



SINTEF Building and Infrastructure

Hedda Vikan and Klaartje De Weerd

Stability of SCC – chemical stabilisers versus filler

COIN Project report 25 - 2010



SINTEF Building and Infrastructure

Hedda Vikan and Klaartje De Weerd

Stability of SCC – chemical stabilisers versus filler

FA 2.1 Robust highly flowable concrete

SP 2.1.4 Workability

COIN Project report 25 – 2010

COIN Project report no 25

Hedda Vikan and Klaartje De Weerd

Stability of SCC – chemical stabilisers versus filler

FA 2.1 Robust highly flowable concrete

SP 2.1.4 Workability

Keywords:

SCC, stability

Project no.: 3D005940

Photo, cover: «Sohlbergplassen»

ISSN 1891–1978 (online)

ISBN 978-82-536-1185-3 (pdf)

ISBN 978-82-536-1186-0 (printed)

20 copies printed by AIT AS e-dit

Content: 100 g Scandia

Cover: 240 g Trucard

© Copyright SINTEF Building and Infrastructure 2010

The material in this publication is covered by the provisions of the Norwegian Copyright Act. Without any special agreement with SINTEF Building and Infrastructure, any copying and making available of the material is only allowed to the extent that this is permitted by law or allowed through an agreement with Kopinor, the Reproduction Rights Organisation for Norway. Any use contrary to legislation or an agreement may lead to a liability for damages and confiscation, and may be punished by fines or imprisonment.

Address: Forskningsveien 3 B
POBox 124 Blindern
N-0314 OSLO

Tel: +47 22 96 55 55

Fax: +47 22 69 94 38 and 22 96 55 08

www.sintef.no/byggforsk

www.coinweb.no

Cooperation partners / Consortium Concrete Innovation Centre (COIN)

Aker Solutions

Contact: Jan-Diederik Advocaat

Email: jan-diederik.advocaat@akersolutions.com

Tel: +47 67595050

Saint Gobain Weber

Contact: Geir Norden

Email: geir.norden@saint-gobain.com

Tel: +47 22887700

Norcem AS

Contact: Terje Rønning

Email: terje.ronning@norcem.no

Tel: +47 35572000

NTNU

Contact: Terje Kanstad

Email: terje.kanstad@ntnu.no

Tel: +47 73594700

Rescon Mapei AS

Contact: Trond Hagerud

Email: trond.hagerud@resconmapei.no

Tel: +47 69972000

SINTEF Building and Infrastructure

Contact: Tor Arne Hammer

Email: tor.hammer@sintef.no

Tel: +47 73596856

Skanska Norge AS

Contact: Sverre Smeplass

Email: sverre.smeplass@skanska.no

Tel: +47 40013660

Spenncon AS

Contact: Ingrid Dahl Hovland

Email: ingrid.dahl.hovland@spenncon.no

Tel: +47 67573900

Norwegian Public Roads Administration

Contact: Kjersti K. Dunham

Email: kjersti.kvalheim.dunham@vegvesen.no

Tel: +47 22073940

Unicon AS

Contact: Stein Tosterud

Email: stto@unicon.no

Tel: +47 22309035

Veidekke Entreprenør ASA

Contact: Christine Hauck

Email: christine.hauck@veidekke.no

Tel: +47 21055000

Preface

This study has been carried out within COIN - Concrete Innovation Centre - one of presently 14 Centres for Research based Innovation (CRI), which is an initiative by the Research Council of Norway. The main objective for the CRIs is to enhance the capability of the business sector to innovate by focusing on long-term research based on forging close alliances between research-intensive enterprises and prominent research groups.

The vision of COIN is creation of more attractive concrete buildings and constructions. Attractiveness implies aesthetics, functionality, sustainability, energy efficiency, indoor climate, industrialized construction, improved work environment, and cost efficiency during the whole service life. The primary goal is to fulfil this vision by bringing the development a major leap forward by more fundamental understanding of the mechanisms in order to develop advanced materials, efficient construction techniques and new design concepts combined with more environmentally friendly material production.

The corporate partners are leading multinational companies in the cement and building industry and the aim of COIN is to increase their value creation and strengthen their research activities in Norway. Our over-all ambition is to establish COIN as the display window for concrete innovation in Europe.

About 25 researchers from SINTEF (host), the Norwegian University of Science and Technology - NTNU (research partner) and industry partners, 15 - 20 PhD-students, 5 - 10 MSc-students every year and a number of international guest researchers, work on presently eight projects in three focus areas:

- Environmentally friendly concrete
- Economically competitive construction
- Aesthetic and technical performance

COIN has presently a budget of NOK 200 mill over 8 years (from 2007), and is financed by the Research Council of Norway (approx. 40 %), industrial partners (approx 45 %) and by SINTEF Building and Infrastructure and NTNU (in all approx 15 %).

For more information, see www.coinweb.no

Tor Arne Hammer
Centre Manager

Summary

The importance of mix design is crucial in order to obtain self-consolidating concrete (SCC) that is fluid enough to completely fill the form and simultaneously stable against segregation of aggregates and bleeding. Stability of SCC can be achieved in at least two ways: By aid of fines and/or filler, and by aid of chemical stabilizer. The main objective of this study is to investigate the influence the effect fillers and chemical stabilizers have on rheological properties of concrete and matrix. The stabilizing properties of two chemical stabilizers (polymer type and cellulose derivate) and three fillers (limestone filler and two fillers sieved from sand) have been investigated. Two types of acrylate based superplasticizers were used in order to investigate effect of dispersion and possible interactions between these admixtures and the two groups of stabilizers.

The results showed that the effect of stabilizers depended on plasticizer type and dosage (i.e. dispersion). However, for both superplasticizers, filler addition gave a stronger viscosity increase than the chemical stabilizers.

Of the two chemical stabilizers, the polymer type had stronger thixotropic effect than the cellulose type. However, for both superplasticizers, addition of filler gave a stronger viscosity increase than the chemical stabilizers.

Matrix viscosity increased with increasing volume fraction of solids in line with the Krieger-Dougherty equation. Viscosity and thixotropy of matrix dispersed with SP2 increased with increasing.

The report is concluded with suggestions for further work, including full-scale experiments in order to investigate effect of stabilizing method and final surface quality of wall elements.

Table of contents

PREFACE	3
SUMMARY	4
1 ABBREVIATIONS	7
2 INTRODUCTION	8
2.1 PRINCIPAL OBJECTIVES AND SCOPE	8
2.2 BACKGROUND	8
2.2.1 <i>Importance of robustness and stability for SCC</i>	8
2.2.2 <i>Rheological description of SCC</i>	8
2.2.3 <i>The particle-matrix model</i>	10
2.2.4 <i>Material factors influencing robustness and stability</i>	11
2.2.5 <i>Influence of additional powder as stabilizer</i>	11
2.2.6 <i>Influence of chemical stabilizers</i>	11
2.2.7 <i>Influence of superplasticizers</i>	12
3 EXPERIMENTAL	13
3.1 MATERIALS	13
3.2 CONCRETE EXPERIMENTAL SET-UP	14
3.2.1 <i>Recipes</i>	14
3.2.2 <i>Mixing procedure</i>	16
3.2.3 <i>Measurements</i>	16
3.2.4 <i>Repeatability</i>	18
3.3 MATRIX EXPERIMENTAL SET-UP	18
3.3.1 <i>Recipes</i>	18
3.3.2 <i>Mixing procedure</i>	19
3.3.3 <i>Rheological measurement sequence</i>	20
3.3.4 <i>Repeatability</i>	21
4 RESULTS	22
4.1 REPEATABILITY	22
4.1.1 <i>Measured fresh concrete properties</i>	22
4.1.2 <i>Measured fresh matrix properties</i>	22
4.2 CORRELATION BETWEEN FRESH PROPERTIES MEASURED ON MATRIX AND CONCRETE	23
4.3 FRESH CONCRETE PROPERTIES	25
4.3.1 <i>Concretes plasticized with SP1</i>	25
4.3.2 <i>Concretes plasticized with SP2</i>	28
4.4 FRESH PROPERTIES OF MATRIX	29
4.5 CONCRETE COMPRESSIVE STRENGTH	33
5 CONCLUSIONS	35
5.1 VISUAL SEGREGATION INDEX AS A MEANS FOR EVALUATING STABILIZING EFFECT	35
5.2 FRESH AND RHEOLOGICAL PROPERTIES OF CONCRETE AND MATRIX	35
5.2.1 <i>Yield stress parameters</i>	35
5.2.2 <i>Viscosity parameters</i>	35
5.2.3 <i>Matrix thixotropy – Structural buildup</i>	36
5.3 CONCRETE COMPRESSIVE STRENGTH	36
6 SUGGESTIONS FOR FURTHER WORK	37
REFERENCES	38

APPENDIX 1 - GRADING CURVE AGGREGATES 116-KURVE 0/8-FRACTION.....	41
APPENDIX 2 – INFLUENCE OF AGGREGATES FROM THE 116-CURVE (0/8 MM) ON THE CONCRETE MIX DESIGN	42
APPENDIX 3 – MEASURED VISUAL SEGREGATION INDEX	43
APPENDIX 4 – THIXOTROPY CURVES.....	44

1 Abbreviations

c.e.	concrete equivalent
C08	Filler sieved from non-washed, crushed 0/8mm sand
LS	Limestone powder
MR	Mass Ratio
SCC	Self-consolidating concrete
SF	Slump flow
SP	Superplasticizer
VMA	Viscosity modifying agent
VSI ^f	Visual segregation index measured on the flow board
VSI ^m	Visual segregation index measured in the concrete mixer
W02	Filler produced crushed 0/8 mm sand (C08). The sand is thereafter sieved to obtain the 0/2 mm fraction and then washed.

2 Introduction

2.1 Principal objectives and scope

The importance of mix design is crucial in order to obtain self-consolidating concrete (SCC) that is fluid enough to completely fill the form and simultaneously stable against segregation of aggregates and bleeding. Stability of SCC can be achieved in at least two ways: by the aid of fines and/or filler, or by the aid of chemical stabilizer.

This study has been made with a practical approach based on scenarios that could occur on an actual building site for ensuring consistent stability and castability of fresh concrete, i.e. addition of filler or stabilizers. Chemical stabilizers are added to concretes with low content of fines i.e. as a substitution for filler. This project studies thus the rheological and fresh properties of concretes and matrices stabilized by addition of fillers versus concretes stabilized with chemical admixtures, the latter generally having a lower matrix content than the filler stabilized concretes. Special attention is given to viscosity and thixotropy since these parameters are believed to influence stability, form-filling ability, the migration and evacuation of entrapped air bubbles and thus the final surface quality of hardened concrete elements.

2.2 Background

2.2.1 Importance of robustness and stability for SCC

Self-consolidating concrete (SCC), first developed in Japan during the mid-1980's, has been described as one of the most innovative developments in the field of concrete technology [De Schutter et al 2008]. Improved placing of the concrete and better work environment are two of the most important benefits of using SCC compared with traditional vibrated concrete. Unfortunately, SCC cast in-situ in Norway has stagnated at a low market share. Main reasons are probably low robustness against fluctuations of the concrete production and that SCC relatively costly. Better control of robustness and cost reduction will thus be of importance to eliminate the reluctance to use more SCC [Vikan 2008].

Robustness can be defined as the ability of fresh concrete to maintain its properties within narrow limits when the proportions of constituent materials change significantly [De Schutter et al 2008]. Stability of fresh SCC defines the ability of a concrete mixture to retain its homogeneity through the fresh phase, both at rest and subject to loads due to transport, form-filling and compaction [Daczko 2002]. Stability can be categorized as dynamic or static. Dynamic stability refers to the characteristic of the concrete mixture to resist segregation during the production, transport and casting process. Static stability refers to the ability of the concrete mixture to resist bleeding, segregation and settlement once all placement and casting operations have been completed [Smeplass 2009].

2.2.2 Rheological description of SCC

Stability and robustness can be described by the rheological properties of the concrete. The behavior of fresh concrete is often approximated by the Bingham model:

$$\tau = \tau_y + \mu_p \cdot \dot{\gamma} \quad (1)$$

where τ_y is the yield stress, $\dot{\gamma}$ is the shear rate, and μ_p is the plastic viscosity.

The yield stress may be associated with filling capacity, more generally, with whether or not concrete will flow or stop flowing under an applied stress. Plastic viscosity may be associated with the velocity

at which a given concrete will flow once it is initiated. In the field of concrete casting, the applied stress is mainly due to gravity. Yield stress is reported to be the most important parameter for formwork filling: A purely viscous fluid (with zero yield stress) will self-level under the effect of gravity. Gravity will then induce a pressure gradient in the fluid if the upper surface of the material is not horizontal. This pressure gradient will generate a shear stress in the material that creates a shear rate and forces the material to flow. The viscosity of the material will influence the time needed to obtain a horizontal surface (i.e. rate of flow) [Roussel 2009].

Thixotropy is defined as the ability of a material to reduce its resistance to flow (apparent viscosity) with increased flow (shear) or agitation and to regain its original stiffness when at rest. The process is repeatable and reversible [De Schutter et al 2008]. Thixotropy has been found to strongly decrease the formwork pressure of SCC [Billberg 2003, Khayat et al. 2005]. Thixotropy has moreover been related to improved SCC stability [Roussel 2006, Lachemi et al 2004, Khayat and Guizani 1997, Rols et al. 1999]. However, highly thixotropic SCC may in specific conditions induce distinct-layer casting that can generate lowered mechanical resistance of the final structure and lowered aesthetic quality of the surface [Roussel and Cussigh 2008].

Thixotropy can be measured as the area between the up- and down-curve for shear stress measured over a shear rate range (hysteresis loop). This area is, however, depending on the shear history of the material. Absolute area values can therefore not be regarded as material parameters [Barnes et al 1989]. Some examples of hysteresis loops shapes are given in Figure 1.

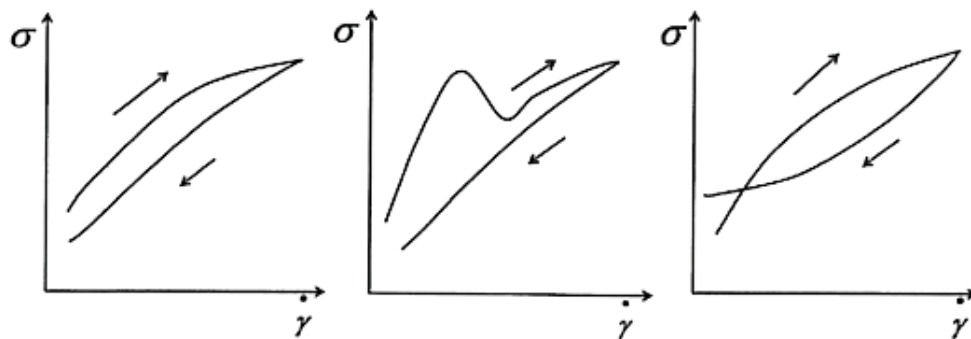


Figure 1: Examples of possible shapes of hysteresis loops [Mewis and Wagner 2009]

The draw backs of the hysteresis loop method can be avoided by instead using stepwise changes in shear rate or shear stress. By shearing the sample at a given shear rate until steady state is reached one can achieve reproducible initial conditions. The shear rate is then dropped and the degree of viscosity recovery recorded [Mewis and Wagner 2009]. The principle of the method is illustrated by Figure 2.

Positive thixotropy values indicate that structure is broken during flow and recovers during rest. Negative thixotropy (rheopexy) indicates that structure builds during flow.

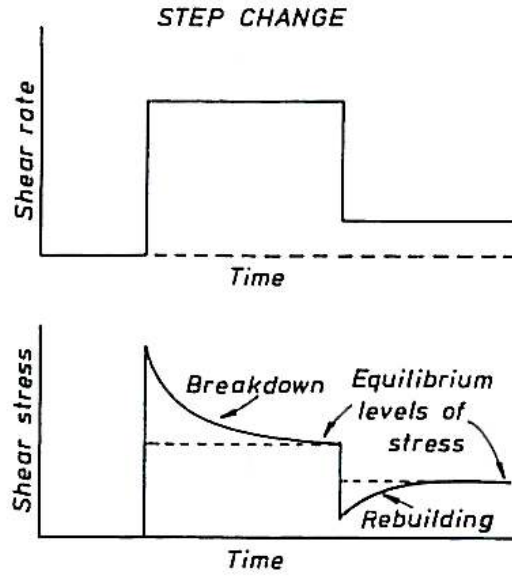


Figure 2: Thixotropy measurements by the shear rate step function. Shear rate profile (above) and theoretical viscosity development (below) as a function of time [Barnes et al. 1989]

2.2.3 The particle-matrix model

A given concrete consist of a wide range of materials such as cement, water, powders, aggregates and admixtures. These materials differ in properties such as particle size, shape and water affinity. The particle-matrix model developed by E. Mørtzell in 1996 is an attempt to simplify the understanding of how the different components, alone or in combination with others, influence the workability of a given concrete. The model assumes that fresh concrete consist of a particle phase (aggregates > 125 μm) dispersed in a fluid matrix phase (water, powders < 125 μm and admixtures). The matrix consists thus according to the model of the fluid component that encloses the solid particle phase and fills all voids [Smeplass and Mørtzell 2003].

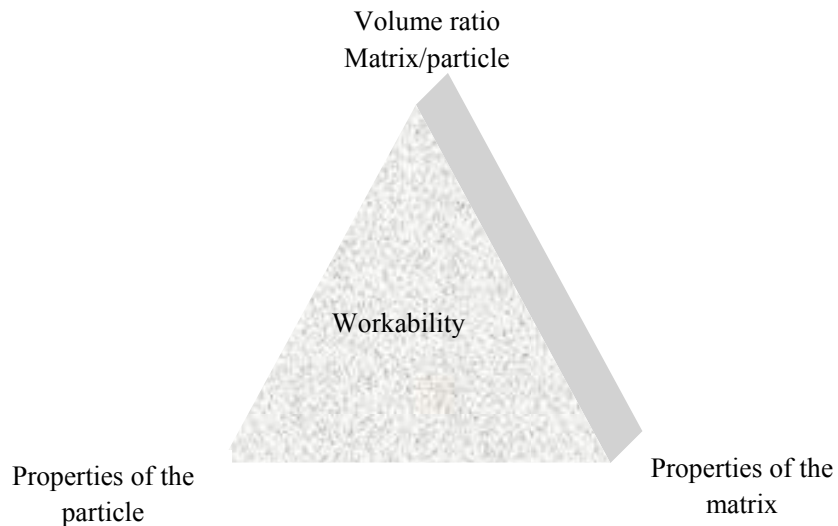


Figure 3: The particle-matrix model (based on the work of Mørtzell, 1996)

2.2.4 Material factors influencing robustness and stability

Segregation within fresh concrete is a phenomenon related to the plastic viscosity and density of the cement paste. It is strongly linked to two assumptions: A solid denser than a liquid tends to sink and a viscous liquid flows with difficulty around a solid [De Schutter et al 2008, Betancourt 1988].

Ways of ensuring stability of SCC include [Takada et al. 1998, Kim et al. 1996, Corradi et al. 2003]:

- Modify viscosity and/or yield stress of the matrix by aid of admixtures known as viscosity modifying agents (VMAs) or stabilizers
- Mineral additions such as ultra-fine amorphous colloidal silica (UFACS), slags and fly ash
- Fillers (defined in Norway as fine aggregate with particle sizes $< 125\mu\text{m}$)
- Use aggregates with the same density as the matrix (probably not an applicable solution with today's natural aggregates)

SCC stabilized with a combination of fines and admixtures have been reported to be more stable against alterations of aggregate humidity than SCCs with either fines or admixture as the only stabilizing component.

2.2.5 Influence of additional powder as stabilizer

Stabilizers in the form of powder can be [Khayat 1998].

- Water swelling materials with high surface area that increases the capacity of retaining water, such as bentonite and silica fume
- Powders with high surface area that increases the content of small particles in the paste and thereby the thixotropy. Examples of this type of material are fly ash, hydrated lime, kaolin, different types of rock dust, diatomaceous earth etc.

Particle symmetry influences both viscosity and particle packing. Deviation from spherical shape results in increased viscosity when the phase volume remains constant. This effect is caused by asymmetrical particles counteracting rotation within the paste to a stronger extent than spherical ones. Correspondingly, particles with a rough surface and oblong shape needs more paste to obtain a given workability than concrete containing spherical particles with a smooth surface. Fillers with unsymmetrical and porous particle shape have also been reported to encapsulate more air than spherical particles [Moosberg-Bustnes 2003].

2.2.6 Influence of chemical stabilizers

Chemical, stabilizing admixtures, also known as anti washout admixtures are added to concretes with low content of fines and can be grouped as [Khayat 1998]:

- Water soluble, synthetic or natural polymers that increases the viscosity of the mixing water. Examples of this type include cellulose ethers, polyethylene oxide, polyacrylamid and polyvinyl alcohol.
- Water soluble flocculants that absorb on the cement surface and increase the viscosity due to increased attraction between the particles. This group includes styrene copolymers with carboxylic groups, synthetic polyelectrolytes and natural rubber.
- Emulsions of different chemical materials that increases the attraction between the particles and supply small particles to the suspension. This group includes acrylic emulsions and clay solutions.

Frequently used stabilizers for cementitious systems have been water soluble polysaccharides such as cellulose-ether derivatives and welan gum. These bind some of the mixing water and increase thereby the viscosity of the matrix [Khayat 1998].

Chemical stabilizers based on cellulose and welan gum have been reported to retard the setting time of cement. This retarding effect has been explained by the polymer chains adsorbing on the cement surface and thus affecting the hydration [Lachemi et al. 2004, Gupta et al 2005, Cooper et al 2005].

Stabilizers have also been reported to result in reduced air content in SCC due to increased viscosity of the paste and thereby increased inner pressure of the air bubbles [Khayat and Assaad 2002].

2.2.7 Influence of superplasticizers

Polycarboxylate based superplasticizers have become popular admixtures for SCC due to their good water reducing properties and ability to maintain flow over time. Polycarboxylate is a generic term for a group of compounds that can be split into the groups acrylate based (A), methacrylic based (B) or maleate based (C) depending on the main chain (see Figure 4) [Pellerin et al 2005].

Stabilizers can through synthesis be integrated into the superplasticizer. Incompatibility between the two admixtures is then avoided [Ghezal and Khayat 2001].

Thixotropic properties of the concrete have been reported to depend on the type of polycarboxylate applied as superplasticizer [Toussaint et al 2001].

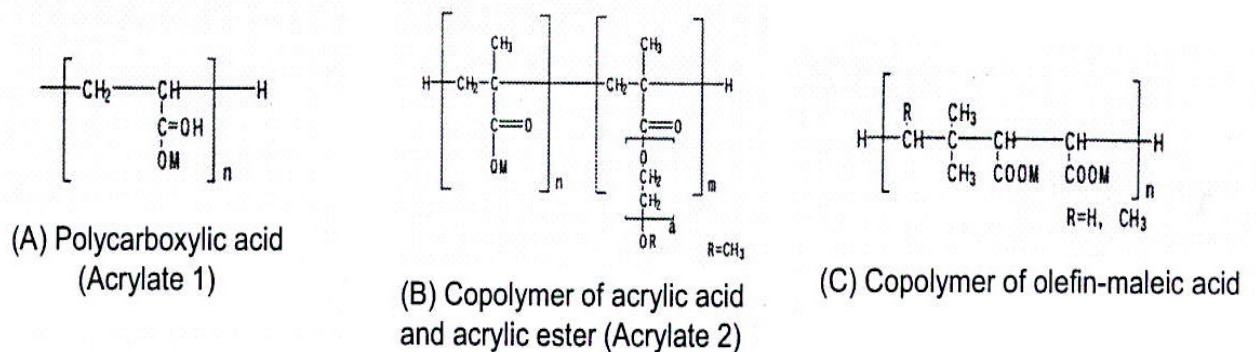


Figure 4: Chemical structure of polycarboxylate based polymers [Pellerin et al 2005].

3 Experimental

3.1 Materials

EN 197-1-CEM II/A-V 42,5 R Portland fly ash cement was used for all experiments. The cement had a Blaine fineness of 450 m²/kg and density of 3010 kg/m³

Gneiss/Granite aggregates of following fractions were used:

- 0/8 mm sand, 111-curve, Årdal
- 8/16 mm stone, Årdal

Grading curves are given in Appendix 1

Filler (< 0.125 µm) sieved from the 0/8 mm sand was used to prepare concrete equivalent matrix.

Three powdered materials have been used as stabilizers. Sieve curves are given below:

- Limestone powder (Norcem) of density 2700 kg/m³ and Blaine 360 m²/kg. Abbreviation: LS
- Filler sieved from non-washed, crushed 0/8mm sand from Årdal, NorStone. Filler density was 2730 kg/m³. Abbreviation: “C 08”/ “Crushed 08”:
- Filler produced from the same material as crushed 0/8 mm sand. The sand was thereafter sieved to obtain the 0/2 mm fraction, washed and sieved once more to obtain the filler. Filler density was 2730 kg/m³. Abbreviation: “W02” / “Washed 02”:

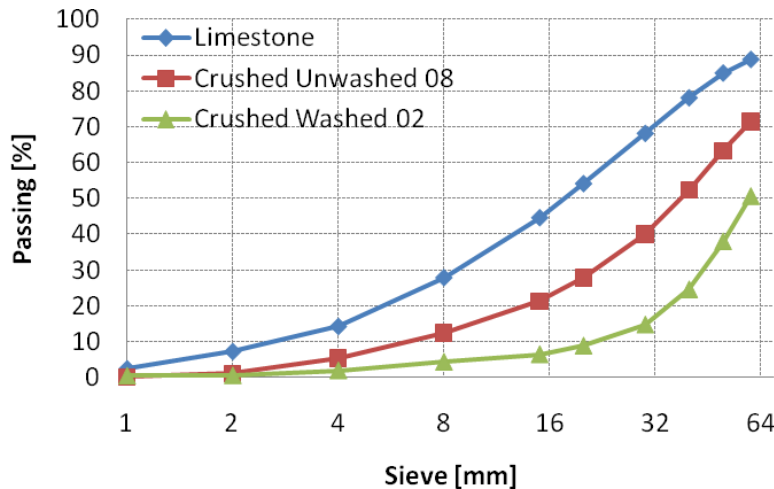


Figure 5: Sieve curves for powdered materials used in the study as stabilizers.

The following chemical stabilizers have been used for the experiments:

- S1: Based on a polymer with high molecular weight. Dry solids: 2.2%. Active ingredients: 5.5%. Normal dosage: 1-4 l/m³ concrete corresponding to 0.3-1.2% of cement weight. Initial dosage used for the experiments: 0.4% to avoid over dose of superplasticizer
- S2: Based on cellulose derivate that according to the producer produce thixotropic properties of the concrete. Normal dosage: 1-2 litre per 100 kg powder (d < 0.125 mm) corresponding to approximately 1-2% of cement weight. Initial dosage used for the experiments: 1% to avoid over dose of SP.

The following superplasticizers were used:

- SP 1: Acrylic polymer with 30% dry solids. Splitting type admixture (i.e. no stabilizing properties). Normal dosage: 0.3-1.2% of cement weight
- SP2: Acrylic polymer with 20.5% dry solids. Splitting type admixture with short open time.

SP2 has lower molecular weight, longer side chains and higher charge density than SP1. These properties result in higher degree of sterical hindrance (i.e. long side chains) and rapid slump loss (adsorbs quickly on the cement particles due to high charge density and relatively low molecular weight).

- SP3: Acrylic polymer with 19.5% dry solids. Normal dosage: 0.4-1.5% of cement weight. The admixture is tailored for ready mix construction with long transport time (long open time). Preliminary experiments showed that the concrete did not respond to increased dosages within the mixing time (up to 20 minutes after water addition). This admixture was thus not suitable for this test series since and therefore excluded from further tests.

Saturation curve of the two superplasticizers SP1 and SP2 are given in Figure 6. The curves were determined by altering the admixture dosage of the reference mixture (without any form of stabilizer addition) and measuring the viscosity of the rheological down curve. The rheological measurement set-up is given in section 3.3.3. The figure shows that a somewhat higher SP2 dosage is needed in order to obtain the same viscosity as for matrices with SP1.

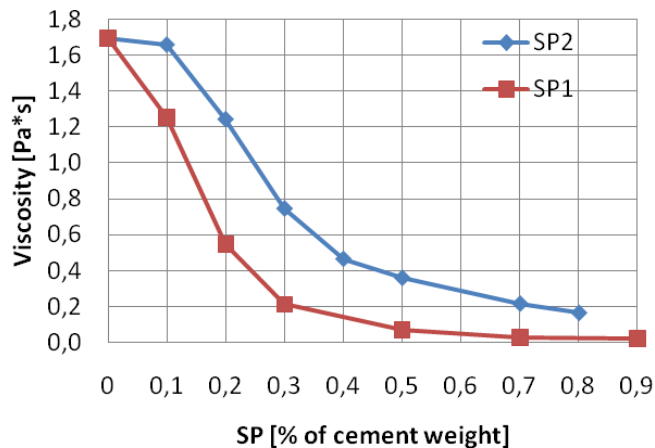


Figure 6: Saturation curve for SP1 and SP2

A set retarder based on Gluconat was added to all concretes at a dosage of 0.4% in order to eliminate the effect of hydration on the rheological measurements. Note that this product has a water reducing effect.

3.2 Concrete experimental set-up

3.2.1 Recipes

The basis of the test matrix is a low grad concrete (M60 according to NS-EN 206-1) with an aimed slump flow of 675 ± 15 mm. The reference concrete is designed in order to be on the verge of separation (i.e. instability).

The mineral fillers were added in two dosages, namely 40 kg/m³ (filler-cement ratio 0.12) and 80 kg/m³ (filler-cement ratio 0.24) while adjusting the superplasticizer dosage in order to keep the slump flow unchanged.

The chemical stabilizers were added according to recommended dosage given by the producer while adjusting the superplasticizer dosage in order to keep the slump flow unchanged.

The concrete mix design is given in Table 1. The superplasticizer was dosed in order to keep the slump flow within a slump flow range of 675 ± 15 mm. The resulting dosages and final experimental matrix are given in Table 2.

Table 1: Concrete mix design

	Reference	40 kg/m ³ Filler	80 kg/m ³ Filler
w/c	0.58	0.58	0.58
w/p	0.46	0.44	0.40
f/c (%)	-	12	24
Matrix (l/m ³)	325	338	352
Paste (l/m ³)	300	314	328
Cement (kg/m ³)	326	326	326
0/8 mm (kg/m ³)	1089.6	1067.1	1043.8
8/16 mm (kg/m ³)	725.0	711.4	695.9

Table 2: Experimental matrix and superplasticizer dosages added in order to obtain a concrete slump flow of 675 ± 15 mm

Stabilizer		Superplasticizer	
Type	Dosage	Type	Dosage (% of cement weight)
0 – Reference	-	SP1	0.86
Limestone powder	40 kg/m ³	SP1	0.67
Limestone powder	80 kg/m ³	SP1	0.83
Crushed 08 mm	40 kg/m ³	SP1	0.77
Crushed 08 mm	80 kg/m ³	SP1	0.94
Washed 02 mm	40 kg/m ³	SP1	0.79
Washed 02 mm	80 kg/m ³	SP1	0.90
S2	1.0 % of cem. weight	SP1	1.00
S1	0.4 % of cem. weight	SP1	0.97
0 – Reference	-	SP2	0.53
Limestone powder	80 kg/m ³	SP2	0.60
Crushed 08 mm	80 kg/m ³	SP2	0.72
Washed 02 mm	80 kg/m ³	SP2	0.78
S2	1.0 % of cem. weight	SP2	0.71
S1	0.4 % of cem. weight	SP2	0.66

3.2.2 Mixing procedure

A forced pan mixer with a volume of 50 litres from Eirich was used to prepare the concretes. The volume of the concretes batches was 40 litres. The concretes were prepared by:

- 1 minute dry mixing of powders and aggregates
- 2 minutes while adding mixing water and half the amount of superplasticizer (previously intermixed with the water) and retarder
- 2 minutes pause/rest
- 2 minutes mixing with addition of remaining superplasticizer until aimed slump flow value was reached

3.2.3 Measurements

Air content and fresh concrete density was measured according to NS-EN 12350-7, 10 minutes after water addition.

Slump flow and T_{500} were measured according to EN 1235080: 2010 10 and 60 minutes after water addition. T_{500} is measured as the time needed for the concrete to reach a diameter of 500 mm as the slump cone is lifted. Slump flow and T_{500} are popular methods since they are quick, simple and can be performed simultaneously. The concrete was remixed for 1 minute before the 60-minutes measurements.

The slump flow was related to yield stress according to the equation derived by Roussel (2007):

$$\tau_{y,SF} = \frac{225 \rho g V^2}{128 \pi^2 R^5} \quad (2)$$

where R is the spread radius and V is the sample volume (6 liters)

Visual Segregation Index (VSI) was also determined 10 and 60 minutes after water addition. VSI was measured on fresh concrete within the mixer (VSI^m) and on the flow board (VSI^f) after determination of slump flow. Table 3 shows the VSI rating within the mixer. Table 4 shows correspondingly the VSI rating on the flow board. A castable concrete should have a VSI^m between 0 and 0.4/0.5 and a VSI^f between 0 and 0.5 – 0.6.

Table 3: VSI^m measured directly after end of mixing in the concrete mixer

0 / 0.1	Stable and homogenous concrete
0.2 / 0.3	Creamy surface and formation of small air bubbles, but still stable.
0.4 / 0.5	Incipient separation, lots of small air bubbles/pores, tendency of sludge layer, formation of black film on the surface.
0.6 / 0.7	Clear signs of separation, strong "boiling", sludge layer, black film, coarse aggregates sinking towards the bottom of the mixer.
0.8 / 0.9	Strong boiling, clear water layer, 5-20 mm sludge layer, aggregates lying at the bottom of the mixer.
1	Complete separation.

Table 4: VSI^f measured on concrete on the flow table directly after a slump flow measurement

0 / 0,1	Stable and homogenous concrete. Aggregates and paste flow towards the rim of the sample.
0.2 / 0.3	Stable and homogeneous concrete that flows well, but has become a shiny surface with possible black spots (usually unburned coal residue liberated from the fly ash when the hollow spheres are crushed upon grinding).
0.4 / 0.5	Has additionally a hint of a paste rim at the outer edge of the spread, but the aggregates follow the flow towards the edge. Still stable.
0.6 / 0.7	Clear rim of paste at the outer edge of the spread. Coarse aggregates tend not to flow towards the edge of the spread (are left in the middle of the spread).
0.8 / 0.9	Additionally separation of water/paste at the outer rim of the spread.
1	Complete separation

Torque (T) was measured simultaneously with the slump flow (10 and 60 minutes after water addition) by aid of a ConTec Rheometer-4SCC. The measurement setup is given in Table 5 and the rotary vane illustrated by Figure 7.

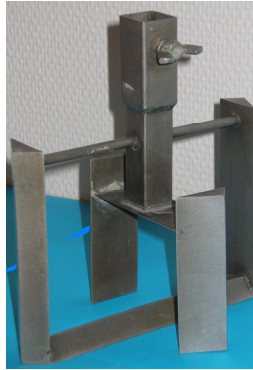


Figure 7: Rotary vane of the ConTec Rheometer-4SCC

Table 5: Setup for ConTec Rheometer-4SCC

Rotational speed, rps	0.47	0.40	0.33	0.27	0.20	0.13	0.07
Duration (s)	10	5	5	5	5	5	5
Measurement points	40	20	20	20	20	20	20

By making a linear regression of the measured torque values the rheological properties G (A) and H (A·s) were obtained as the intersection with the ordinate and the slump of the line respectively. These values can theoretically be translated into the Bingham parameters yield stress (τ_y) and plastic viscosity (μ_p) by the aid of the Reiner-Riwlin equation:

$$T = \frac{8\pi^2 \mu_p h}{1/R_i^2 - 1/R_o^2} + \frac{4\pi\tau_y h}{1/R_i^2 - 1/R_o^2} \ln\left(\frac{R_o}{R_i}\right) = Hf_o + G \quad (3)$$

were R_i and R_o is the inner and outer radius of the rheometer as illustrated by Figure 8, h is the height of the immersed part of the sensor measuring torque, and f_o is the rotational frequency.

Equation (2) shows that plastic viscosity and yield stress are functions of the rheometer geometry. The rotary vanes of the ConTec Rheometer-4SCC have a complicated geometry making use of the Reiner–Riwlin equation in the given form impossible. There are thus currently no equations or programs available for the conversion of G and H to τ_y and μ_p . Values of G and H are therefore used throughout this report.

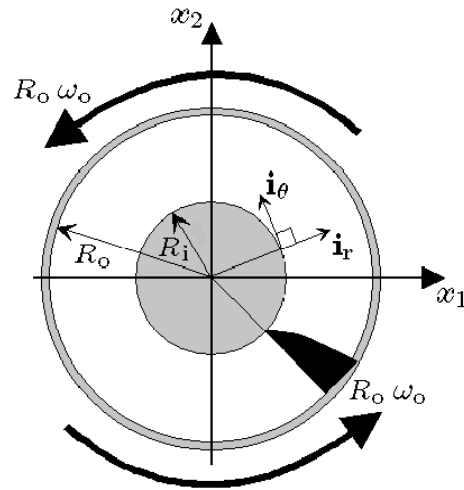


Figure 8: Principle of equation (2). ω_o is the angular velocity and equals $2\pi f_o$ [Wallevik 2003]

Cubes (100x100x100mm) were cast 60 minutes after water addition for determination of compressive strength. The cubes were all cast in one pour without compaction or vibration. The forms were covered with plastic and cured in laboratory atmosphere for 24 hours. The samples were thereafter demoulded and cured in water bath until time of testing. Compressive strength was measured according to NS-EN 12390-3:2009 after 7, 28 and 90 days of curing.

3.2.4 Repeatability

Repeatability of fresh concrete measurements was measured by producing the SP2-reference mix three times and thereafter measuring the fresh concrete properties.

3.3 Matrix experimental set-up

3.3.1 Recipes

The water-cement ratio was 0.45 for all mixes. All pastes were added 0.4% gluconate per cement weight in order to limit effects of early hydration. Total paste volume was 200 ml.

The mixes were designed as concrete equivalent matrices. The pastes were thus added filler sieved from the 0/8 mm sand. Some mixes were also added filler stabilizers. The basis for the matrix recipes are given in Table 6. Experimental matrix is given in Table 7.

Concrete equivalent (abbreviated c.e.) superplasticizer dosages were used for the main test series. In order to eliminate the effect of variable superplasticizer dosage within the test series, additional test series were made for which it was kept constant (see Table 6).

Table 6: Basis of matrix recipes

	Reference	Stabilizer 1	Stabilizer 2	Filler dosage 1	Filler dosage 2
Cement (g)	184.7	184.7	184.7	175.5	167.2
Water (g)	73.9	73.9	73.9	70.2	66.9
Filler (matrix) (g)	37.0	37.0	37.0	34.5	32.1
Filler (stabilizer) (g)	0.0	0.0	0.0	21.1	40.1
Gluconate (%)	0.4	0.4	0.4	0.4	0.4
S1 (%)	-	0.4	-	-	-
S2 (%)	-	-	1.0	-	-
SP1 (%)	c.e*, 0.8, 0.9	c.e*, 0.8, 0.9	c.e*, 0.8, 0.9	c.e*, 0.8, 0.9	c.e*, 0.8, 0.9
SP2 (%)	c.e*, 0.4, 0.5, 0.7, 0.8	c.e*, 0.4, 0.5, 0.7, 0.8	c.e*, 0.4, 0.5, 0.7, 0.8	c.e*, 0.4, 0.5, 0.7, 0.8	c.e*, 0.4, 0.5, 0.7, 0.8

*c.e. = concrete equivalent superplasticizer dosage as given by Table 2

Table 7: Superplasticizer dosages of concrete equivalent matrices

Mixture no.	Superplasticizer		Stabilizer		Filler	
	Type	Dosage (%)	Type	Dosage (%)	Type	Dosage (filler/cement)
Ref	SP1	0.86			-	
1	SP1	0.67			LS	0.12
2	SP1	0.83			LS	0.24
3	SP1	0.79			W 02	0.12
4	SP1	0.90			W 02	0.24
5	SP1	0.77			C 08	0.12
6	SP1	0.94			C 08	0.24
7	SP1	0.97	S1	0.4	-	-
8	SP1	1.00	S2	1.0	-	-
9	SP2				-	
10	SP2				LS	0.24
11	SP2				W 02	0.24
12	SP2				C 08	0.24
13	SP2	0.97	S1	0.4	-	-
14	SP2	1.00	S2	1.0	-	-

3.3.2 Mixing procedure

The matrices were blended in a high shear mixer from Braun (MR5550CA). The blending was performed by adding solids to the water and liquid admixtures (superplasticizer, retarder and stabilizer), mix for ½ a minute, rest for 5 minutes and blend again for 1 minute.

3.3.3 Rheological measurement sequence

The rheological parameters of the paste were recorded by a parallel plate (1 mm gap, upper plate and lower plate serrated) rheometer MCR 300 from Physica. The rheometer temperature control was set to 20°C. The following measurement sequence started 10 minutes after water addition:

- 1 minute pre-shearing with constant shear rate ($\dot{\gamma}$) of 60 s⁻¹ to produce uniform initial conditions
- 1 minute rest without shearing
- Flow curve (hysteresis):
 - Stress (τ) – shear rate ($\dot{\gamma}$) curve with linear sweep of $\dot{\gamma}$ from 1 up to 100 s⁻¹ in 30 points lasting 6 seconds each
 - Stress (τ) – shear rate ($\dot{\gamma}$) curve with linear sweep of $\dot{\gamma}$ from 100 down to 1 s⁻¹ in 30 points lasting 6 seconds each
- Thixotropy:
 - $\dot{\gamma} = 0.1$ in 10 measuring points each lasting 12 seconds (level 1)
 - $\dot{\gamma} = 250$ in 5 measuring points each lasting 6 seconds (level 2)
 - $\dot{\gamma} = 0.1$ in 50 measuring points each lasting 3.6 seconds (level 3)
- 10 seconds rest
- Shear rate ($\dot{\gamma}$) - stress (τ) curve with logarithmic sweep of τ from 0.5-250 Pa in 28 points each lasting 5 seconds in order to measure the gel strength
- 1 minute rest
- Static yield stress: $\dot{\gamma} = 0.02$ s⁻¹ in 60 points each lasting 2 seconds
- Static yield stress: $\dot{\gamma} = 0.02$ s⁻¹ in 60 points each lasting 1 second (sequence not used for analysis)

The thixotropy value is calculated as structure build up by subtracting minimum viscosity (equilibrium value) in thixotropy level 2 from the maximum/peak viscosity (equilibrium value) in thixotropy level 3.

Full mixing and measurement sequence applied on the matrices are illustrated by Figure 9.

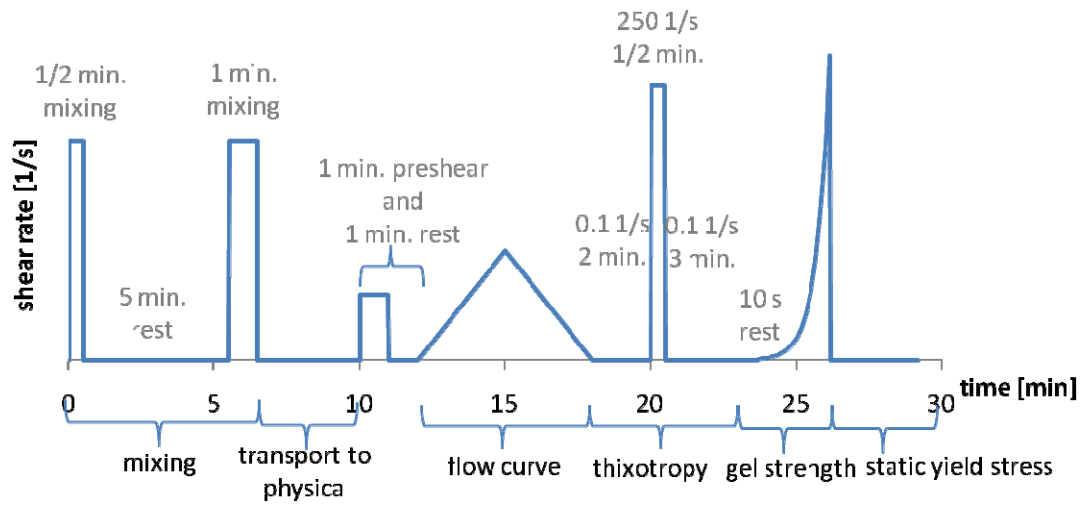


Figure 9: Mixing and measurement sequence of matrix

3.3.4 Repeatability

Repeatability of the rheological measurements was determined by measuring the rheological properties of three identical matrices, all with 0.60% SP2 of cement weight and limestone-cement mass ratio of 0.24. All pastes were stable.

4 Results

4.1 Repeatability

4.1.1 Measured fresh concrete properties

Results of the reproducibility experiments are given in Table 8. The standard deviation of the slump flow experiments are found to be satisfactorily as the basis of the test matrix is a concrete with an aimed slump flow of 675 ± 15 mm. Correspondingly the reproducibility of the calculated yield stress was considered to be satisfactorily. Note however, that the yield stress values are extremely low as the concrete is on the verge of separation.

A major draw back of T_{500} measurements are short flow times (only a few seconds) for very flowable concretes and thus relatively low degree of accuracy [De Schutter et al. 2008]. The standard deviation of these T_{500} measurements is, however, satisfactorily (only 3.4%).

The standard deviation of G and H are very high, corresponding to 19% and 37% respectively. This result is to be expected since the mixes are on the verge of separation and a homogeneous sample is of utmost importance for a reliable result. The low reproducibility of the G and H measurements indicate that these measurements are indicative and should not be used to quantify absolute differences within the test matrix.

Table 8: Repeatability of fresh concrete properties measured on SP2-reference mix 10 minutes after water addition

	SP dosage (%)	Air (%)	Slump flow (mm)	$\tau_{y,SF}^*$ (Pa)	T_{500} (sec)	G (A)	H (A·s)	VSI mixer	VSI table
	0.52	2	662	1.2	1.23	0.71	4.7.	0.75	0.3
	0.53	2	645	1.4	1.16	0.94	3.30	0.75	0.3
	0.53	1.5	658	1.2	1.19	0.66	2.23	0.75	0.4
<i>Average</i>			655	1.3	1.19	0.8	3		
<i>STDEV</i>			9	0.1	0.04	0.2	1		

*Calculated by aid of equation (2)

4.1.2 Measured fresh matrix properties

Results of the reproducibility experiments are given in Table 9. The negative yield stresses are artefacts of the Bingham model that occur due to curvature of the flow curve. Such matrices could in effect be evaluated to have yield stress values approximately equal to zero. The repeatability of viscosity, thixotropy value and static yield stress is considered to be satisfactory. Gel strength values are linked to considerably higher uncertainty than the other parameters.

Table 9: Repeatability of fresh matrix properties measured on three identical matrices, all with 0.60% SP2 of cement weight and limestone-cement mass ratio of 0.24. All pastes were stable.

	Yield stress (Pa)	Viscosity (Pa·s)	Thixotropy value (Pa·s)	Gel strength (Pa)	Static yield stress (Pa)
	-3.7	0.81	23.3	3.9	3.1
	-2.7	0.74	23.3	4.8	3.3
	-3.7	0.81	22.7	3.9	3.5
<i>Average</i>	-3.4	0.79	23.1	4.2	3.3
<i>STDEV</i>	0.6	0.04	0.3	0.5	0.2

4.2 Correlation between fresh properties measured on matrix and concrete

All viscosity related parameters, namely H and T₅₀₀ measured on concrete as well as plastic viscosity measured on matrix are shown in Figure 10. All viscosity parameters are clearly interrelated in spite of the high standard deviation of H and low degree of accuracy for the T₅₀₀ measurement technique. This means that trends found in matrix can be expected to occur also in concrete and vice versa.

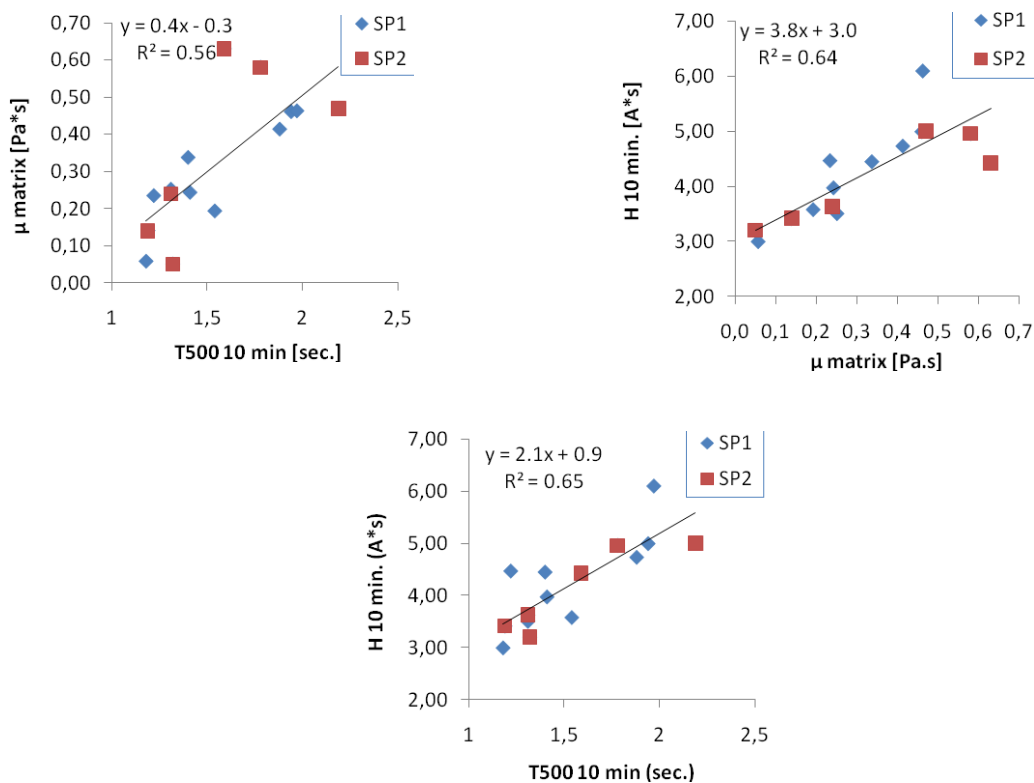


Figure 10: Relationship between viscosity related parameters measured in matrix and concrete

The flow spread diameter correlated with the yield strength (here measured as G). A similar relation between slump flow and yield stress has been reported by Wallevik [2003]. Roussel [2007] reports on the other hand that the thickness of the concrete sample at flow stoppage must be at least five times the size of the largest particle size in order for fluid dynamic principles to apply. Assuming a slump flow diameter of 680 mm and maximum aggregate size 16 mm results in a sample volume of

29 liters for the principles of fluid dynamics to apply. In response to high sample volumes dictated by these principles, Roussel suggests the LCPC box as an alternative method of measuring fresh concrete properties.

There were only vague correlations between yield stress parameters measured on matrix and concrete. Remember that all mixes are designed in order to obtain the same slump flow. Yield stress related parameters are thus varying in a narrow range which can result in a relatively large scatter of results and no clear relationship between the yield stress parameters. Another, and maybe the most important reason, is unreasonable results given for matrix by the Bingham model. As seen by the matrix measurements, very fluid and shear thinning pastes render negative yield stress values without physical meaning.

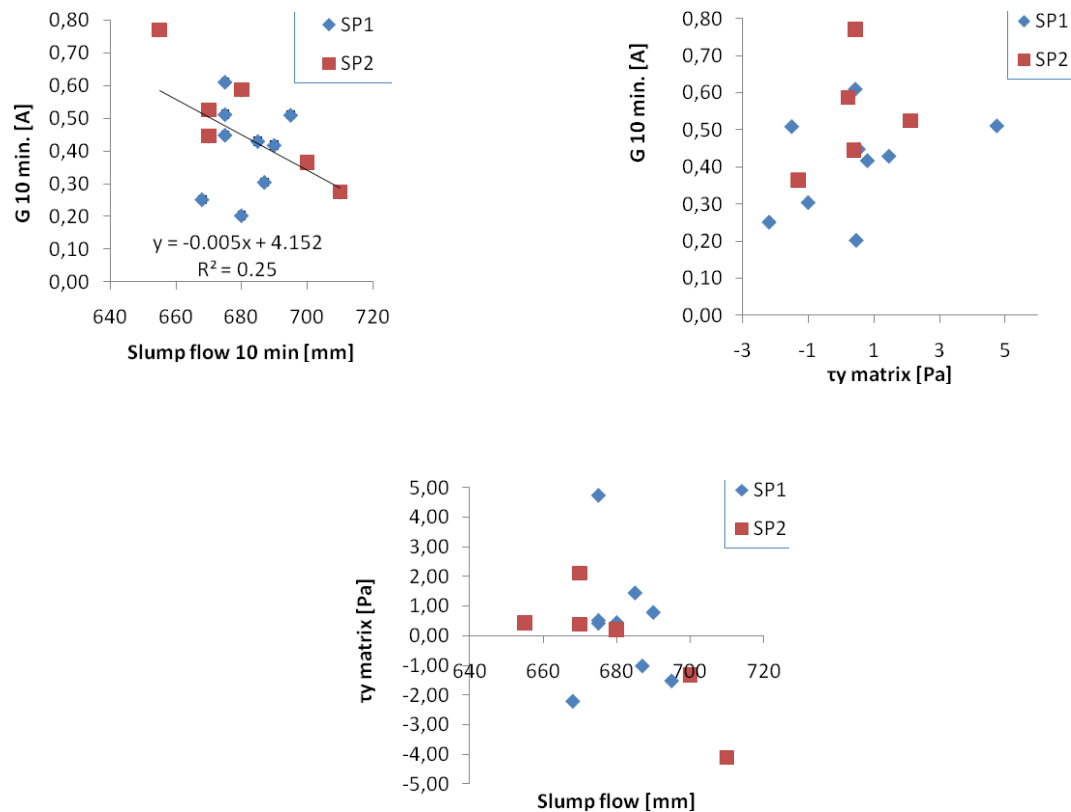


Figure 11: Relationship between yield stress related parameters measured in matrix and concrete

4.3 Fresh concrete properties

4.3.1 Concretes plasticized with SP1

Fresh properties and rheological properties of concretes plasticized with SP1 are given in Table 10 and Table 11 respectively. Yield stresses calculated from the slump flow measurements vary within a very narrow range which is natural considering the concretes being proportioned in order to obtain slump flow of 675 ± 15 mm.

G decreased with the addition of the two chemical stabilizers, probably due to the increased superplasticizer dosages that were added in order to obtain constant slump flow.

G decreased also with addition of fillers. For these mixes, however, the alteration of superplasticizer is probably not the main reason as the dosage in fact decreased by the lowest filler addition. This effect may thus be caused by increased matrix content and “dilution”/dispersion of cement particles.

Loss of slump flow was observed 60 minutes after water addition. The reduction was within the range of 0-6%. No distinction between fillers or stabilizers could be made. Correspondingly, no trends could be found for the development of G over time.

Table 10: Fresh properties of concrete plasticized with SP1

SP dosage (%)	Stabilizer	Matrix (l/m ³)	Slump flow (mm)			$\tau_{y,SF}$ (Pa)	T ₅₀₀ (sec)		
			10 min	60 min	Δ (%)		10 min	60 min	Δ (%)
0.86	-	325	675	650	-4	1.1	1.2	1.8	50
0.97	0.4 S1	325	675	670	-1	1.1	1.5	2.2	47
1.00	1.0 S2	325	685	660	-4	1.0	1.4	1.8	29
0.67	0.12 LS	338	675	635	-6	1.1	1.2	1.8	50
0.77	0.12 c08	338	680	680	-	1.0	1.4	1.6	14
0.79	0.12 w02	338	690	655	-5	1.0	1.3	2.0	54
0.83	0.24 LS	352	668	665	-0	1.1	1.9	2.4	26
0.94	0.24 c08	352	695	665	-4	0.9	2.0	1.8	-10
0.90	0.24 w02	352	687	690	0	1.0	1.9	2.0	5

*Calculated by aid of equation (2)

Table 11: Rheological properties of concrete plasticized with SP1

SP dosage (%)	Stabilizer	Matrix (l/m ³)	G (A)			H (A·s)		
			10 min	60 min	Δ (%)	10 min	60 min	Δ (%)
0.86	-	325	0.6	0.5	-18	3.0	3.4	13
0.97	0.4 S1	325	0.5	0.4	-14	3.6	4.5	24
1.00	1.0 S2	325	0.4	0.5	19	4.0	3.6	-9
0.67	0.12 LS	338	0.5	0.6	36	4.5	4.3	-4
0.77	0.12 c08	338	0.2	0.5	145	4.5	4.1	-7
0.79	0.12 w02	338	0.4	0.4	-7	3.5	4.1	18
0.83	0.24 LS	352	0.3	0.3	12	5.0	4.7	-6
0.94	0.24 c08	352	0.5	0.1	-78	6.1	6.3	3
0.90	0.24 w02	352	0.3	0.1	-55	4.7	6.6	39

The flow time, T₅₀₀, increased with the addition of chemical stabilizer or filler. The T₅₀₀ measurements indicated that highest viscosity was obtained for the highest filler dosage. A differentiation between the effect of the different fillers was, however, not possible.

H increased correspondingly with the addition of chemical stabilizer or filler as illustrated by Figure 12. Filler and thus increased matrix content had generally a stronger effect on H than the addition of chemical stabilizers. Filler from crushed sand obtained the highest H for mixtures dispersed with SP1.

T₅₀₀ increased generally with time (5-50% except for mixture with highest dosage of “crushed 08” that obtained a reduction of 10%). The mixture with S2 had the lowest T₅₀₀ and H increase. Of the fillers “washed 02” had the highest increase of H with time. No distinctions could, however, be made for T₅₀₀ measurements due to different response with different filler dosage.

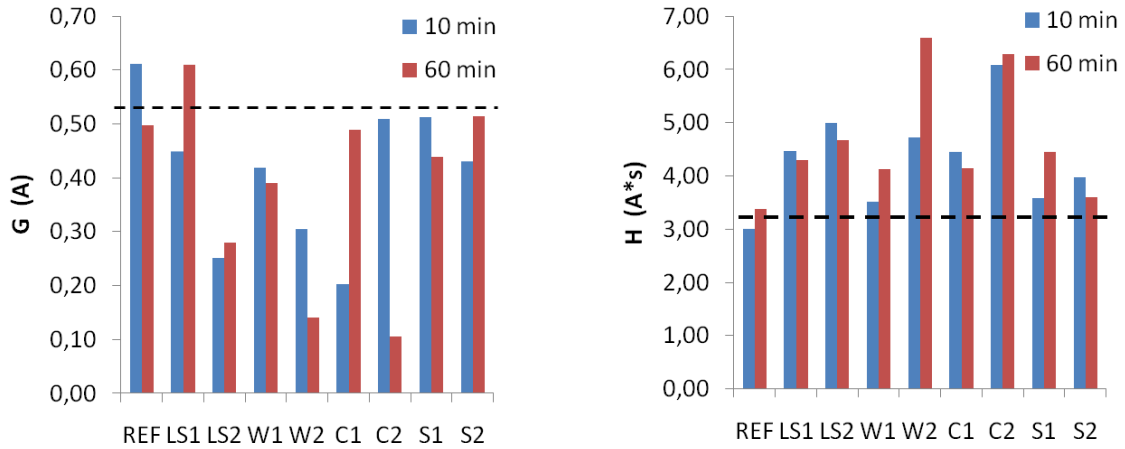


Figure 12: G and H measured on concretes with SP1. Dashed line marks the reference value. No correlations were found for G and H.

The visual segregation indices of mixes with SP1 are illustrated by Figure 13 while the tabulated data are given in Appendix 3. The VSI is generally higher in the mixer than on the flow board, probably since it is easier to evaluate the segregation of aggregates in the mixer. The concretes are, moreover, rated differently when VSI is evaluated in the mixer or on the board. VSI^m indicates that only mixes with limestone powder and “washed 02” are deliverable concretes. VSI^f illustrates on the other hand that all mixes except reference, S2 and lowest dosage of “crushed 08” are deliverable. Summarized results from the two indices are that mixes with limestone filler, “washed 02” and lowest dosage “crushed 08” are considered deliverable.

The VSIs do not directly correlate to the other parameters measured on the fresh concrete. Note how the VSI seems difficult to apply on concretes with chemical stabilizers, resulting in contradicting indices for concrete in the mixer (VSI^m) and on the flow board (VSI^f). It appears thus that this method is difficult to use in order to evaluate relative differences of concrete mix design.

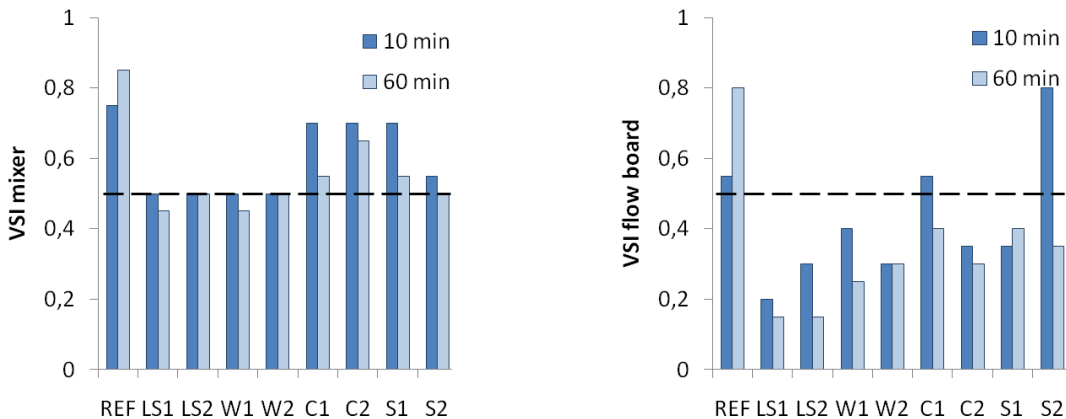


Figure 13: VSI measured on mixes with SP1 in the concrete mixer (left) and on the flow board (right). Dashed line indicates maximum VSI for deliverable concrete.

4.3.2 Concretes plasticized with SP2

Concretes dispersed with SP2 experienced a marked slump loss 60 minutes after water addition in spite of the hydration being retarded as seen by the reference mixture. As a result, the concrete was no longer self-consolidating. The molecules of the superplasticizer seemed thus to be “deactivated” within 60 minutes, an effect that can be attributed to this polymer having lower molecular weight and longer side chains than S1. As a consequence of the slump loss, fresh concrete properties were only measured 10 minutes after water addition.

Both T_{500} and H increased with addition of chemical stabilizers or additional filler. The strongest effect was, however, found, for the addition of fillers. Concretes dispersed with SP1 obtained a stronger viscosity increase with addition of chemical stabilizers than concretes with SP2. The saturation curves given in Figure 6 indicate that the SP1 dosages exceeded saturation, while the SP2 dosages are on the verge of saturation. The higher effect of chemical stabilizers on concretes with SP1 could thus be related to more bleeding water being available for the stabilizers.

Table 12: Fresh properties of concrete plasticized with SP2.

SP dosage (%)	Stabilizer	Matrix (l/m ³)	Slump flow (mm)		$\tau_{y,SF}^*$ (Pa)	T_{500} (sec)	
			10 min	60 min	10 min	10 min	60 min
0.53	-	325	662	415	1.3	1.23	-
0.66	0.4 S1	325	670	-	1.1	1.31	-
0.71	1.0 S2	325	680	-	1.0	1.32	-
0.60	0.24 LS	352	670	-	1.1	1.59	-
0.72	0.24 c08	352	700	-	0.9	2.19	-
0.78	0.24 w02	352	710	-	0.8	1.78	-

*Calculated by aid of equation (2)

Table 13: Rheological properties of concrete plasticized with SP2

SP dosage (%)	Stabilizer	Matrix (l/m ³)	G (A)		H (A·s)	
			10 min	60 min	10 min	60 min
0.53	-	325	0.8	1.57	3.4	4.9
0.66	0.4 S1	325	0.5	-	3.6	-
0.71	1.0 S2	325	0.6	-	3.2	-
0.60	0.24 LS	352	0.5	-	4.5	-
0.72	0.24 c08	352	0.4	-	5.0	-
0.78	0.24 w02	352	0.3	-	5.0	-

None of the stabilizers had any marked effect on the VSI as illustrated by Figure 14. This finding is in line with the low response of H to the addition of these admixtures. “C08” rendered the most stable concrete according to VSI^f. None of the concretes were, however, deliverable when both VSI^m and VSI^f are taken into account. The results are not consistent

with VSIs measured on concretes dispersed with SP1. Tabulated VSI can be found in Appendix 3.

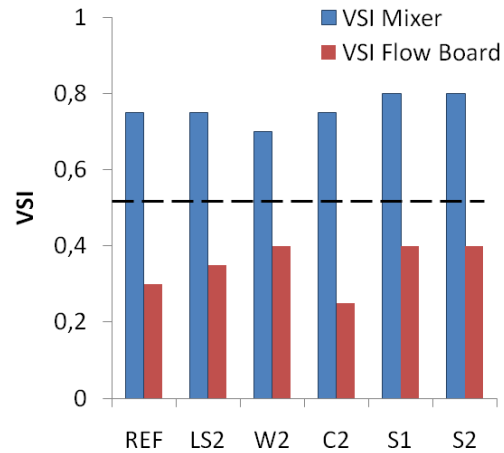


Figure 14: VSI measured on concretes dispersed with SP2

4.4 Fresh properties of matrix

Rheological properties measured on concrete equivalent matrix plasticized with SP1 are given in Table 14. The yield stress increased with the addition of organic stabilizers in accordance with other reports [Khayat et al. 2010]. The yield stress is on the other hand reduced by increasing filler addition. Negative yield stress is a Bingham model artefact caused by curvature of the flow curve [Vikan 2005].

Table 14: Rheological properties of matrices with concrete equivalent SP1 dosage

SP dosage	Stabilizer	Bingham values		Static yield stress 2sec (Pa)	Gel strength (Pa)	Thixotropy value (Pa·s)	Hysteresis area (Pa/s)
		Plastic viscosity (Pa·s)	Yield stress (Pa)				
0.86	-	0.06	0.42	0.26	1.2	3.1	79
0.97	0.4 S1	0.19	4.73	0.64	2.2	20.7	-4
1.00	1.0 S2	0.24	1.45	0.61	3.4	15.7	23
0.67	0.12 LS	0.23	0.52	0.35	1.5	8.2	40
0.77	0.12 c08	0.34	0.45	0.23	2.2	12.0	-92
0.79	0.12 w02	0.25	0.79	0.53	1.5	7.8	13
0.83	0.24 LS	0.46	-2.20	0.56	1.8	6.0*	-319
0.94	0.24 c08	0.46	-1.51	0.15	1.8	5.1*	-389
0.90	0.24 w02	0.41	-1.01	0.28	1.2	3.4*	-267

*Equilibrium values in section 3

Static yield stress increased by the addition of chemical stabilizers, indicating a build up of structure. The effect of the two stabilizers were comparable. No consistent trends could be found for matrices where filler was added as stabilizer.

The gel strength values were not affected by the addition of chemical stabilizers or filler.

Plastic viscosity increased with the addition of chemical stabilizers and by adding filler. The effect of the lowest filler addition was in the same range as found for the chemical stabilizers. This result corresponds with measurements done on concrete. The viscosity increased with increased filler addition, probably due to adsorption of free water in the mix. The differences in plastic viscosity for pastes with w02, c08 and limestone filler were too small to make any distinction between them.

Addition stabilizers resulted in both positive and negative hysteresis areas. Figure 15 indicates that the prefix of the hysteresis area depends on the plastic viscosity: Low viscosities resulting in positive hysteresis and vice versa. A negative hysteresis area may be related to structure build up during flow. However, no conclusions on stabilizing effect can be drawn based on the calculated hysteresis areas.

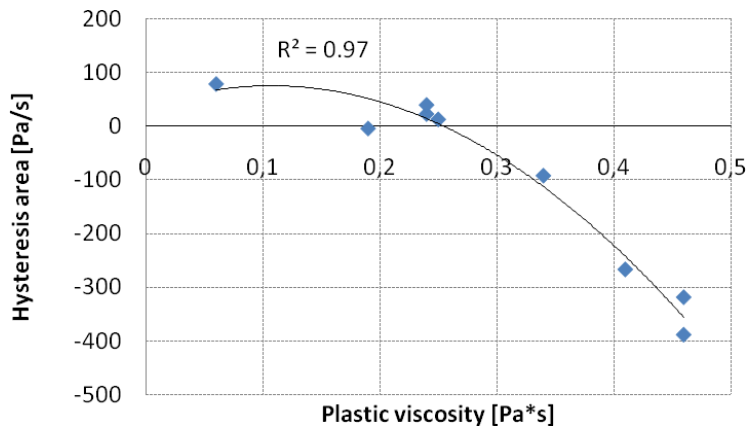


Figure 15: Relation between hysteresis area and plastic viscosity

Thixotropy values measured on matrices plasticized with SP1 are given in Table 15 while the thixotropy curves are illustrated in Appendix 4. Both chemical stabilizers resulted in increased thixotropy values compared to the reference mix. The stabilizer of polymer type, S1, produced the strongest effect of all stabilizers tested within the study. The thixotropic property of this admixture was, however, unstable resulting in structural breakdown with time. This property could result from alignment of the polymer in the direction of flow. The cellulose based stabilizer, S2, produced matrices with stable thixotropic property. The thixotropic effect of the chemical stabilizers increased with decreasing superplasticizer dosage. This illustrates that increased dispersion is linked to decreased structural buildup in form of thixotropy .

In case of the addition of filler as stabilizer combined with the concrete equivalent superplasticizer dosages, maximum thixotropy value was found for the lowest filler dosage. Results obtained on matrices with constant superplasticizer dosage showed, however,

increased thixotropic value with increasing filler dosage. The lowest filler dosage rendered, however, lower thixotropic values than the reference when constant superplasticizer dosage was applied. This effect may be caused by filler dispersing the paste. Increased filler dosage will thereafter absorb more of the free water that again can be the cause of increased thixotropy as seen by the results.

Table 15: Thixotropy values for matrices with SP1

Stabilizer	Concrete equivalent SP dosage		Constant SP dosage	
	[%]	Thix. Value [Pa·s]	[%]	Thix. Value [Pa·s]
-	0.86	3.1	0.8	9.2
0.4 S1	0.97	20.7	0.8	38.5
1.0 S2	1.00	15.7	0.8	19.5
0.12 LS	0.67	8.2	0.8	4.9
0.12 c08	0.77	12.0	0.8	10.9
0.12 w02	0.79	7.8	0.8	3.1
0.24 LS	0.83	6.0*	0.8	6.7
0.24 c08	0.94	5.1*	0.8	19.5
0.24 w02	0.90	3.4*	0.8	16.1

*Equilibrium values in section 3

Thixotropy values and Bingham parameters measured on matrices plasticized with SP2 are given in Table 16 and Table 17 while the curves are given in Appendix 4.

The following effects of stabilizers were found for matrices with concrete equivalent SP2 dosages: Yield stress increased by addition of S1 (polymer type) despite of increasing SP2 dosage. No such effect was found for S2 (cellulose type), an effect that can be explained by the SP2 dosage being increased even more than for the matrix with S1 addition. No effect was found on viscosity or thixotropy by addition of S1 and S2.

Addition of filler resulted in decreased yield stress. Filler addition resulted moreover in increased viscosity and thixotropy. The increase seemed to relate directly to the filler fineness; limestone producing the highest values and filler from crushed 08 mm (c08) producing the lowest. Increased viscosity by increased volume fraction of solids is in line with the Krieger-Dougherty equation [1959].

Following effects of stabilizers were found for matrices with constant SP2 dosages within the series:

Yield stress increased by addition of S1 (polymer type) and S2 (cellulose type). The strongest effect was found for the polymer type. The highest filler dosage resulted in increased yield stress when 0.5% SP2 was added. No filler effect was found on the yield stress when the SP2 dosage increased to 0.7%. Addition of chemical stabilizers had no effect on the viscosity. The thixotropy increased, however, by the addition of these admixtures. The strongest effect was found for the polymer type.

Table 16: Thixotropy and Bingham parameters for concrete equivalent matrices with SP2

Stabilizer	Concrete equivalent			
	SP [%]	Yield stress [Pa]	Viscosity [Pa·s]	Thix. Value [Pa·s]
-	0.53	0.43	0.14	3.2
0.4 S1	0.66	2.91	0.20	1.2
1.0 S2	0.71	0.21	0.05	3.2
0.12 LS	-	-	-	-
0.12 c08	-	-	-	-
0.12 w02	-	-	-	-
0.24 LS	0.60	-3.49	0.80	19.3
0.24 c08	0.78	-5.01	0.65	0.0
0.24 w02	0.72	-4.90	0.64	5.3

The fillers had a stronger effect on the rheological results than the chemical stabilizers. Addition of filler resulted in decreased yield stress for the lowest dosage, followed by increased yield stress for the highest dosage. Yield stress values of the lowest filler dosage are rated according to the filler fineness, the finest filler giving the highest yield stress value and vice versa. Viscosity increased with addition of filler. No distinctions between the filler types could be made in respect to viscosity.

The thixotropy value increased by filler addition and increased filler fineness. The thixotropy value decreased markedly with increasing superplasticizer dosage (i.e. dispersion). The highest filler dosage had a much stronger effect on thixotropy for these mixes contrary to the findings made for matrices with S1.

Table 17: Thixotropy and Bingham parameters for matrices with constant SP2 dosages

Stabilizer	0.5% SP			0.7% SP		
	Yield stress [Pa]	Viscosity [Pa·s]	Thix. Value [Pa·s]	Yield stress [Pa]	Viscosity [Pa·s]	Thix. Value [Pa·s]
-	-1.10	0.41	8.5	-1.73	0.26	0.0
0.4 S1	4.76	0.47	43.3	0.87	0.33	6.8
1.0 S2	3.92	0.43	37.8	0.19	0.18	2.0
0.12 LS	-2.16	0.63	23.1	-3.65	0.37	0.1
0.12 c08	-0.76	0.43	18.2	-3.47	0.37	1.0
0.12 w02	-0.47	0.54	19.9	-1.56	0.27	0.0
0.24 LS	14.39	0.91	189.0	-1.34	0.76	28.5
0.24 c08	6.66	0.89	107.2	-4.35	0.54	7.1
0.24 w02	1.87	0.71	50.3	-4.75	0.53	1.4

4.5 Concrete compressive strength

Compressive strengths of concretes plasticized with SP1 and SP2 are illustrated in Figure 16 and Figure 17 respectively. The polymer type stabilizer had a positive effect on the compressive strength while no effect can be found for addition of the cellulose type stabilizer. Khayat et al. [2010] has similarly found that adding viscosity enhancing admixtures increased the concrete compressive strength. The effect varied with type of VEA and superplasticizer. The authors regarded, however, the differences to be minor.

“Crushed 08” and “washed 02” resulted in an increase of compressive strength (8-11% at 28 days). The effect was close to identical for the two fillers and seemed independent of filler dosage.

Addition of limestone powder increased the compressive strength notably: 28 days compressive strength was increased by 16 and 21% for dosage 1 and 2 respectively.

Limestone powder has previously been found to have a positive effect on the compressive strength of SCC [Pettersson 2001]. This effect has been related to the limestone particles functioning as nucleation sites for the hydration products which accelerate the early cement hydration [Ye et al. 2005]. This effect will, however, probably not affect the 28 days strength much. Some studies conclude that limestone powder do not take part of the cement hydration, while other report formation of carboaluminate. Carboaluminate formation reportedly boosted by fly ash giving more carboaluminate hydrate to the system [De Weerd 2011]. Improved particle packing by addition of filler has, moreover, been reported to increase the density of the matrix phase and the transition zones within cured concrete [Bosiljkov 2003].

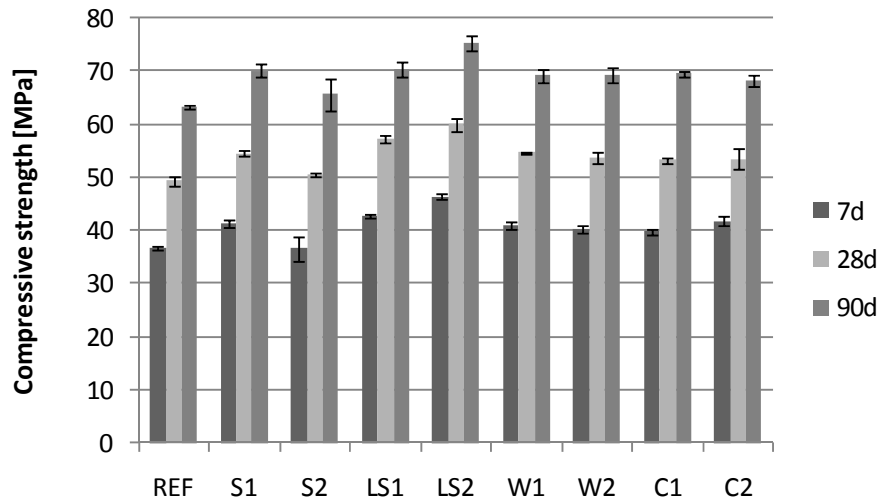


Figure 16: Concrete compressive strength for mixes with SP1. Each value is calculated as an average and standard deviation of 3 cubes

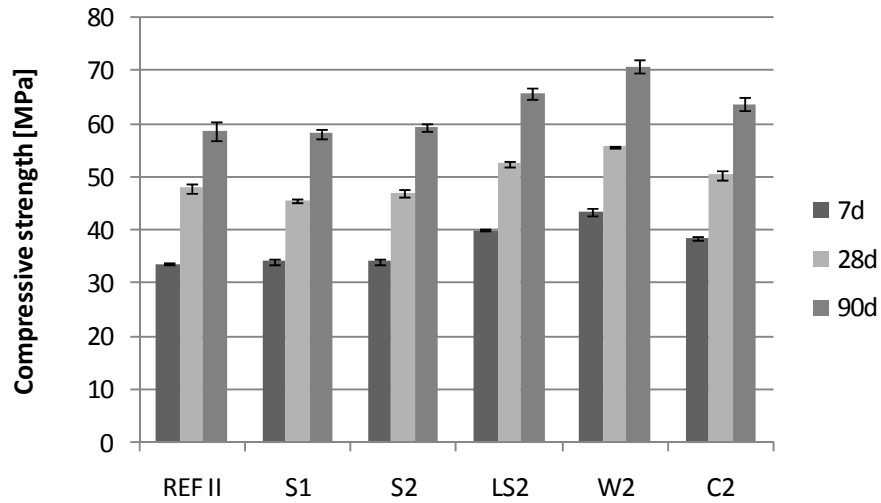


Figure 17: Concrete compressive strength for mixes with SP2. Each value is calculated as an average and standard deviation of 3 cubes.

5 Conclusions

All concretes were designed in order to obtain a slump flow of 675 ± 15 mm. The slump flow was adjusted by altering the SP1 and SP2 dosages. The superplasticizer saturation curves indicated that the SP1 dosages selected for concrete and concrete equivalent matrix exceeded saturation, while the SP2 dosages were on the verge of saturation. The test series with SP1 consisted thus of concretes that were more unstable than the series with SP2. This finding is supported by SP2-concretes generally, apart from the reference mixes, showing higher visual stability in the concrete mixer than the SP1-concretes. No distinctions could, however, be made on basis of visual segregation index measured on the flow board.

Thus, direct comparison of the two test series was difficult as the SP2-test series were somewhat more stable than the SP1-test series. For instance, higher effect of chemical stabilizers on concretes with SP1 could be related to more bleeding water being available for the stabilizers.

5.1 Visual Segregation Index as a means for evaluating stabilizing effect

The VSI does not directly correlate to other rheological properties measured on concrete namely slump flow, T_{500} , G or H.

VSI measured in the mixer (VSI^m) and on the flow board (VSI^f) did not always give consistent results: The concrete is generally rated more unstable (higher VSI) in the mixer than on the flow board, probably since it is easier to evaluate the segregation of aggregates in the mixer.

The VSI seems difficult to apply on concretes with chemical stabilizers, resulting in contradicting indices for concrete in the mixer (VSI^m) and on the flow board (VSI^f). It appears thus that this method is difficult to use in order to evaluate relative differences.

5.2 Fresh and rheological properties of concrete and matrix

Consistent rheological results were obtained in concrete and concrete equivalent matrix.

5.2.1 Yield stress parameters

For both superplasticizers, SP1 and SP2, addition of chemical stabilizers resulted in increased matrix yield stress. The polymer based admixture resulted in a stronger yield stress increase than the cellulose based admixture.

Addition of filler resulted in no matrix yield stress trends, which was to be expected since the concretes were composed in order to obtain slump flows of 675 ± 15 mm. Comparison of slump flow and G gave correspondingly little information about the stabilizing properties of the fillers.

5.2.2 Viscosity parameters

Viscosity parameters measured on concretes and matrices with either SP1 or SP2 increased with the addition of chemical stabilizer.

Matrix viscosity increased with increasing volume fraction of solids in line with the Krieger-Dougherty equation. The viscosity increase of matrices and concretes with SP2 related directly to powder fineness; the finest producing the highest viscosity. Corresponding results were found for concrete equivalent matrices with SP1, but the differences between the concretes were too small to make any distinctions between them.

Concretes and concrete equivalent matrices with chemical stabilizers and SP1 produced viscosity parameters in the same range as the lowest powder dosages.

In case of SP2, however, the effect of powder on viscosity was much stronger than the effect of the chemical stabilizers.

5.2.3 Matrix thixotropy – Structural buildup

Both S1 and S2 increased the matrix thixotropy values independently of superplasticizer type. Generally, the polymer type, S1, produced higher thixotropy values than the cellulose type, S2. The thixotropic effect decreased, moreover, with increasing superplasticizer dosage (i.e. increased dispersion). The thixotropic rating of stabilizers depended however upon plasticizer type:

- SP1: The polymer based admixture, S1, had the strongest influence on thixotropy of all stabilizers tested within the study.
- SP2: Matrices with powder addition produced the highest thixotropy values. This finding coincides with the viscosity measurements.

As for the viscosity measurements, there were no clear trends of powder type and thixotropy when SP1 was applied as superplasticizer. In case of matrices with SP2, the thixotropy values increased with increasing powder fineness.

5.3 Concrete compressive strength

Powder addition resulted in increased compressive strength. The effect was largest for concretes with limestone powder for which the 28 days compressive strength was increased by 16 and 21% for dosage 1 and 2 respectively.

“Crushed 08” and “washed 02” resulted in a slight increase of compressive strength (8-11% at 28 days). The effect was close to identical for the two fillers and seemed independent of filler dosage.

The polymer type stabilizer had a positive effect on the compressive strength. No effect could be found for addition of cellulose based stabilizer.

6 Suggestions for further work

The Visual Segregation Indices gave contradictory results. Further studies should therefore include a stability test, for instance as sieve segregation test, electrical conductivity or bleeding in order to better evaluate stability of the concretes.

Effect of chemical stabilizer dosages should be studied.

An additional type of superplasticizer (for instance SX-600 that has longer open time than SP2) could be of interest.

Combinations of chemical stabilizers and powders (fillers, silica fume, fly ash, slag etc) could be of interest.

Effect of cement type and batch on stability and robustness could be studied. Field experience has for instance shown that production and casting of concrete with white cement can be difficult in respect to stability and final surface quality.

Finally, a few concrete mixes shall be selected for full-scale experiments were wall elements shall be cast in laboratory conditions in order to evaluate castability and final surface quality.

References

- Barnes, H. A., Hutton, J. F., Walters, K., "An Introduction to Rheology", Elsevier, Amsterdam, 1989
- Betancourt G.H., Admixtures, workability, vibration and segregation, *Materials and Structures* **21** (1988) 286-288
- Billberg P., Form pressure generated by self-compacting concrete, proceedings of the 3rd international RILEM Symposium on Self-Compacting Concrete, RILEM PRO33 Reykjavik Iceland, 2003, pp. 271-280
- Bosiljkov, V.B., "SCC mixes with poorly graded aggregate and high volume of limestone filler", *Cem. Concr. Res.* **33** (2003) 1279-1286
- Collepari, M., "Self Compacting Concrete: What is new?", Proceeding of the Seventh CANMET/ACI Conference on Superplasticizers and Other Chemical Admixtures in Concrete, Berlin (Germany), pp.1-16, 2003.
- Cooper, C., Liotta, F.J., Shawl, E.T., "Optimization of polycarboxylates for use in self-consolidating concrete", The Fourth International RILEM Symposium on Self-Compacting Concrete, Chicago 2005, pp. 25
- Corradi, M., Khurana, R., Magarotto, R., "User friendly self-compacting concrete in precast production", The Third International RILEM Symposium on Self-Compacting Concrete, Reykjavik 2003, pp. 457
- Daczko, J.A., "Stability of Self-Consolidating Concrete, Assumed or Ensured?", First North American Conference on the Design and Use of Self-consolidating Concrete, 2002
- De Schutter G., M. Bartos P. J., Domone P., Gibbs J., "Self-Compacting Concrete", Whittles Publishing, Scotland, UK, 2008
- De Weerd K., Kjellsen K.O., Sellevold E.J. and Justnes H., "Synergy between fly ash and limestone powder in ternary cements" *Cem. Conc. Comp.* **33** (2011) 30-38
- Ghezal, A., Khayat, K.H., "Optimization of cost-effective self-consolidating concrete", The Second International Symposium on Self-Compacting Concrete, Tokyo 2001 pp. 329
- Gupta, S., Kumar, P., Lepcha, K.C., Tripathi, S.M., "Study of the effect of hyperplasticizer on the characteristics of concrete", The Fourth International RILEM Symposium on Self-Compacting Concrete, Chicago 2005, pp. 33
- Khayat K., H., Hwang S.-D., Belaid K., Performance of Cast-in-Place Self-Consolidating Concrete Made with Various Types of Viscosity-Enhancing Admixtures, *ACI Materials Journal* **107** (4) (2010), 403-412.
- Khayat K.H., Assad J., Mesbah M., Lessard M., Effect of section width and casting rate on variations of formwork pressure of self-consolidating concrete, *RILEM Mat. Struct.* **38** (1) (2005) 73-78

- Khayat, K.H., Assaad, J., "Air-Void Stability in Self-Consolidating Concrete", *ACI Materials Journal* **99** (4) (2002) 408-416
- Khayat, K.H., "Viscosity-Enhancing Admixtures for Cement-Based Materials – An Overview", *Cem. Concr. Comp.* **20** (1998) 171-188
- Khayat K.H., Guizani Z., use of viscosity modifying admixtures to enhance stability of fluid concrete, *ACI Mater. J.* **94** (4) (1997) 332-340
- Kim, J.K., Han, S.H., Park, Y.D., Noh, J.H., Park, C.L., "Experimental research on the Material Properties of Super Flowing Concrete", Proceedings of the International RILEM Conference on Production and Workability Methods of Concrete, Paisley, June 3-5, 1996, pp. 271-284
- Krieger I.M., Doughert T.J., "A Mechanism for Non-Newtonian Flow in Suspensions of Rigid Spheres", *Trans. Soc. Rheol.* **3** (1959) 137-152
- Lachemi M., Hossain K.M.A., Lambros V., Nkinamubanzi P.C., Bouzoubaâ N., Self-consolidating concrete incorporating new viscosity modifying admixture, *Cem. Concr. Res.*, **34** (6) (2004) 917-926
- Mewis J., Wagner N.J., Thixotropy, *Advances in Colloid and Interface Science* 147-148 (2009) 214-227
- Moosberg-Bustnes, H., "Characterisation of fillers", The Third International RILEM Symposium on Self-Compacting Concrete, Reykjavik 2003, pp. 540
- Mørtzell E.: "Modellering av delmaterialenes betydning for betongens konsistens" Dr. ing avhandling, Institutt for konstruksjonsteknikk, NTH, Trondheim 1996.
- Pellerin, B., Maitrasse, P., Micollier, S., "Improving consistency of SCC rheological behaviour by means of appropriate admixturization", The Fourth International RILEM Symposium on Self-Compacting Concrete, Chicago 2005, pp. 17-24
- Persson, B., "A Comparison between Mechanical Properties of Self-Compacting Concrete and the Corresponding Properties of Normal Concrete", *Cem. Concr. Res.* **31** (2001) 193-198
- Rols S., Ambroise J., Péra J., Effects of different viscosity agents on the properties of self-leveling concrete, *Cem. Concr. Res.* **29** (2) (1999) 261-266
- Roussel N., From Rheology of Fresh Concrete to Casting processes. Correlating properties with field performance. *Concr. Int.* **March** (2009) 48-52
- Roussel N., The LCPC BOX: a cheap and simple technique for yield stress measurements of SCC, *Materials and Structures* **40** (2007) 889-896
- Roussel N., A theoretical frame to study stability of fresh concrete, *RILEM Mat. Struct.* **39** (1) (2006) 81-91
- Roussel N., Cussigh F., Distinct-layer casting of SCC: The mechanical consequences of thixotropy, *Cem. Concr, Res.* **38** (2008) 624-632

Smeplass S., "Chapter 3 - Fresh concrete workability, TKT 4215 Concrete Technology 1", NTNU, revision, modification and English translation May 2009 by Stefan Jacobsen and Jon Håvard Mork

Smeplass S., Mørtzell E.: "The applicability of the particle matrix model to self compacting concrete (SCC)", Nordic Concrete Research Publ. 26 (2003)

Takada, K., Pelova, G.I., Walraven, J.C., "Self-Compacting Concrete Produced by Japanese Method with Dutch Materials", Proceedings of the 12th European Ready Mixed Concrete Congress, 23-26 June 1998, Lisbon, Vol. 2 pp.775-785,

Toussaint, F., Juge, C., Laye, J.M., Pellerin, B., "Assessment of thixotropic behaviour of self compacting microconcrete", The Second International Symposium on Self-Compacting Concrete, Tokyo 2001 pp. 89

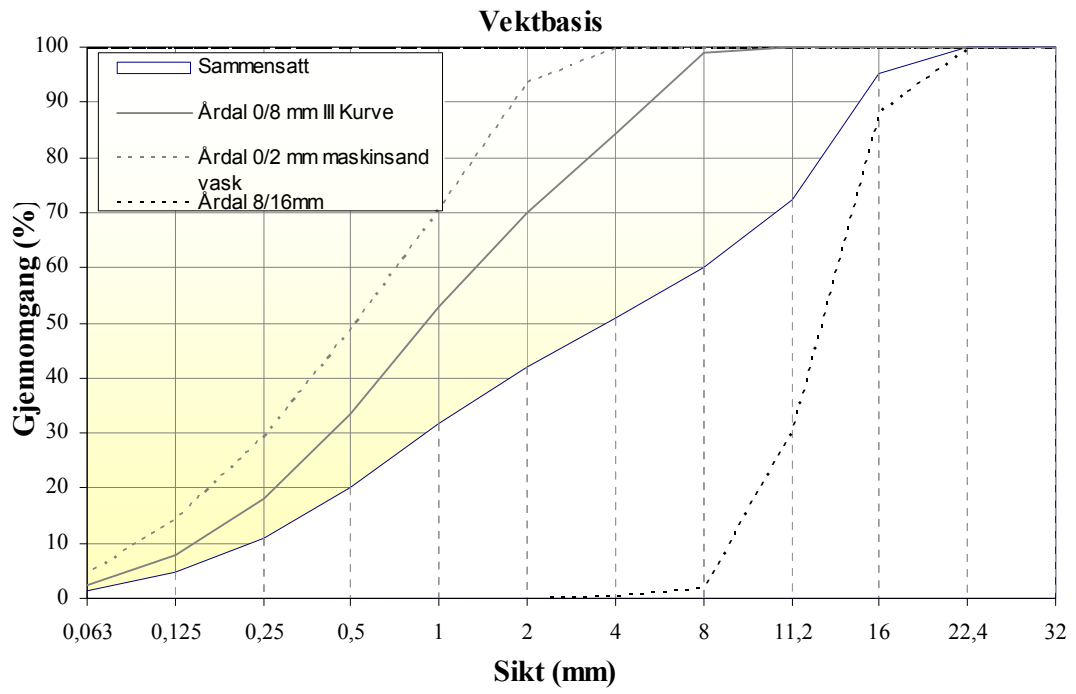
Vikan H., "Means of improving concrete construction productivity – State of the art", COIN Project report 8 – 2008, ISSN 1891–1978 (online), ISBN 978–82–536–1068–9 (pdf)

Vikan, H., "Rheology and reactivity of cementitious binders with plasticizers", Doctoral Theses at NTNU 2005:189, Also available at <http://www.diva-portal.org/ntnu/theses/abstract.xsql?dbid=689>

Wallevik J.E., Rheology of particle suspensions: Fresh concrete, mortar and cement paste with various types of lignosulfonates, PhD Thesis, NTNU, 2003:18

Ye, G., Poppe, A. –M., De Schutter, G., van Breugel, K., Liu, X., "Microstructure of self-compacting concrete: experimental study and numerical simulation", The Fourth International RILEM Symposium on Self-Compacting Concrete, Chicago 2005, pp. 881

APPENDIX 1 - Grading curve aggregates 116-kurve 0/8-fraction

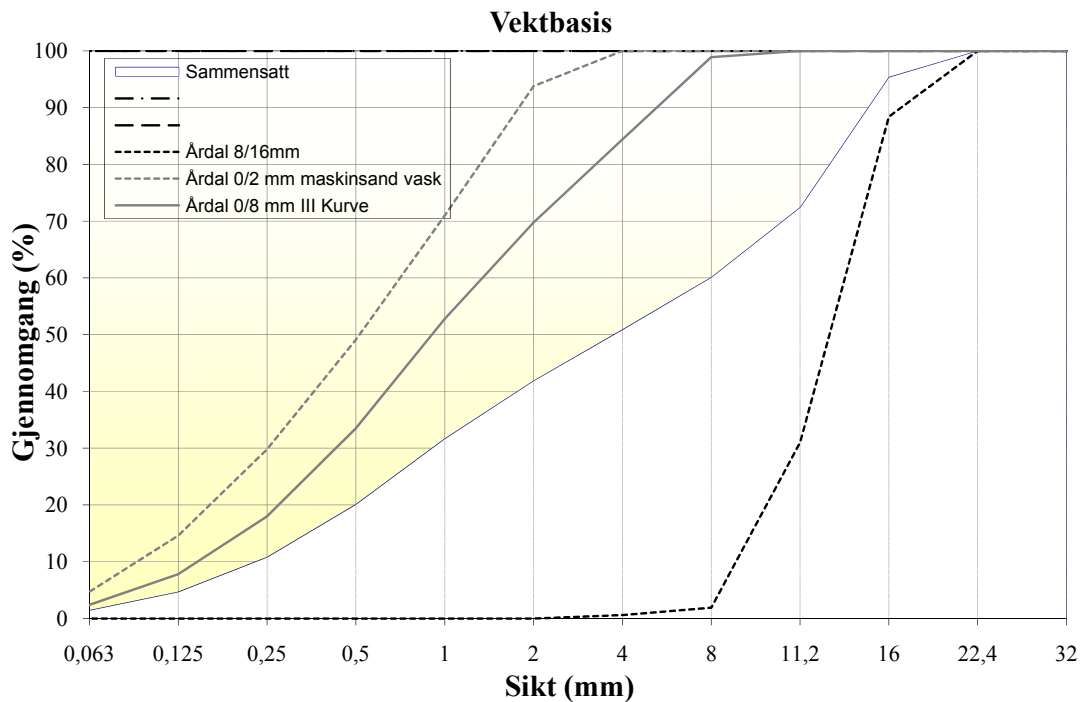


APPENDIX 2 – Influence of aggregates from the 116-curve (0/8 mm) on the concrete mix design

Preliminary mixes were made with aggregates with too high filler content of the 0/8 mm fraction for the purpose of the experiments. The grading curve is given below. It was consequently very difficult to design SCC compositions on the verge of separation. Table A1 shows the resulting matrix content as additional filler is added to the mixture as stabilizers.

Table A1: Influence of filler from 116-curve (too high filler content) 0/8 fraction on concrete composition

	Basis/Reference	40 kg/m ³ Filler	80 kg/m ³ Filler
w/c	0.58	0.58	0.58
w/p	0.46	0.43	0.39
f/c (%)	-	12	24
Matrix (l/m ³)	340	355	370
Paste (l/m ³)	308	324	340
Cement (kg/m ³)	335	336	336



APPENDIX 3 – Measured Visual Segregation Index

Visual segregation index for concretes plasticized with SP1

SP dosage (%)	Stabilizer	Matrix (l/m ³)	VSI ^m		VSI ^f	
			10 min	60 min	10 min	60 min
0.86	-	325	0.75	0.85	0.55	0.80
0.97	0.4 S1	325	0.70	0.55	0.35	0.40
1.00	1.0 S2	325	0.55	0.80	0.80	0.35
0.67	0.12 LS	338	0.50	0.45	0.20	0.15
0.77	0.12 c08	338	0.70	0.55	0.55	0.40
0.79	0.12 w02	338	0.50	0.45	0.40	0.25
0.83	0.24 LS	352	0.50	0.50	0.30	0.15
0.94	0.24 c08	352	0.70	0.65	0.35	0.30
0.90	0.24 w02	352	0.50	0.50	0.30	0.30

Visual segregation index for concretes plasticized with SP2

SP dosage (%)	Stabilizer	Matrix (l/m ³)	VSI ^m		VSI ^f	
			10 min	60 min	10 min	60 min
0.53	-	325	0.75	-	0.30	-
0.66	0.4 S1	325	0.80	-	0.40	-
0.71	1.0 S2	325	0.80	-	0.40	-
						-
0.60	0.24 LS	352	0.75	-	0.35	-
0.72	0.24 c08	352	0.75	-	0.25	-
0.78	0.24 w02	352	0.70	-	0.40	-

APPENDIX 4 – Thixotropy Curves

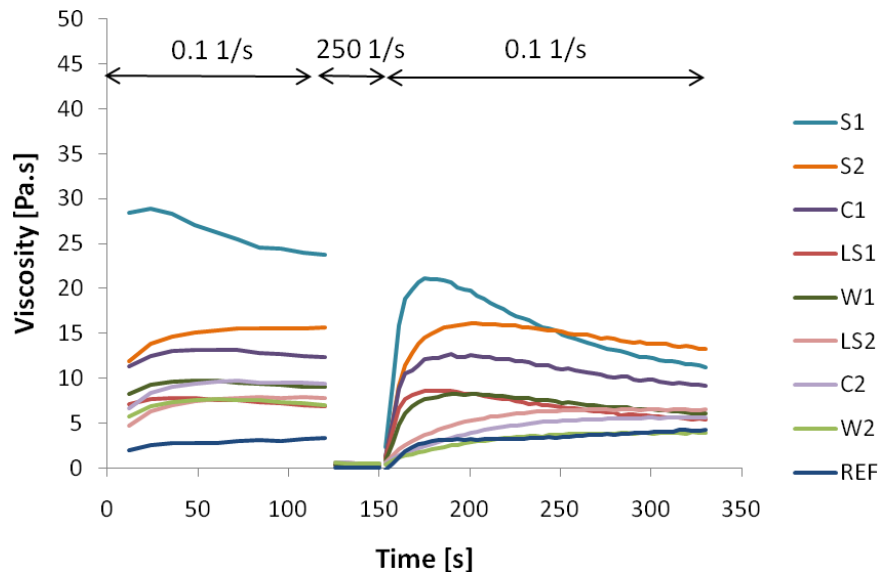


Figure A4.1: Thixotropy curves for matrices plasticized with concrete equivalent dosages SP1

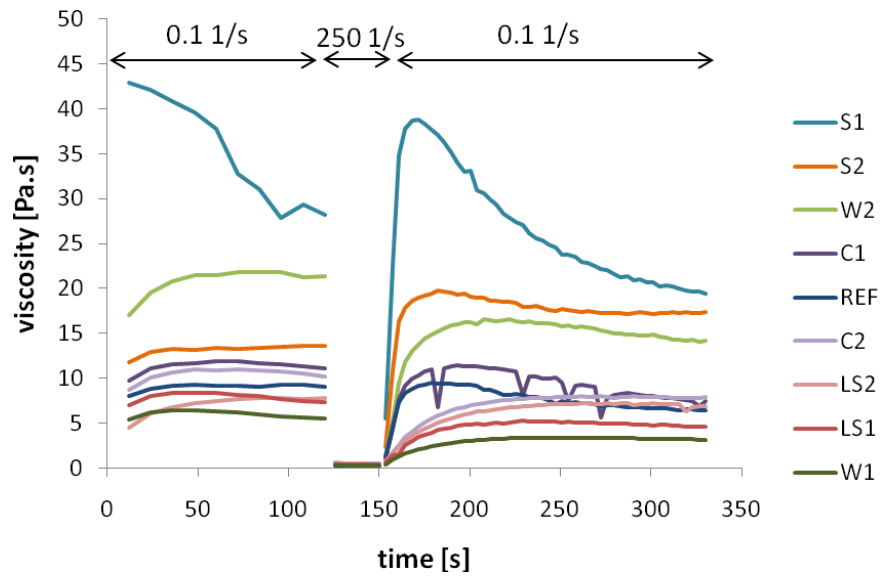


Figure A4.2: Thixotropy curves for matrices plasticized with 0.8% SP1

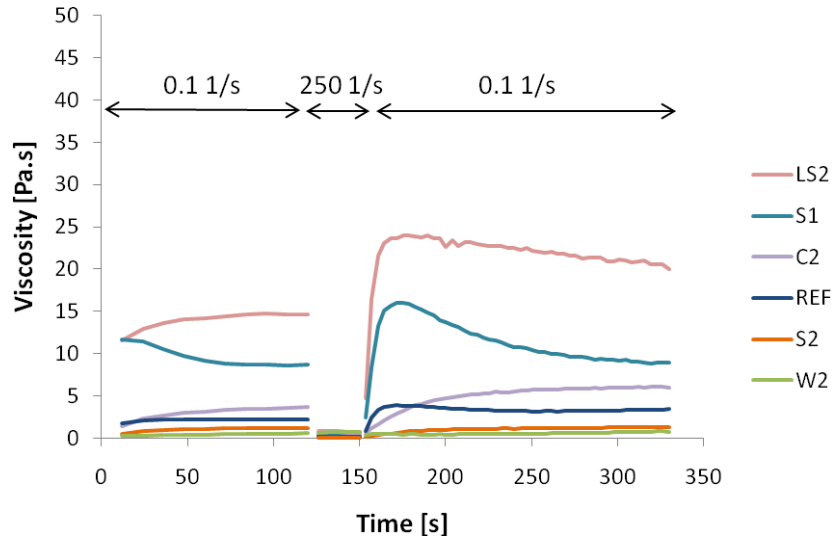


Figure A4.3: Thixotropy curves for matrices with concrete equivalent SP2 dosages

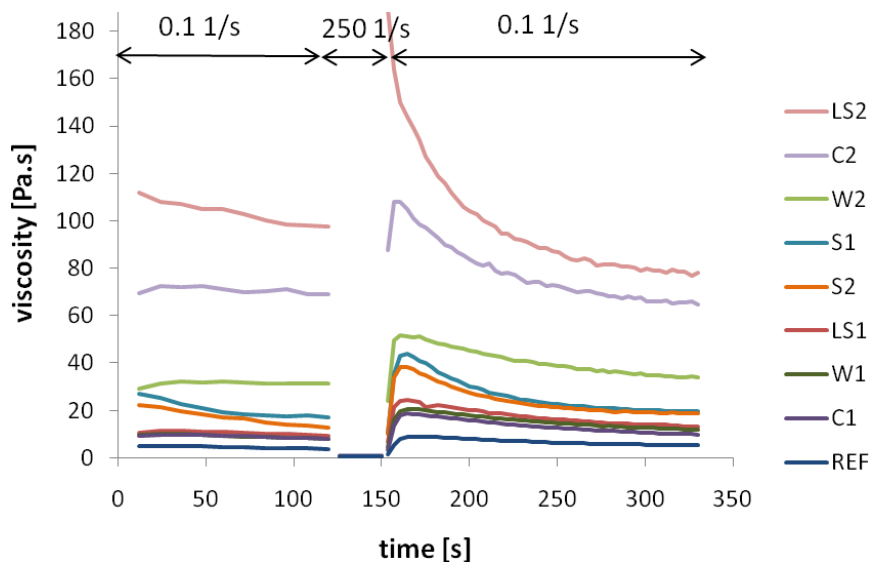


Figure A4.4: Thixotropy curves matrices plasticized with 0.5% SP2

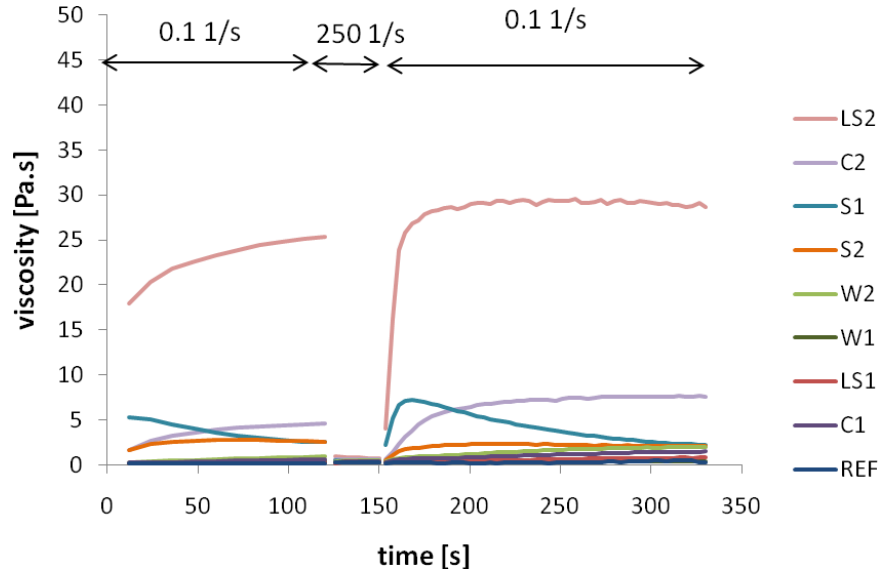


Figure A4.5: Thixotropy curves matrices plasticized with 0.7% SP2

SINTEF Building and Infrastructure is the third largest building research institute in Europe. Our objective is to promote environmentally friendly, cost-effective products and solutions within the built environment. SINTEF Building and Infrastructure is Norway's leading provider of research-based knowledge to the construction sector. Through our activity in research and development, we have established a unique platform for disseminating knowledge throughout a large part of the construction industry.

COIN – Concrete Innovation Center is a Center for Research based Innovation (CRI) initiated by the Research Council of Norway. The vision of COIN is creation of more attractive concrete buildings and constructions. The primary goal is to fulfill this vision by bringing the development a major leap forward by long-term research in close alliances with the industry regarding advanced materials, efficient construction techniques and new design concepts combined with more environmentally friendly material production.

