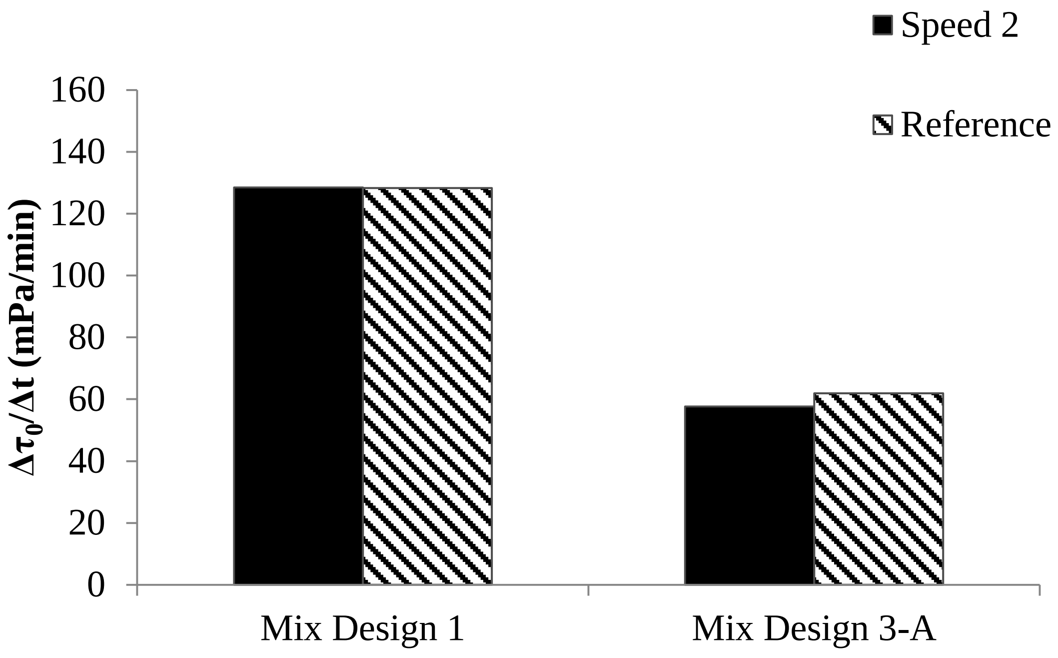

Figure 66. The rate of change of yield stress with time, as a function of the mixing speed (reference: speed 1, or high: speed 2).

8.1.6 *Combined Influence of Amount of Added Water and Addition Time of the SP*

It was shown in sections 5 and 6 that the amount of water and the addition time of the SP are two major factors influencing the robustness of cement-paste mixtures. The influence of both factors is combined and the influence of different constituent elements on the workability loss variations is also determined.

<u>Combined effect of amount of water and addition time of SP 1</u> – Figures 67 and 68 display, respectively, the rate of change of plastic viscosity and yield stress for the combined effect of a change in water content (corresponding to ± 10 l/m^3 in concrete) and the adding time of the SP. It can be seen that both parameters have a significant influence on the rate of change of the rheological parameters with time. Increasing the water content slows down the increase in plastic viscosity and yield stress significantly, regardless of the adding time of the SP. However, the delayed addition of the SP increases the rate of change of yield stress and plastic viscosity, regardless of the water content. The 50/50 addition (50% with the water, 50% delayed) delivers more complex results but does not generally provide specific advantages in workability loss. It is also worthy to mention that the initial properties (at 15 min) were lower with a delayed addition of SP, but the values increase faster when the SP addition is delayed.

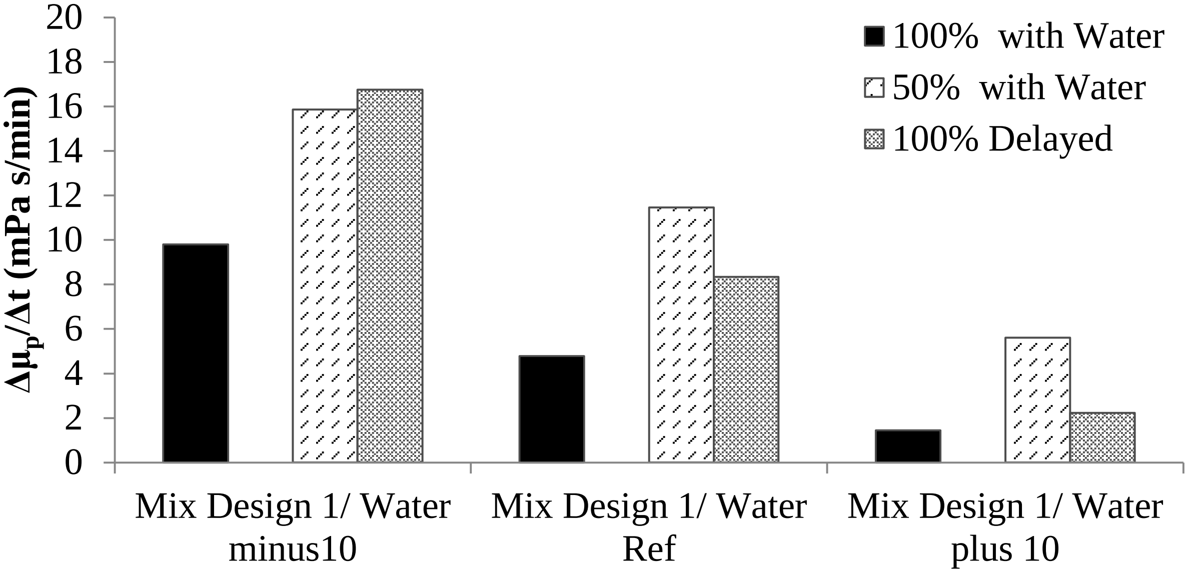

Figure 67. The rate of change of plastic viscosity with time, as a combined function of a change in water content (Water minus 10, Ref, water +10) and a change in adding time of SP.

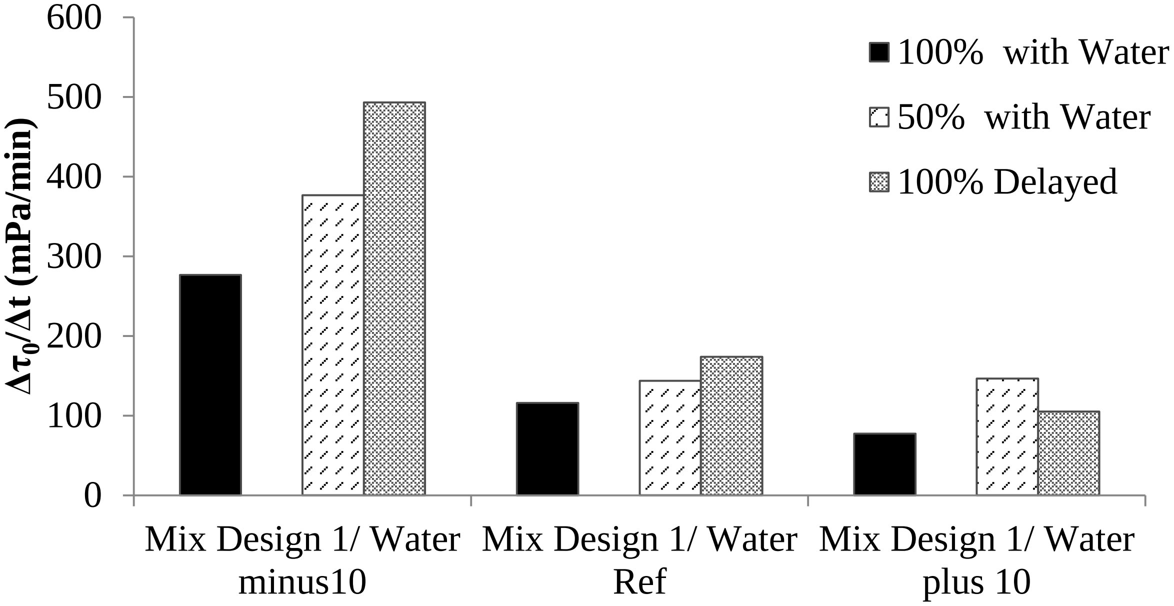

Figure 68. The rate of change of yield stress with time, as a combined function of a change in water content (Water minus 10, Ref, water +10) and a change in adding time of SP.

<u>Combined effect of amount of water and addition time of SP 2</u> – Figure 69 and 70 show similar results as Figures 67 and 68, but the magnitude of the values is significantly lower due to the specific workability retention capacity of SP 2. The influence of adding time of SP and a variation in water content is similar to SP 1 for the yield stress. Delaying the addition of the SP causes the yield stress to increase faster with time. In fact, an increase in yield stress with time is noticed with the delayed addition, while a decrease in time is observed for the addition of SP with water. For the viscosity, however, an opposite effect is noticed for SP 2, compared to SP 1. It appears, at least for the mixtures with increased and decreased water contents, that the delayed addition of SP slows down the increase of viscosity with time. The influence of a change in water content is similar for SP 1. In contrast to SP 1, for which a delayed addition of the SP made the workability loss less robust to a variation in water content, the opposite can be noticed for SP 2, at least for the yield stress. The rate of change of the yield stress is to a lesser extent influenced by a change of water content when the SP addition is delayed. Also similar to SP 1, the 50/50 case reveals complex results and no significant improvement of the robustness. The 50/50 results are no longer included in the following sections.

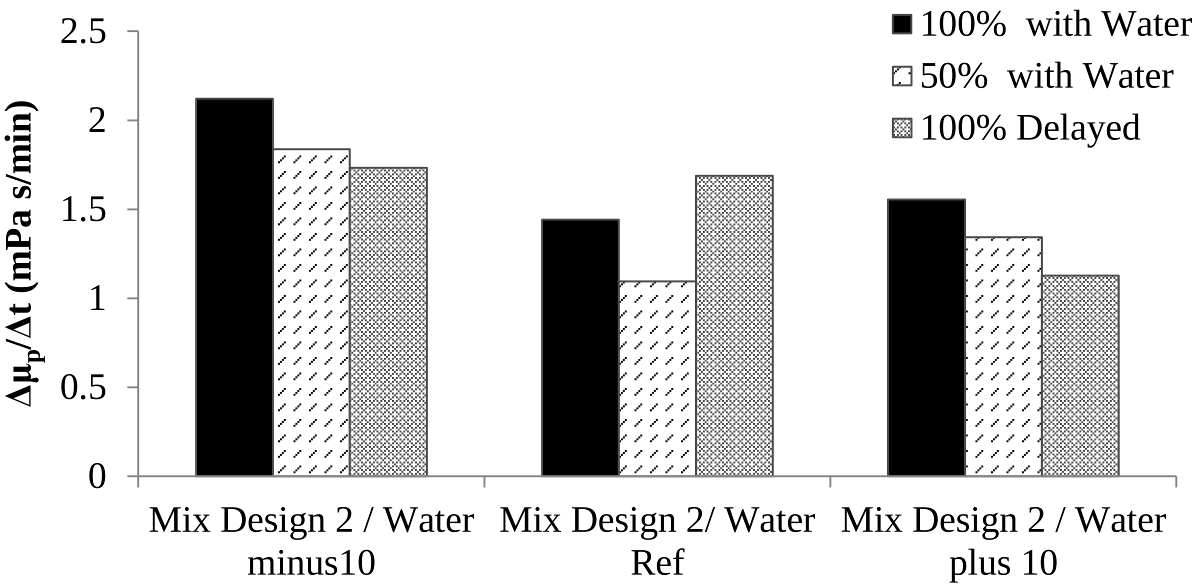

Figure 69. The rate of change of plastic viscosity with time, as a combined function of a change in water content (Water minus 10, Ref, water +10) and a change in adding time of SP.

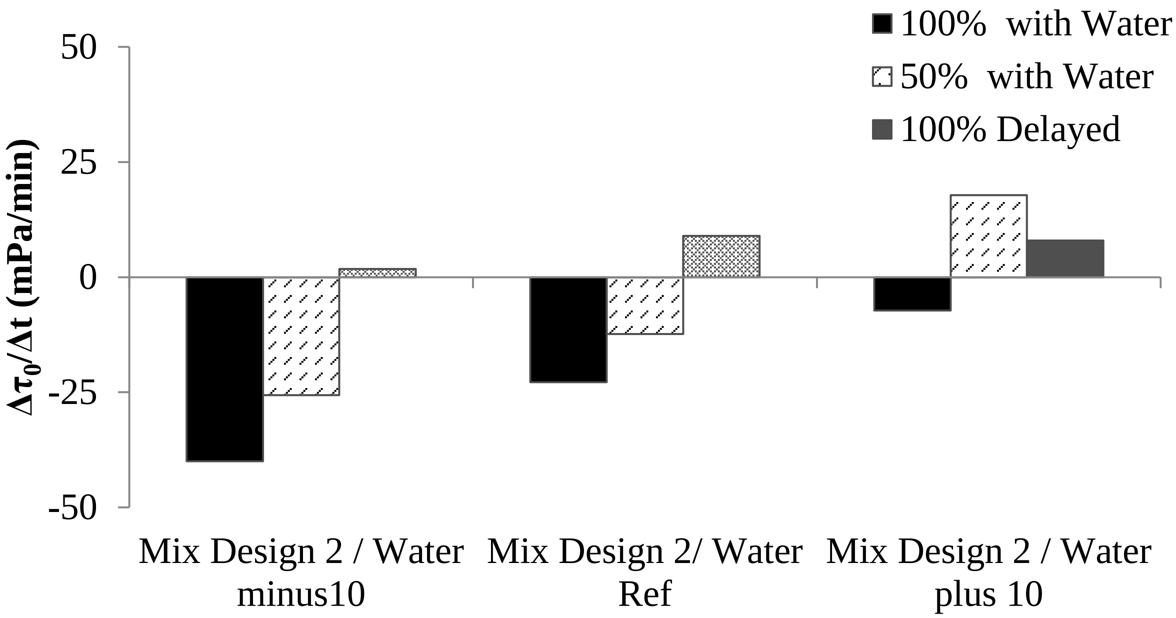

Figure 70. The rate of change of yield stress with time, as a combined function of a change in water content (Water minus 10, Ref, water +10) and a change in adding time of SP.

<u>Presence of VMA</u> – Figures 71 and 72 show the rate of change of plastic viscosity and yield stress with time, respectively, for mixture 2 with and without VMA and different adding times of the SP. The presence of VMA only slightly affects the rate of change of the plastic viscosity, but the yield stress decreases faster when no VMA is added to the mixture, regardless of the adding time of SP. The presence of VMA does not seem to affect the influence of the adding time of the SP, except for the slight variation noticed for the plastic viscosity. It should also be noted, when comparing the results on the left of Figures 71 and 72 to the center of Figures 69 and 70 that the

results are different. Especially a different trend for the change of yield stress with time can be noticed. The difference between the results in Figures 69 and 70, and the results from 71 and 72 is a change in delivery of cement from the same manufacturer. The influence of water content, admixture type and addition sequence on the workability loss is thus a complex interaction between the admixture composition and its interaction with the cement. But the admixture type and intended workability retention is the dominant parameter on the workability loss.

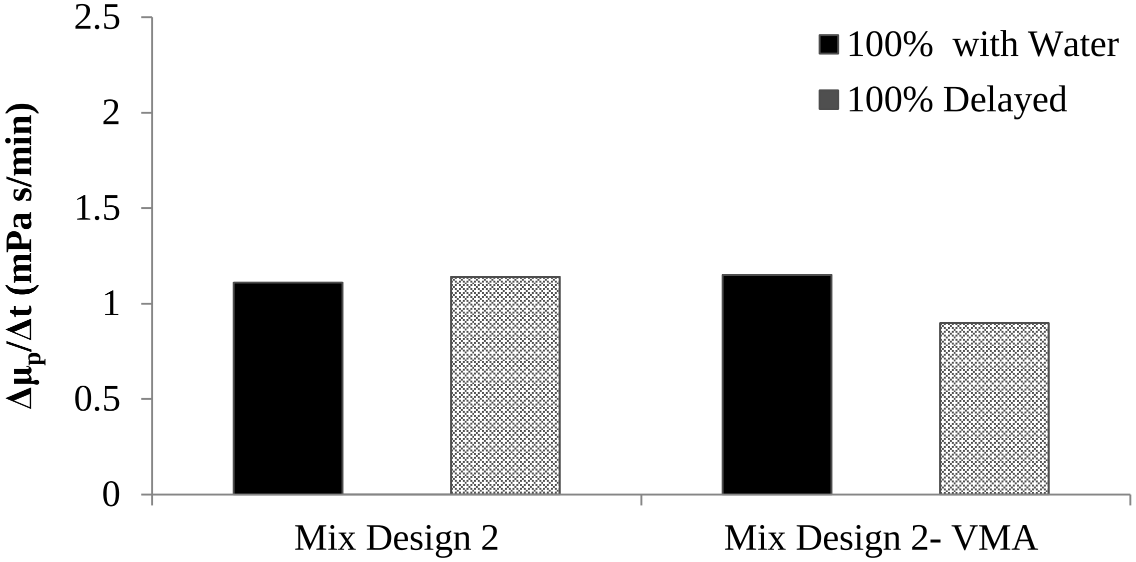

Figure 71. The rate of change of plastic viscosity with time, as a function of a change in adding time of SP for reference mixture 2 with (left) and without VMA (right).

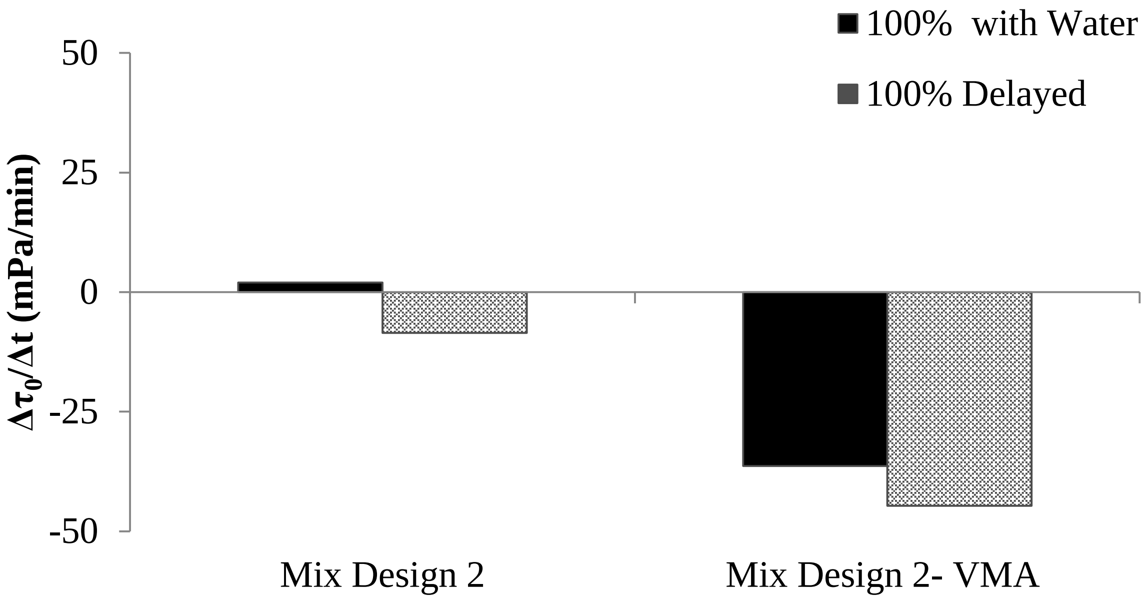

Figure 72. The rate of change of yield stress with time, as a function of a change in adding time of SP for reference mixture 2 with (left) and without VMA (right).

8.2 Thixotropy and Loss of Workability of Concrete

8.2.1 *Influence of Variations in Water Content*

The evolution of slump flow, V-funnel time, rheological properties and static yield stress with time is summarized in Table 19 and in Figures 73 to 75. The V-funnel time at 75 minutes was not measured for the mixture with 10 l/m³ less water because of its poor workability (a slump flow of only 465 mm). For all mixtures, plastic viscosity increased with time, and the increase is more pronounced when the water content is lower. The dynamic yield stress (and the slump flow) appears to remain approximately constant over time, which can be attributed to the working mechanism of the SP. Even a slight "maximum" in the slump flow is observed at 45 mins. The static yield stress shows a clear increase with time, indicating the thixotropic behavior of the concrete. Thixotropy is largely affected by the water variations, as is clearly shown by the rate of increase of static yield stress with time in Figure 75.

Table 19. Influence of variations in the water content on the workability retention.

	- 10 l/m³ water	Reference	+ 10 l/m³ water
Slump flow (mm)			
20 min	465	595	700
45 min	470	650	760
75 min	405	605	730
V-funnel time (s)			
20 min	12.5	5.1	4.5
45 min	17.3	7.7	5.0
75 min	-	7.1	4.6
Dynamic yield stress (Pa)			
10 min	85	36	20
30 min	113	29	17
60 min	90	24	14
90 min	-	37	17
Plastic viscosity (Pa s)			
10 min	52	31	17
30 min	55	37	19
60 min	81	48	24
90 min	-	67	33
Static Yield Stress			
A_{thix} (Pa/s)	2.50	1.18	0.46

8.2.2 *Influence of Variations in Mixing Time*

For the mixing time intervals studied, small changes in the mixing time do not affect the time evolution: the V-funnel time and plastic viscosity increase continuously during the first 90 minutes, while the slump flow and dynamic yield stress reach an optimum during that period. As the mixing time increases, the superplasticizer is better dispersed and a mixture with a lower plastic viscosity but a similar dynamic yield stress is created. The static yield stress evolution is also not affected by the mixing time. More experiments are needed to determine the mechanisms of this influence.

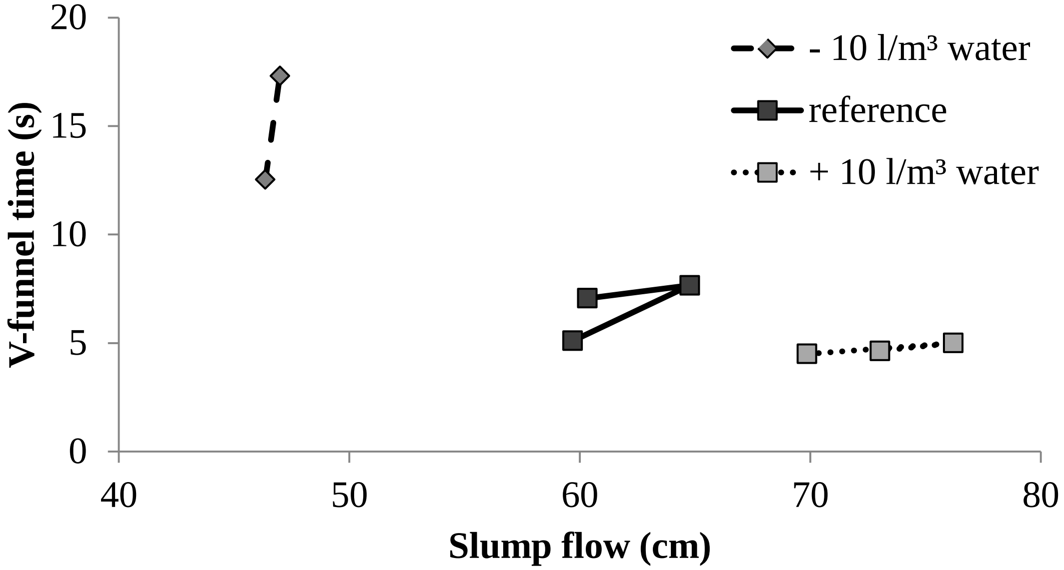

Figure 73. The influence of a variation in the water content on the slump flow and V-funnel evolution.

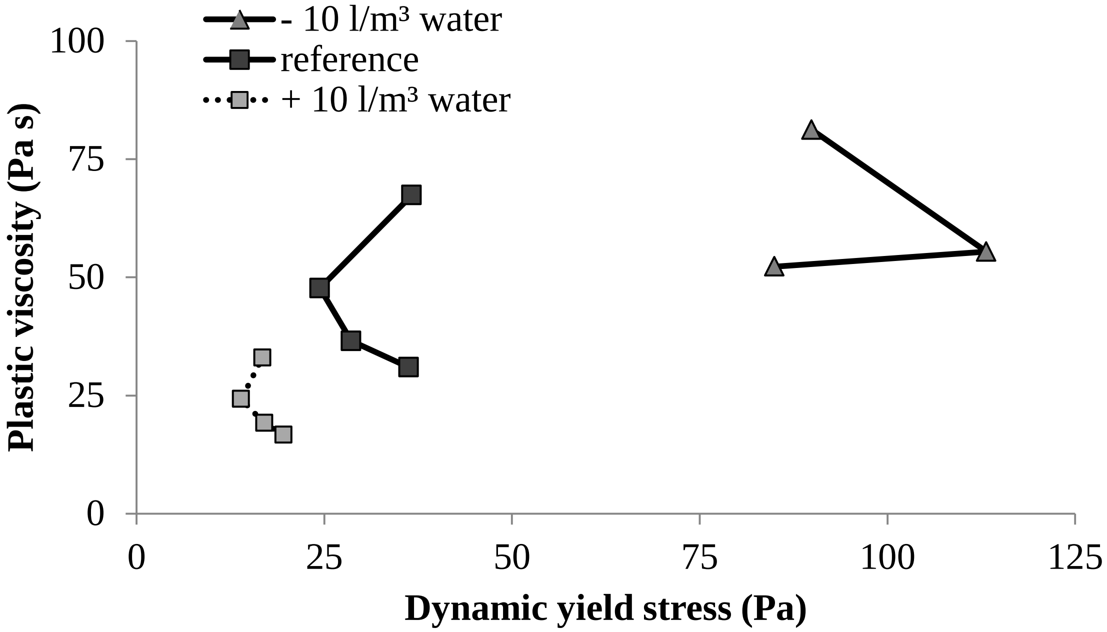

Figure 74. The influence of a variation in the water content on the Bingham parameters evolution.

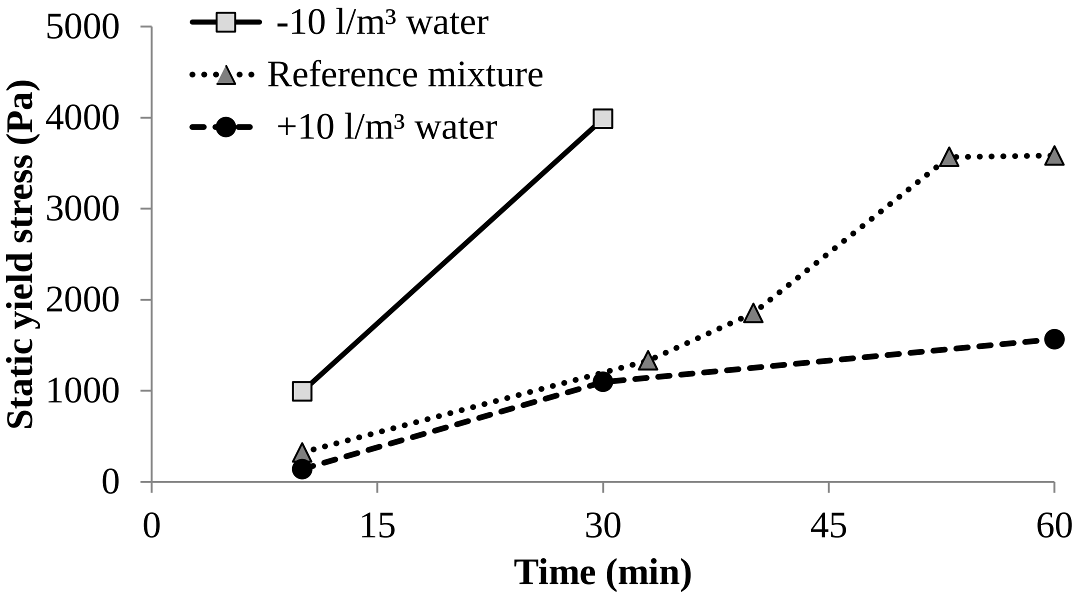

Figure 75. The influence of a variation in the water content on the static yield stress evolution.

Table 20. The influence of the mixing time on the workability retention.

	Short mixing time (2.5 min)	Intermediate mixing time (3.5 min)	Long mixing time (4.5 min)
Slump flow (mm)			
20 min	760	705	730
45 min	770	745	760
75 min	745	725	735
V-funnel time (s)			
20 min	4.0	3.9	3.6
45 min	4.5	4.3	4.0
75 min	5.3	4.7	5.4
Dynamic yield stress (Pa)			
10 min	17	16	16
30 min	8	14	14
60 min	8	12	12
90 min	19	15	15
Plastic viscosity (Pa s)			
10 min	38	27	22
30 min	44	31	26
60 min	51	37	32
90 min	64	51	44
Static Yield Stress			
A_{thix} (Pa/s)	0.72	0.87	0.86

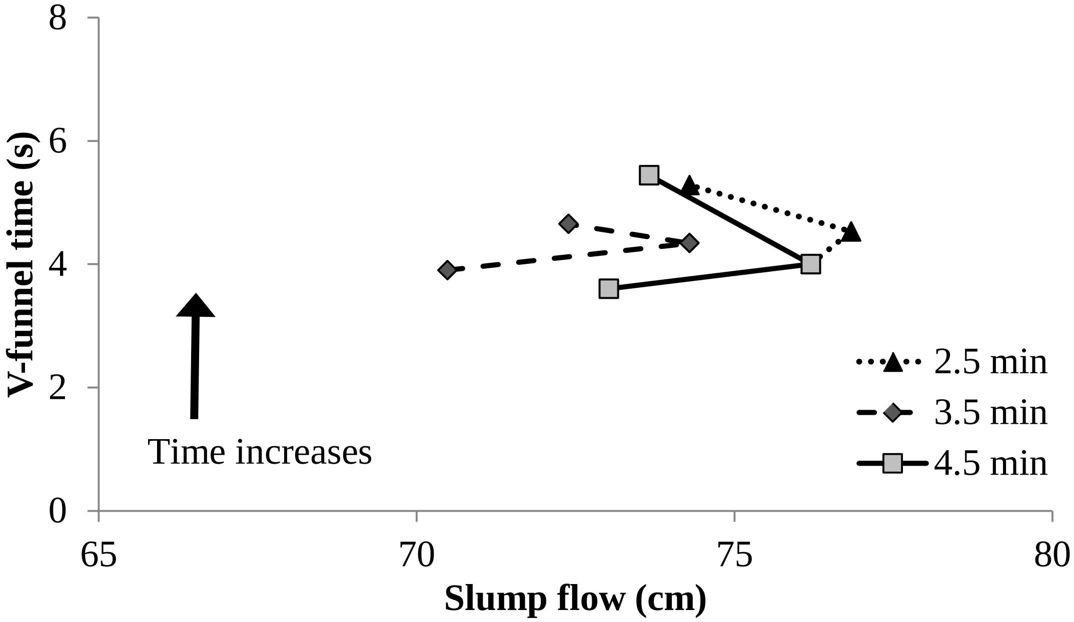

Figure 76. The influence of the mixing time on the slump flow and V-funnel time evolution.

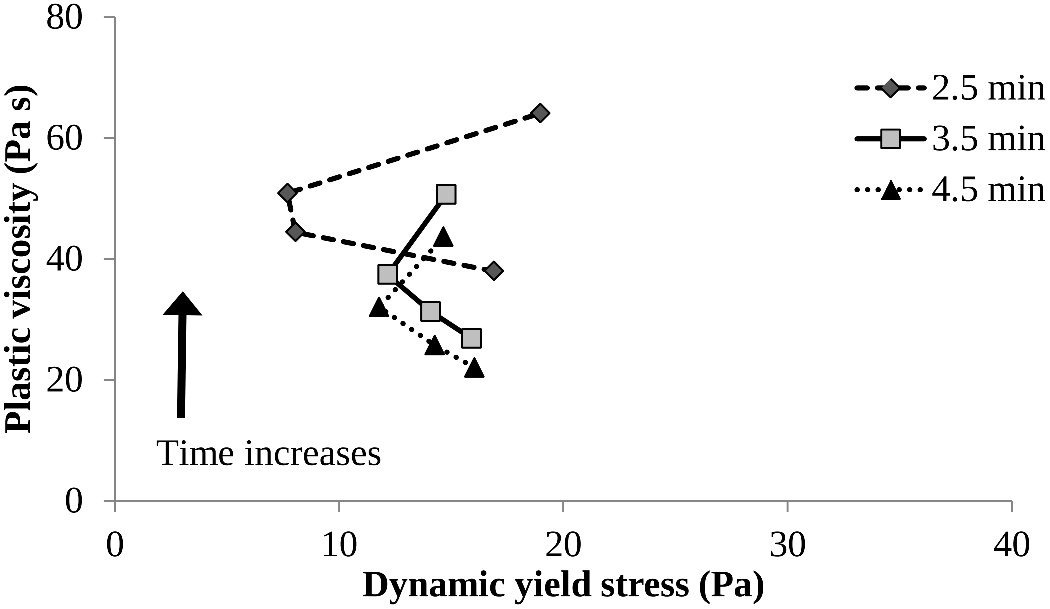

Figure 77. The influence of the mixing time on the Bingham parameters evolution.

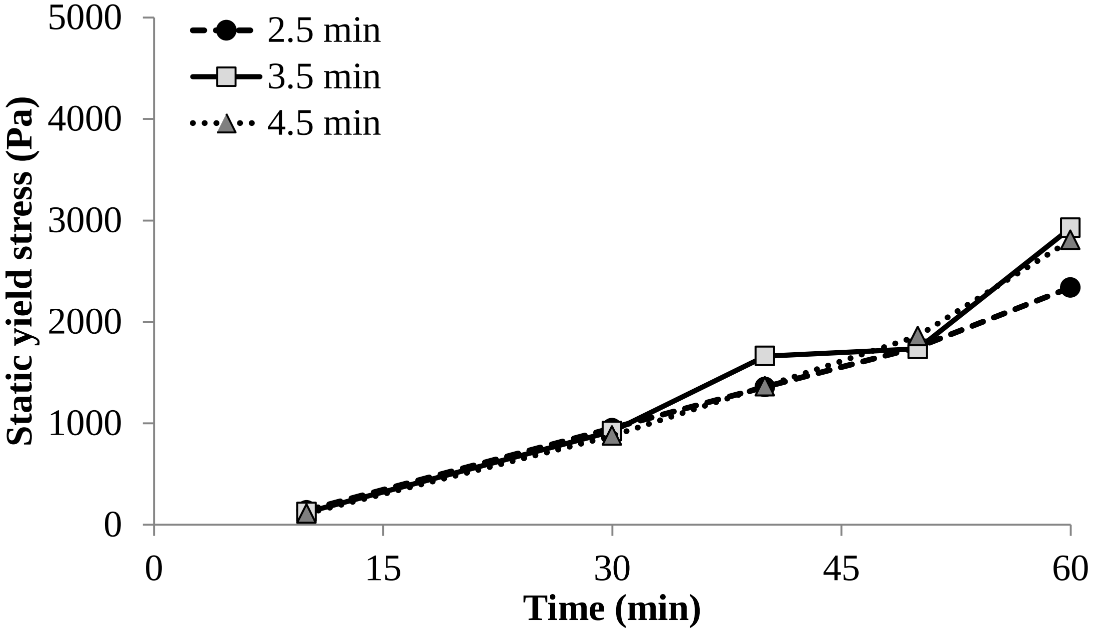

Figure 78. The influence of the mixing time on the static yield stress evolution.

8.2.3 Influence of the Addition Sequence of the Aggregates

As can be seen in Table 21 and Figures 79 and 80, no significant variations in workability loss are observed. The evolution of slump flow, V-Funnel, dynamic yield stress and plastic viscosity all follow a similar trend with similar orders of magnitude, regardless of the addition sequence of the materials. The static yield stress shows different results, however (Figure 81). Premixing the aggregates with half of the mixing water delivers the largest increase of static yield stress with time, while the mixtures for which the aggregates were added in the cement paste show the lowest increase in static yield stress with time. Similar to the discussion on the results of yield stress and plastic viscosity measured at 10 min, the difference in thixotropic behavior can be explained by a change of water available in the cement paste, leading to an increase in thixotropic behavior when aggregates absorb more water. This is in agreement with the findings on the variations in the water content, as a decrease in water content increased thixotropy.

Table 21. The influence of the addition sequence on the workability retention.

	A C W SP	A $\frac{W}{2}$ C $\frac{W}{2}$ SP	C W SP A	C W A SP
Slump flow (mm)				
20 min	705	680	775	760
45 min	725	725	775	780
75 min	725	700	735	745
V-funnel time (s)				
20 min	3.9	4.6	3.8	3.8
45 min	4.3	4.6	4.1	4.2
75 min	4.7	5.6	5.0	5.0
Dynamic yield stress (Pa)				
10 min	16	27	9	13
30 min	14	24	8	13
60 min	12	19	7	11
90 min	15	30	6	11
Plastic viscosity (Pa s)				
10 min	27	32	29	26
30 min	31	37	35	29
60 min	37	47	41	35
90 min	51	64	50	47
Static Yield Stress				
A_{thix} (Pa/s)	0.87	0.87	0.40	0.67

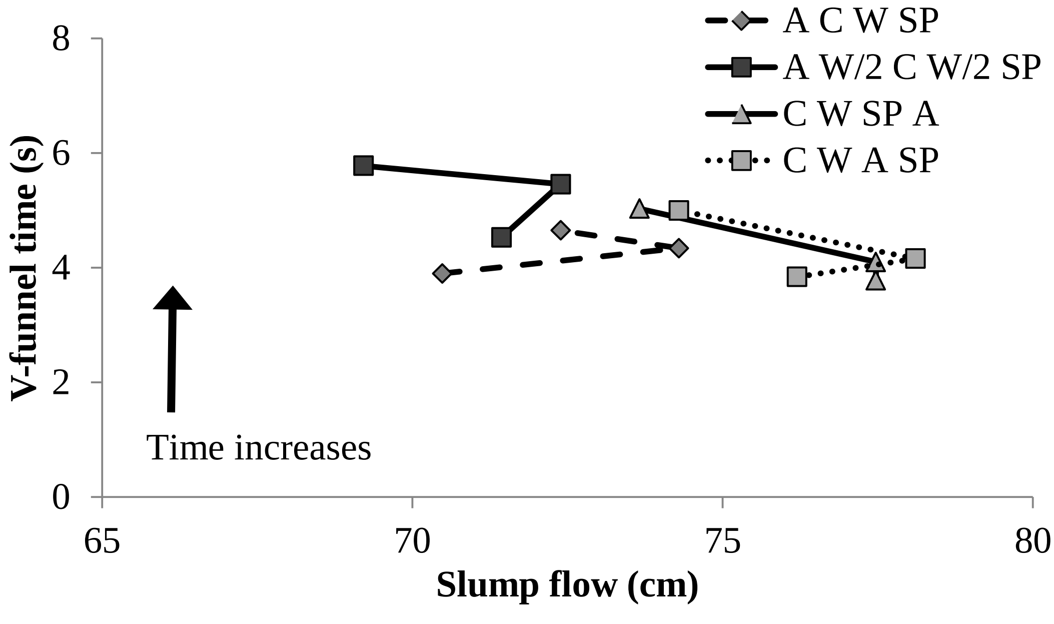

Figure 79. Influence of the mixing sequence on the slump flow and V-funnel time evolution.

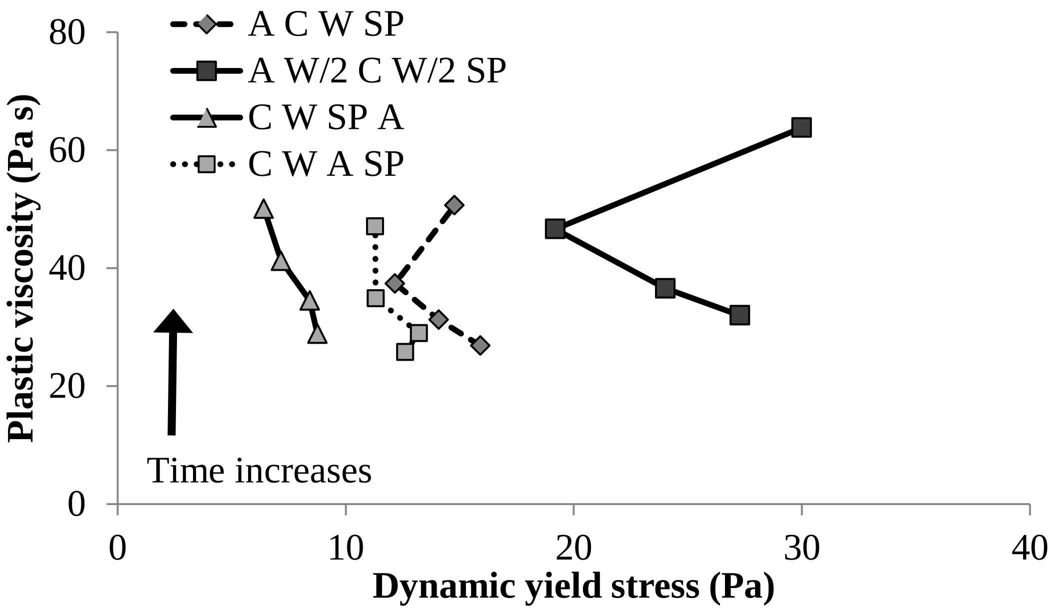

Figure 80. Influence of the mixing sequence on the Bingham parameters evolution.

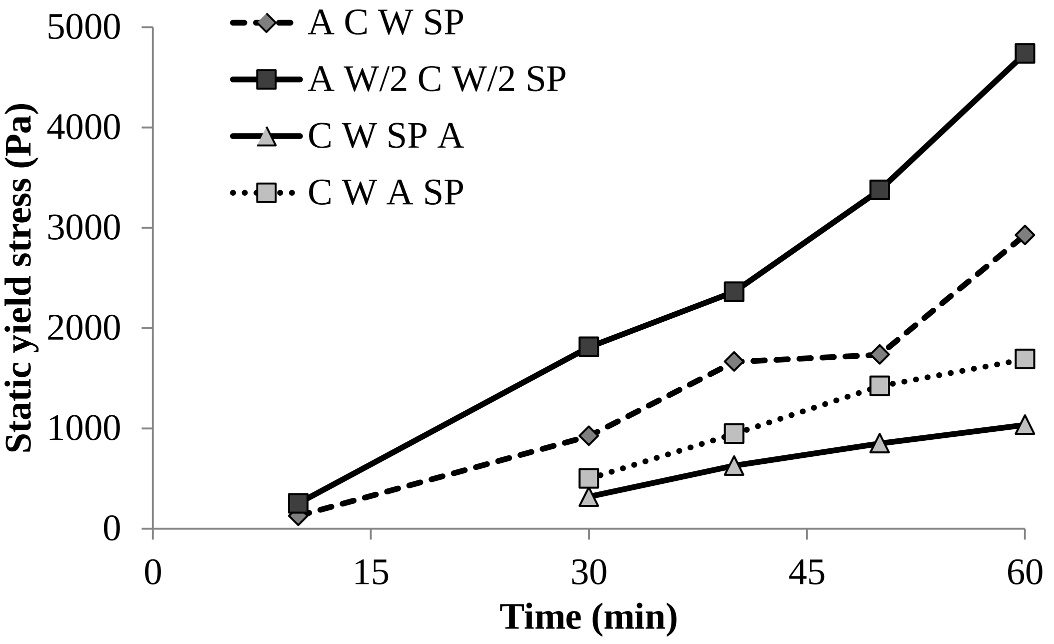

Figure 81. Influence of the mixing sequence on the static yield stress evolution.

8.2.4 Influence of the Initial Moisture Content of the Aggregates

No differences in the V-funnel time and plastic viscosity evolution can be observed whether air-dry or soaked sand is used, keeping the total water content of the mixture constant (Table 22, Figure 82, and Figure 83). The slump flow and dynamic yield stress evolution of both mixtures has the same shape, but shifts to a less fluid state when soaked aggregates were used (the dynamic yield stress was about 5 Pa higher at each measuring time, which was discussed in section 7.2.5). The plot of static yield stress as a function of the time is slightly steeper when air-dry sand is used.

Table 22. The influence of the aggregates moisture content on the workability retention.

	Air-dry aggregates	Soaked aggregates
Slump flow (mm)		
20 min	760	690
45 min	760	715
75 min	740	700
V-funnel time (s)		
20 min	5.1	4.5
45 min	5.3	4.9
75 min	6.2	6.2
Dynamic yield stress (Pa(		
10 min	15	19
30 min	9	14
60 min	6	10
90 min	8	14
Plastic viscosity (Pa s)		
10 min	37	34
30 min	42	40
60 min	51	48
90 min	65	65
Static Yield Stress		
A_{thix} (Pa/s)	0.77	0.51

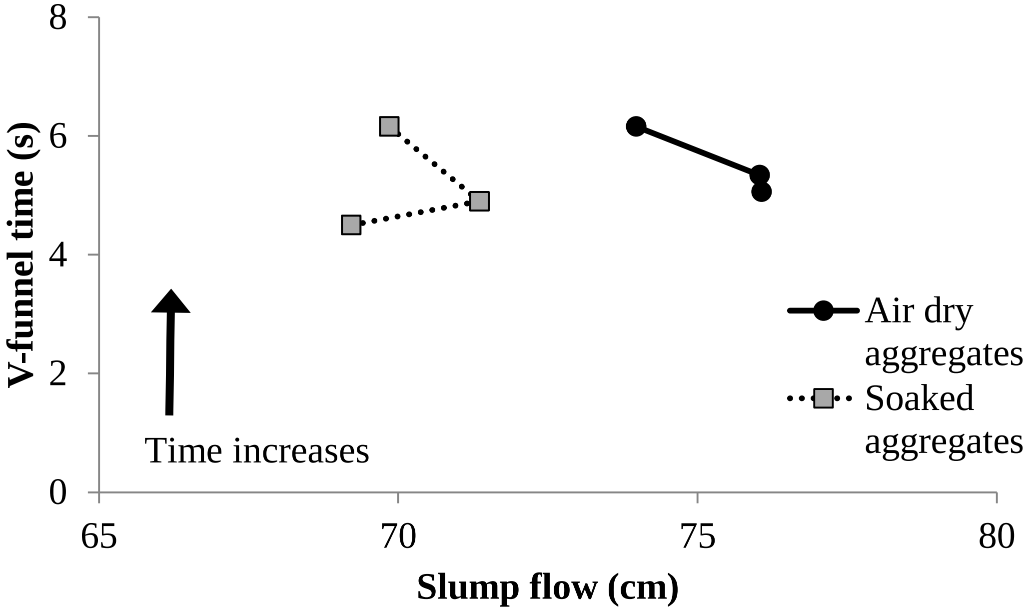

Figure 82. Influence of the aggregates moisture content on the slump flow and V-funnel time evolution.

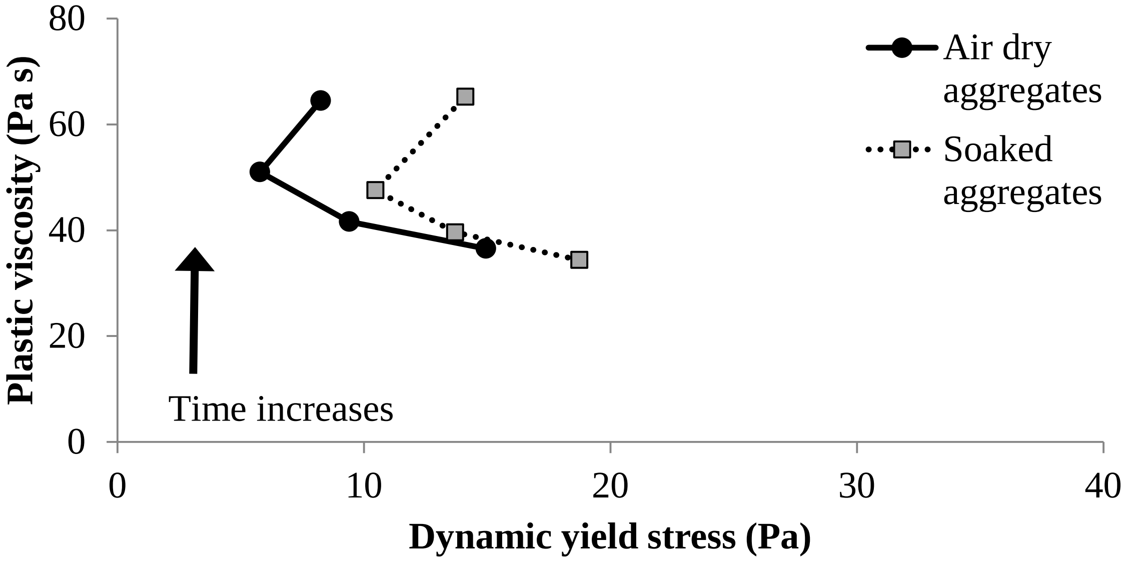

Figure 83. Influence of the aggregates moisture content on the Bingham parameters evolution.

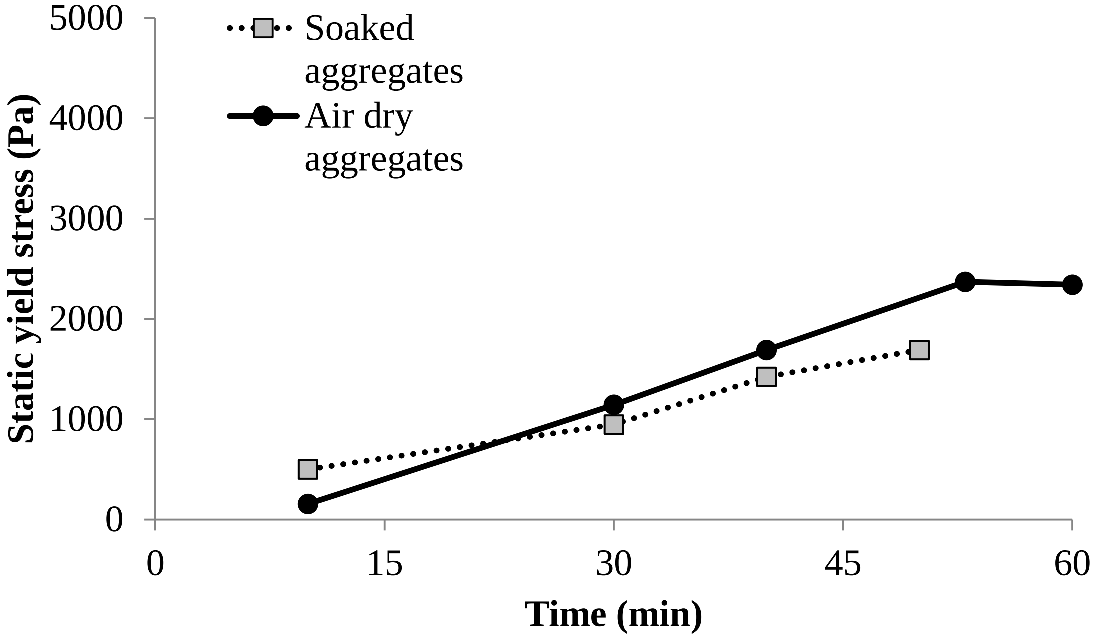

Figure 84. Influence of the aggregates moisture content on the static yield stress evolution.

8.3 Summary

The workability loss was monitored by the change of yield stress and plastic viscosity over a period of 60 min for the cement pastes and 90 min for the concrete mixtures. The main parameter influencing loss of workability is the type of SP employed. Specifically, SP 2 is designed to maintain the workability for an extended time period, which is reflected by a significantly slower increase in plastic viscosity and even by occasion a decrease in yield stress for cement pastes. The variation in water content and the addition time of SP were the two most significant factors, after the type of SP, influencing workability loss. Decreasing the water content caused yield stress and plastic viscosity to increase faster with time, regardless of the addition time of the SP. The influence of the water content on the workability loss is further confirmed on concrete scale. The influence of the adding time of the SP depends, however, on the type of SP used. For SP 1, a delayed addition caused a significantly faster workability loss than when the SP was added with the mixing water. While for SP 2, similar results were observed for the increase in yield stress, but to a lesser extent, but the results on the increase in viscosity indicate the opposite behavior. Furthermore, the final behavior depends also on the delivery of the cement, so it is expected that the final behavior depends on the water content, type of SP, addition time of the SP and the interaction between the SP and the cement. Compared to the influence of the parameters described above, the amount of SP added, the mixing time and mixing speed (to a certain extent), only have a minor or even no influence on the workability loss. The results on the addition sequence of the air-dry aggregates (below SSD conditions) can be related to the amount of water in the mixture. The more water absorbed by the aggregates before the cementitious materials are added, the larger the workability loss.

The influence on thixotropy of the amount of water, mixing time and addition sequence of the air-dry aggregates was also determined on concrete mixtures. Decreasing the water content significantly increased thixotropic build-up, and vice-versa. The mixing time did not significantly affect thixotropy, while the addition sequence of the aggregates can be related to a change in the amount of mixing water.

9 Conclusions

9.1 General Conclusions of this Research Project

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) in transportation infrastructure. Typically, small variations in the water content, through inaccuracies of the balances in the concrete production plant or small mistakes in the determination of the aggregate moisture content, is the most investigated parameter in robustness studies.

In this project, the influence of the mixing procedure and addition sequence of constituent elements on the robustness of SCC was investigated on cement-pastes with SCC consistency and on concrete mixtures, by means of rheology. The mixtures' rheological properties: yield stress and plastic viscosity were determined using the Anton Paar MCR 302 (cement pastes) and the ConTec Viscometer 5 (concretes). Two reference SCC mix design strategies were employed: the powder-type mix design and the VMA-type mix design.

For cement pastes, it was concluded that variations in water, variations in the adding time of the superplasticizer: SP (with the mixing water or delayed), variations in mixing energy (especially mixing speed) and different deliveries of cements all have significant influence on the measured plastic viscosity and yield stress. Type I or Type I/II cements from different manufacturers and different cement deliveries from the same manufacturer can significantly alter the fresh properties of the cement pastes, all other variables remaining constant. For the VMA-type cement paste investigated, the influence of the adding time of the SP on the plastic viscosity appeared as large as the influence induced by varying the water content and the robustness of this mixture to variations in water content can be improved by delaying the addition of the SP to the paste. The two main constituent elements which alter the sensitivity of the mixture to a change in adding time of the SP are the cement properties (as different cement deliveries yielded different results) and the presence of limestone filler (improving the robustness to a variation in adding time of the SP).

On concrete scale, the sensitivity of yield stress (and slump flow) and plastic viscosity (and V-Funnel flow time) to variations in water content and mixing speed were confirmed. The mixing time appeared to have a minor influence on the rheological properties of the SCC, which is in line with the cement paste results. The addition sequence of aggregates, water, cement and filler and SP appeared to have an important influence, but to a lesser extent than the variations in water content and mixing speed. When the aggregates are air-dry, meaning their moisture content is below saturated surface-dry conditions, it is anticipated that the aggregates will absorb mixing water until full saturation is reached. However, if the aggregates do not have sufficient time to reach full saturation, more mixing water is available in the cement paste, leading to more fluid concrete mixtures. Furthermore, it is not certain how long it takes for the aggregates to reach full saturation.

Workability loss, which is defined as the change in rheological properties with time due to chemical reactions, is especially influenced by the type of SP (long workability retention vs. short workability retention), variations in the water content, the adding time of the SP and differences in cement deliveries. Decreasing the water content causes yield stress and plastic viscosity to increase faster with time. However, the response to a change in the adding time of the SP (with the mixing water or delayed) depends on the SP employed. It appears that the yield stress increases faster with time, but the results on the viscosity are different for the two different SPs used. The other parameters: amount of SP added and mixing energy, appeared only to have minor influences on the workability loss. These results were confirmed on concrete scale for the variations in water content. The consequences of changes in addition sequence of the materials, knowing that the sand was initially air-dry, can be related to variations in available water in the cement pastes.

Thixotropy, which is the reversible decrease in stress under shear and build-up at rest due to physical effects, was only investigated on concrete scale. The increase in static yield stress with time, measured with the ICAR rheometer is clearly influenced by the amount of water. Less water causes a faster thixotropic build-up. Similar to the yield stress and viscosity after mixing and to the workability loss, the variation in adding sequence of the materials follows the observed behavior for variations in the water content. Mixing time, for the variations induced in this project, appears not to influence thixotropy.

9.2 Recommendations for Self-Consolidating Concrete Producers

To enhance the application of SCC for the construction and repair of transportation infrastructure, two key concepts are of importance: quality control and consistency. The consistency refers to the mixing operations and transportation of SCC. It is recommended to keep the mixing procedure constant for every SCC produced. This includes the addition sequence of the materials, the mixing time, the mixing speed and the concrete volume (parameter not tested, but it is reflected in mixing energy). Although variations in water content are predominant in the robustness, keeping all other factors under control will reduce the final variations in fresh SCC properties. Operating with a well-equipped batching plant will facilitate the production of robust SCC compared to truck-mixing.

The quality control is not only necessary to determine the moisture content of the aggregates, but also for any of the other constituent elements used. Especially the cement-admixture interaction has a significant importance on the robustness. Monitoring the physical and chemical properties of the cement and having a supplier delivering cement with low variations provides a significant advantage in producing SCC. Performing workability tests on SCC pastes or mortars, using the mini-slump cone and the marsh-cone or mini V-Funnel test can help detecting variations in the cement or admixture deliveries and provide an indication to adjust the SP dosage in case of a change in one of the constituent materials. These simple workability tests, which replace the more complicated rheometer test, can also be used to monitor robustness of the cement paste or mortar mixtures to variations in water content and adding time of the SP. For the two SPs tested, the delayed adding time delivered in the best case a better robustness to variations in water content and in the worst case no change was noticed. However, these findings may change when a different SP or cement type (or cementitious materials) are used.

Extended transport of SCC complicates matters significantly, as the workability loss is also affected by water variations, adding time of SP and cement-SP interactions. Essential for SCC being placed on-site, is the choice of the most adequate admixture to limit the variations in time. Several chemical admixtures with extended workability or “slump”-retention are available on the market. But extended mixing during transportation can cause a significant loss of workability, not mixing however can enhance segregation (due to vibrations) or stiffening due to thixotropy. The energy induced by remixing the SCC in the truck is hard to monitor (precise time, rotations per minute, etc.) and can significantly influence the final concrete properties. Re-tempering of the SCC with SP is thus a matter of quality control and experience, as the research is not sufficiently advanced to predict the amount of SP needed.

Despite the stricter control on the constituent materials and SCC production, the successes of SCC should be kept in mind. More extended quality control and consistency are the extra efforts required to make the use of SCC successful. However, savings are made on labor use, energy, formwork repair (no more vibration), construction time, worker absenteeism (due to improved working conditions), etc.

9.3 Future Work

When studying the robustness of cement-based materials, the cement is an essential ingredient. Due to changes in deliveries of cement (from the same manufacturer or from different producers), substantial variations in the response of rheological properties were noticed, even if the cement is produced within the limits of the specifications. Changes in the physical and chemical properties of the cement influence the interaction between cement and admixtures, resulting in different fresh properties. To avoid a full physical and chemical characterization for each cement delivery at a concrete production unit, research should focus on determining specific key properties of the cement which dominate the behavior in the fresh state. These key properties could be physical (e.g. grain size distribution) or chemical (e.g. chemical composition) of nature. The research should further focus on different admixture types (e.g. SP, VMA, AEA) and subgroups (PCE, PNS, etc.). The obtained results can be expanded to

SCMs and mineral fillers. In this way, simple quality control guidelines can be developed to estimate the change in fresh properties of SCC, enhancing the application of this novel concrete type and reducing time and cost for quality control. A similar study can be performed on the variations between different deliveries of chemical admixtures.

One of the main findings on concrete in this project was the influence of the addition sequence of aggregates, cement and filler, water and SP. Especially the absorption of water by the aggregates appeared to play a significant role, as the aggregates were air-dry. When aggregates, with moisture contents below SSD condition, are given sufficient time to reach full saturation, the resulting workability of the SCC will be lower than when the aggregates are not given this time. However, the time the aggregates need to reach full saturation is unknown. Attempts undertaken in this project to determine the rate of absorption, meaning how fast dry aggregates reach full saturation, were unsuccessful. However, it is probable that, dependent on the aggregate properties, the premixing time of the aggregates and the mixing water is insufficient to reach full saturation. The correction of the moisture content is thus inadequate in this case, as too much water is present in the cement paste, potentially reducing hardened concrete quality.

It has been shown in this project that robustness does not only consider variations in water content. The addition sequence of the materials and mixing procedure can also significantly influence the robustness of the mixture. In this project, the study was limited to two different PCE-SP, showing different results dependent on the SP employed. The research should thus be extended to other SP types, from the same (PCE) and other subgroups, as well as to investigate the response to variations in use of VMA (the adding sequence of VMA was not investigated) and AEA.

Initial steps have been taken to investigate the robustness of the thixotropic response of SCC mixtures, which is important for formwork pressure and to determine critical delay times beyond which subsequent casting layers do not intermix. Also the response of workability loss to variations in water, adding sequence and mixing procedure were monitored. Further research for these properties is needed, as they are of particular interest in the ready-mix industry.

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