THE EFFECT OF RHEOLOGICAL PARAMETERS ON STATIC SEGREGATION OF SELF-COMPACTING CONCRETE MORTAR

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DECLARATION

I, Mzwandile Jakuja, declare that the content of this thesis represents my own unaided work, and that the thesis has not previously been submitted for academic examination towards any qualification. Furthermore, it represents my own opinions and not necessarily those of the Cape Peninsula University of Technology.

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ABSTRACT

The stability of mortar is generally understood as the ability of the suspension to remain homogeneous during and after fresh mortar placement or casting. This is mostly associated with the segregation of the suspension that can be defined at a static and dynamic level. At a static level, this phenomenon is simplified by Stokes' equation, while at a dynamic level the characterisation becomes more complex due to the horizontal and translation motion of solid particles that must be considered simultaneously. Static segregation consists primarily of the downward migration of solid particles from the liquid medium remaining on top of the suspension (bleeding). Available literature has established a relationship between the rheology of fresh mortar and its stability, stating that viscosity is the determinant rheological parameter of the suspension able to dictate mortar stability.

Cement mortar is a suspension with two mediums consisting of sand particles as the solid phase and cement paste as the liquid phase. It is argued that the overall performance of the suspension depends on the individual behaviour of the two phases. In accordance with Stokes' law, solid particles should overcome the physical characteristics of the intermediate medium to settle effectively. Cement paste has to therefore exhibit microstructural strength to avoid sand particle settling. This is normally attributed to the yield stress of the cement paste. This means that the cement yield stress is the strength of the liquid phase that cannot be overlooked at the expense of overall mortar viscosity, as currently noted in the literature.

High performance cement mortars also require the inclusion of superplasticisers whose effectiveness depends primarily on their chemical structure (group function) and the dosage at which they are used. It is thus important to understand the compatibility between the superplasticisers and cements since their interactions affect the cement paste that can in turn alter the stability of the cement mortar.

Three different cements and two superplasticisers were used in this study. All cements were CEMI with distinct contents of aluminate and silicate phases manufactured in three different plants. The superplasticisers were poly-carboxylates with a specific molecular structure that defines their impact on the setting time. Mortars with different pastes exhibiting discrete yield stress values were designed. These yield stresses were achieved at optimum dosage of the product resulting from the blending of the two superplasticisers. The products consisted of mixing superplasticisers in different proportions at the set dosage. Rheological measurements were performed both at mortar and paste scale to estimate their yield stress and viscosity values. The Total Organic Carbon (TOC) was done at paste scale to determine the adsorption of superplasticiser on the cement particles within the suspension.

This research confirmed that the stability of mortar depends not only on its overall viscosity, but also on the yield stress of its cement paste phase that defines the strength that opposes gravity acting on the sand solid particles to cause them to settle. Moreover, the study highlights the possibility of achieving a high performing superplasticiser by blending two different superplasticisers at an optimum dosage. In particular, mortar with high yield stress cement pastes exhibited more stable suspensions with lower segregation indexes. In contrast, mortar with lower cement yield stress values exhibited higher segregation indexes resulting in a mortar with poor stability. There is no definitive evidence, according to the results, to indicate that yield stress and viscosity have an effect on bleeding. Results from TOC measurements were in agreement with the literature showing that cement pastes with higher adsorption superplasticisers have lower yield stress values and vice versa.

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DEDICATION

I dedicate this to every person with a dream and vision who is stuck because of circumstances, persecution, negativity, financial difficulties, oppression, lack of opportunity, lack of support, injustice, fear to fail, jealousy, background, race, religion, addiction, discouragement, depression, health, gender or age.

‘Stand firm and you will win life’ (Luke 21:19)

And

To my parents: I am trying to achieve what you desired to achieve but could not.

To my children: Be whom God has called you to be and do not let anyone tell you otherwise.

To my lovely wife: Thank you for all your support and words of wisdom through my studies.

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TERMS AND CONCEPTS

Admixtures	Chemical additives added to concrete
ASTM	American Society for Testing and Materials
B	Final bleed water
Bleeding	Displacement of water to the concrete surface due to particle settlement
C-S-H	Calcium Silicate Hydrate
Clinker	Round small aggregates produced from the kiln heat during cement manufacturing
Cement Paste	Mixture of water and cement with admixtures added in SCC
EFNARC	European Federation for Specialist Construction Chemicals and Concrete Systems
Hydration	Chemical process between cement, water and admixtures
Mortar	Mixture of cement, sand and water with admixtures added in SCC
PEG	Polyethylene glycol
PPC	Pretoria Portland Cement
Rheology	Study of the deformation and flow of matter
SANS	South African National Standards
SCC	Self-Compacting Concrete
SCCM	Self-Compacting Concrete Mortar
SCCP	Self-Compacting Concrete Paste
Segregation	When concrete cannot remain homogenous and separation of materials occurs

SI	Segregation Index
Solution	Mixture of more than a single constituent
Stability	Capability for a cementitious mix to remain homogeneous throughout
Superplasticiser	Chemical admixture which acts as a water reducing agent in cement solutions
SP X	X fraction of SP within the product superplasticiser
TMH	Technical Methods for Highways
TOC	Total Organic Carbon
UHPC	Ultra-High-Performance Concrete
w/c	Water cement ratio
Viscosity	Physical parameter that describes a fluid's resistance to flow
Yield Stress	Minimum stress which is required for a material to begin flowing

NOMENCLATURES

Constants

A_1	Surface tension of the material	nM/m
CO_2	Carbon Dioxide	
C_2S	Dicalcium Silicate	
C_4AF	Tetracalcium Aluminoferrite	
C_3A	Tricalcium Aluminate	
C_3S	Tricalcium Silicate	
g	Gravitational force	m/s^2
h	Stoppage depth	mm
K	Particle shape coefficient	
M_{bot}	Oven dry mass of the sample obtained from the top mini-column section	
M_{top}	Oven dry mass of the sample obtained from the bottom mini-column section	
R	Spreading diameter	mm
r	Radius of a particle	m
V	Volume of cone	m^3
v	Settling velocity	m/sec
V_1	Volume of bleed water at the end of test	
V_w	Volume of the mortar at the beginning of test	

Greek and Latin Letters

λ	Coefficient of the fluid and the unknown angle	
ρ	Density of sample	Kg/m^3
τ_y	Yield stress	N/m^2
τ	Shear stress	N/m^2
μ	Plastic viscosity	N/m^2. s
θ	Wetting contact angle	
$\dot{\gamma}$	Shear rate	S^{-1}
γ	Specific gravity of a particle	Kg/m^3

CHAPTER 1

INTRODUCTION

1.1 Background and motivation

Self-compacting concrete (SCC) is concrete that flows by gravity and does not segregate, nor does it require the use of mechanical vibrators during placing (EFNARC, 2005). This, however, presents a challenge where stability of self-compacting concrete, mortar or paste is concerned. The different particle sizes and densities are considered to be one factor that would influence the suspensions to not remain homogeneous due to the gravitational force which sinks the aggregates (segregation) and push the water to the surface (bleeding). Yan et al. (2020) suggest that ensuring that aggregates within an SCC system are well graded (ensure the fines sufficiently fill the voids between the coarse particles) and adjusting the SCCM rheology will provide stability in SCC. On this account, rheological parameters such as yield stress and viscosity are therefore necessary to assure the flowability and the stability of the concrete mix for a higher quality SCC. Rheological parameters are affected by many factors such as cement and aggregate characteristics, admixtures, water demand and quantity. The yield stress of a cementitious suspension has to be adequately sufficient and the viscosity sufficiently increased to provide a strong net that will slow down segregation or prohibit the aggregates from sinking (Margarita et al. 2019).

This study focused on cement mortar of self-compacting concrete as opposed to conducting concrete experiments. According to Navarrete and Lopez (2017), static segregation of concrete is more strongly influenced by the yield stress of its mortar and the difference in density between the aggregates and the mortar as the suspending medium. Cement mortar is a suspension with two phases. The liquid phase is primarily the cement paste and the solid phase is constituted by sand particles. The rheology of cement paste, however, is of great importance since it allows the transportation and cohesion of aggregates during flow and at stop. The effect of mortar rheology on stability is investigated.

Nunes et al. (2011) suggest that the rheological properties of self-compacting concrete paste (SCCP) dictates the flowability of the actual concrete depending on the type of aggregates used within a mix. This ultimately renders it less complicated to conduct experiments at either cement paste or mortar scale as variables and experimental costs are reduced. Yahia et al. (2016) argue that rheological behaviour observed at paste scale cannot be used to predict that of corresponding concrete. However, a rheological trend between these two cementitious materials can be established.

Ferraris et al. (2001) also supported this, stating the main cause of the inability to obtain a direct correlation between paste mortar and concrete rheology is that the conditions under which cement pastes are assessed differ from concrete, including the omission of aggregates and sand, amongst other things. They suggest that reliable results of paste can be obtained to estimate concrete performance by ensuring that the method and speed of mixing are similar. The findings from Kabagire et al. (2019) acknowledge the complexity of this, but found that it is possible to establish a rheological correlation between two adjacent phases such as cement paste and cement mortar, rather than two extreme phases such as cement paste and concrete.

Producing good flowability without using additional water can be achieved by the addition of superplasticisers in SCC. Two superplasticisers were used in this research at optimum dosages. They were later blended together at the optimum dosage to investigate the stability at various yield stress values and to evaluate if there could be a possible improvement in the rheological properties by doing so. SP1 is an old type of superplasticiser and SP2 is from the newest range of superplasticisers from the manufacturer. Superplasticisers not only disperse cement particles but also significantly affect the rheology of SCC. Rheology is deemed to be a major contributing factor and solution to address the in-situ concrete placing and formwork problems prevalent in the construction industry (Varela et al., 2020).

According to Malherbe (2015), SCC in South Africa has not been utilised extensively as compared to other countries resulting in limited knowledge about product availability locally when compared to the international market. Malherbe’s statement reiterated the conclusion in earlier studies by Geel et al. (2007). Their study concluded that SCC was less popular than traditional concrete in South Africa at the time. The non-existent conformity standards, lack of specifications for SCC and the dearth of engineers who can comfortably design or draw up specifications for the product are major reasons for its unpopularity. Many experienced contractors lacked expertise on SCC, while the vast majority of people who have used SCC locally have used it primality as an alternative construction method that allows the easy placement of concrete in complex situations above everything else.

Vance et al. (2015) suggest that the manner in which cement particles interact with each other create forces such as van Der Waals and steric forces, this is regarded as a main contributing factor to plastic viscosity in cementitious solutions as this, according to the authors, results in the reduction of flow. However, the influence of these forces on segregation in a cementitious mix still requires in-depth exploration. It is acknowledged that the instability in concrete renders in undesired effects such as bleeding and segregation which can negatively affect the strength and result in high shrinkage (Margarita et al., 2019). While previous research has shown the effect of rheological properties of the suspension medium on the segregation that occurs within concrete systems, the effect of yield stress on the static segregation still requires further investigation.

In their study, Hallal et al. (2010) used various mineral admixtures and different superplasticisers to obtain different results or each superplasticiser with each cement type. Furthermore, Massoussi et al. (2017) investigated bleeding in cement pastes, finding that if sufficient yield stress is maintained in cement pastes, bleeding can be reduced.

There is an evident gap in research concerning how different superplasticisers react with different cements using locally available materials. The aim of this study was to investigate the yield stress and viscosity values at which the stability in cement suspension is satisfactory, as their effect on static segregation and bleeding is not well known. As superplasticisers are sensitive to the chemical and physical characteristics of cements, this investigation was conducted using two different superplasticisers from one manufacturer and three cements that were manufactured at three different plants, physically and chemically different. In addition, this study explored the effects of the different cements and mixing of the two superplasticisers at different ratios to determine the impact this had on the rheology and stability of the mortar and paste mixes.

1.2 Research problem

The effect that rheological parameters have on static segregation of SCCM is not well understood.

1.3 Research questions

- Is there a relationship between the yield stress of the cement paste and the segregation of cement mortar (SCCM)?
- Do chemical characteristics of cements affect the stability of corresponding cement mortar?
- Can the rheology and stability of cement mortar be differently affected by the blending of two different superplasticisers?

1.4 Aims and objectives

The aim of this research was to determine the effect of yield stress and viscosity on the stability of self-compacting concrete mortar (SCCM) by evaluating the relationship between the stability (segregation and bleeding) of cement mortar and the rheological parameters (yield stress and viscosity) of cement paste. The research objectives were as follows:

- to optimise each superplasticiser (SP1 and SP2) with all three cements
- to blend the superplasticisers (SP1 and SP2) by mixing them in variant proportions at the obtained optimum concentration; and
- to evaluate the stability of the SCCM system for different cement mortars using the combined superplasticisers (SP3).

1.5 Delineation

The hardened properties of cement mortar were not assessed, other rheological parameters apart from yield stress and viscosity were not considered, and the rate of adsorption of each superplasticiser on the cement particles was not evaluated. The clay content of the sand and its effect on the superplasticisers was not considered.

1.6 Assumptions

The sand was considered inert and only the interaction of the superplasticisers and cement were considered.

1.7 Context of research

This research is categorised under ‘concrete technology’ in the field of Civil Engineering.

1.8 Expected outcomes

The aim of this research was to determine the effect of yield stress on the stability of SCCM. To determine this, the following outcomes were investigated:

- The relationship between yield stress and each superplasticiser concentration.
- The relationship between yield stress of each cement and the modified superplasticiser at the established optimum concentration.
- The relationship between the yield stress and segregation of cement paste and mortar with different cements using the blended superplasticiser at optimum dosage.

1.9 Methodology

This research study was conducted using the experimental technique. A suitable concrete mix design played a key role, using multiple methods and calculations from the literature.

All tests, conducted under a controlled laboratory environment, adhered to the ASTM, European Guidelines and SANS standards referenced in the literature, including the apparatus used.

Optimisation of the two superplasticisers was accomplished by evaluating the workability of the mixes. The mini-slump cone test was used as a measuring and evaluating tool for the optimum mix.

The two superplasticisers were then blended in each mix. This was done to investigate any effects this had on the flow properties using the mini-slump cone test.

Stability assessment was the final evaluation method explored. The mini-column segregation test was used to determine the segregation properties of all mixes guided by the ASTM C 1610/C 1610M-06 standards. The bleeding test was conducted according to the ASTM C 940-98a standards by using a 1000 ml plastic cylinder.

1.10 Organisation of thesis

Chapter 1 provides some background about the study by highlighting the key research questions and research objectives.

Chapter 2 presents an overview of current relevant studies relating to the research, emphasising the significance of the study and conclusions drawn from the literature review.

Chapter 3 clarifies the research strategy relating to the testing methods, standards, equipment, measurements and assessments.

Chapter 4 displays a summary of the obtained results.

Chapter 5 analyses and discusses the findings.

Chapter 6 presents the conclusions and recommendations for further research.

CHAPTER 2

LITERATURE REVIEW

In this chapter, relevant investigations pertaining to the stability of self-compacting concrete cement mortar (SCCM) are reviewed. A general overview on the self-compacting concrete and cement mortar focuses primarily on the physical criteria for mixes to be classified as self-compacting material. Challenges related to self-compacting concrete (SCC) manufacturing are highlighted with more emphasis on the mix stability. The physical interpretation of segregation and bleeding are pointed out and existing empirical methods to predict their occurrences are briefly discussed. Factors susceptible to compromising the stability of cement mixes are reviewed. A general overview of rheology of self-compacting concrete is provided and the implications of rheological parameters relating to the stability of cement mixes are reviewed. Available testing methods to assess the stability of cementitious materials are discussed in terms of their effectiveness for determining stability.

2.1 Introduction and background on self-compacting concrete

In late 1989, Okamura and Ouchi (2003) suggested that the main reason giving rise to the interest of developing self-compacting concrete (SCC) was the desire to create long-lasting concrete buildings in Japan.

Even so, SCC became a solution to other challenges that the construction and building industry were experiencing such as compaction through closely spaced and highly congested reinforcement and the reduction of labour and noise levels during placement (Brouwers & Radix, 2005).

Self-compacting concrete, in fact, is one of the most innovative developments in concrete technology. It makes it possible to achieve high levels of fluidity in concrete mixes without adding any water. This can be achieved by adding chemical admixtures, known as superplasticisers, to the mix (Alonso et al., 2013).

From its inception, SCC has been used extensively for minor structural repairs to large quantities in civil construction projects in Asia, Europe and parts of America (Domone, 2006). SCC was preferred for practical advantages during placing over its ability to reduce overall project costs and its flexibility for use in structural elements where the smallest vibrator cannot fit.

The finding on the preferential usage of SCC based on technical advantages was verified by Nikbin et al. (2014): their study indicates that SCC capability to provide robust structures, to eliminate the use of vibrators, to pass through confined reinforcement, and to allow architects to design complicated structures without worrying about defects resulting from workmanship were some of the reasons that SCC use has proliferated in the construction industry.

Given the advantages listed above and its ability to fill up formwork by itself without segregating and pass through congested areas without mechanical assistance, SCC is highly suitable and more advantageous than normal vibrated concrete in several specific applications (Shi et al., 2015).

Much research has been conducted pertaining to SCC worldwide. Artificial aggregates have been used for research on producing lightweight SCC which reduces the current enormous pressure that concrete exerts on formwork and yield a lightweight structure (Kaffetzakis & Papanicolaou, 2016). There are also paste and mortar scale studies, chemical dynamics of cement and admixtures, rheology, and adsorption behaviour studies being conducted as part of the ongoing research on SCC to eliminate unwanted negative effects such as low slump retention, low fluidity and high viscosity when casting concrete onsite (Qian et al., 2018).

2.2 Rheology of self-compacting concrete

Cementitious materials behave as non-Newtonian fluids and many researchers have made an assumption that the materials can be characterised using the Bingham model (Rosenbaum et al., 2019). According to Robert et al. (2018), *rheology* is defined as the study of the flow of matter. In SCC, it is defined as the study of the flow behaviour of cementitious materials which are influenced by a variety of factors such as type and dosage of the superplasticisers. According to Varela et al. (2020), rheometers in conjunction with other tests such as the mini-slump cone are used to determine rheological parameters such as yield stress and viscosity. Rheometers are used to determine rheological parameters from flow curves (shear stress versus the shear strain relationship) of cementitious materials. Constitutive models, such as the Bingham and modified Bingham, predict the yield stress and viscosity.

Yield stress is defined as the minimum stress responsible for the initial flow which results in the deformation of material (Roussel & Coussot, 2005); viscosity *as a* measure of the resistance to flow of a fluid when subjected to a range of shear stresses (Vance et al., 2015).

Rheological properties are important in understanding the behaviour of cementitious materials (Vance et al., 2015). The study suggested that initial particle interactions influence the outcomes of the concrete or cement paste in its hardened state. This is in agreement with the previous study by Bouvet et al. (2010) and a later study by Rubio-hernández et al. (2020) who indicate that the flow behaviour of SCCM or SCC is a direct consequence of rheological properties of the paste within the mix. The study deemed the yield stress of any cement system originated from the paste of that system as all the chemical and physical reactions occurred in that part of the mix.

Abeyruwan (2016) argues that cement paste rheology is what governs the rheology of SCC as the paste carries the solid particles in the cementitious solution. Abeyruwan's study investigated the yield stress of both the SCCP and SCCM, considering any change that occurred in concrete rheology to be a direct consequence of the change of its paste and mortar within the system. Earlier studies by Schwartzentruber et al. (2006) also investigated the rheology of SCC by studying the rheology of the cement paste. These researchers suggest that cement paste rheology is what influenced the rheology of the entire concrete system.

Winnefeld et al. (2007), studying both SCCM and SCCP, suggest that measuring the workability of mortar using only the mini-slump test was inadequate as the stability of the aggregates can influence the slump flow results. Their study recommends that cement paste workability of the same mortar be assessed by rheometer measurements. Asghari et al. (2016) prefer investigating rheology using cement paste as opposed to SCC because paste has fewer variables and so the results are more credible. Their study indicated that rheological properties of cement paste were also applicable to SCC.

Earlier studies by Ferraris et al. (2001), however, warned that the rheology of a neat paste can differ from that of SCC due to different dynamics that aggregates add when producing SCC. Another study by Massoussi et al. (2017) investigated concrete stability and rheology by using SCCP and SCCM to avoid the complexity of using concrete. Their study determined that yield stress obtained from SCCM was much higher than that of the SCCP due to the influence of the addition of sand on the rheology of the paste.

Nunes et al. (2011) insist that rheological properties are mainly dependent on the effectiveness of adsorption between the cement and the admixtures, the microstructure of the dispersed solid particles within the solution and the number of phases in the hydration products. Their study listed the following factors as those which influenced the flow properties of cement paste of SCC:

- dosage amounts of admixtures, other admixtures and mineral additives present in the mix;
- cement surface properties and other variables present;

- batching method and time when the superplasticiser is added to the mix; and
- chemical and structural composition of the cement and superplasticiser.

Kabagire et al. (2019) list similar factors in recent studies, but add that the water-to-binder ratio, aggregate content and grading thereof were also influencing factors. Given the factors listed above, it is clear that rheology of SCCM is a complicated dynamic subject to investigation. Each of the articles reviewed only considered particular elements of rheology and certain factors that affect it. Indeed, no study has investigated *all* possible factors that affect rheology simultaneously to establish precisely how they affect each other. This present research focuses on the effect of the rheological parameters on static segregation of SCCM. However, because of the complex nature of rheology, other factors such as adsorption, thixotropy, hydration, cement phases, and fines content are all significant when dealing with cementitious systems.

He et al. (2017), different from other studies, looked into the fluidity and hydration properties and concluded that the rheology of cement paste is directly influenced by the adsorption process which occurs between superplasticisers and cement particles. The fluidity is increased by the dispersing effect of the admixture when coming into contact with the cement particles. Moreover, these researchers suggest that as hydration advances, reactions occur that cause the formation of larger solid particles from the cement and other particles within the solution. The quantity and rate of formation of these solid particles at a particular time influence the flow of the mix tremendously.

Kaleta and Grzeszczyk (2015), investigating rheology from a slightly different perspective, explain that the hydration products formed from the tricalcium silicate and tricalcium aluminate governed the rheology during the initial period of hydration. Recent studies by Bogner et al. (2020) confirm that the hydration product calcium silicate hydrate (C-S-H) and phases associated with it occur also within 2.5 hours from mixing. The study determined that this contributed to the increase in the yield stress and stiffness of cement pastes.

The aluminate phases influence the rheology in the early stages while the amount of fines present in the mortar component of SCC also substantially influence the rheology of an SCCM as in the study by Westerholm et al. (2008) which suggests that the fines content must be kept precise for each mix design: excessive fines increase the yield stress and water demand while insufficient fines lead to an increase in void content causing viscosity to increase.

2.3 Rheological properties of self-compacting mortar (SCCM)

2.3.1. Yield stress and viscosity

Yield stress is a fundamental factor associated directly with influencing the flow behaviour of cement suspensions, according to Kjeldsen et al. (2006).The yield stress value of cement suspensions are usually estimated by using rheological models due to the complex nature of determining the actual yield stress of the materials (Li et al., 2020).

Viscosity helps prolong the settling rate of aggregates while the setting rate of the concrete still proceeds normally, keeping the mix homogeneous (Tregger et al., 2012). Assessing the rate at which the shear stress increases in relation to the strain of a particular mix, Vance et al. (2015) and Stolz and Masuero (2018) deem viscosity as a significant factor in rheological studies of cement mortar.

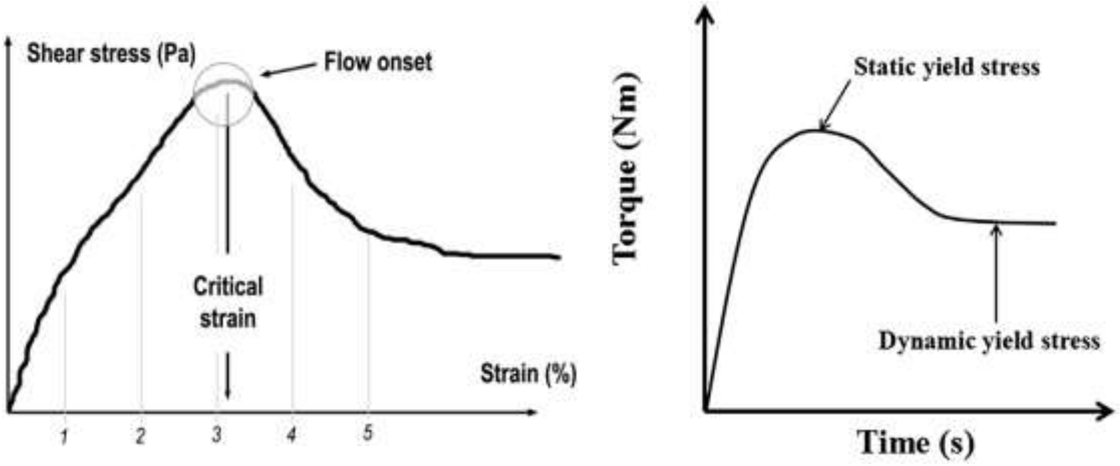
A study by Fernàndez-Altable and Casanova (2006) shows that viscosity is very high at low superplasticiser dosages. An increase in the superplasticiser dosage resulted in a reduction of the viscosity in the mixes. This was later confirmed by Kaleta and Grzeszczyk (2015) as they concluded that cement pastes tend to be less viscous when a polycarboxylate superplasticiser is used as compared to neat pastes. However, the study warned that the time that a superplasticiser is added to a mix is very important and can have a significant impact on the mix and the results.

Furthermore, Aiad (2003) showed that all rheological properties were improved and lower yield stress values were achieved by allowing more mixing time between water and cement to react with each other first before adding the superplasticiser in the solution. This is evident in the results listed in Table 2.1.

Table 2.1: Yield stress and plastic viscosity of different cement pastes at different delaying times after a hydration time of 30 min (Aiad, 2003)

Delaying Time (min)	OPC/MFS		SRC/MFS		OPC/NFS	
	$\tau_{y\ (N/m^2)}$	μ (N/m^2. s)	$\tau_{y\ (N/m^2)}$	μ (N/m^2. s)	$\tau_{y\ (N/m^2)}$	μ (N/m^2. s)
0	13.1	0.71	14.47	0.96	10.2	0.253
5	8.6	0.62	7.1	0.19	7.72	0.176
10	4.7	0.59	5.7	0.2	6.77	0.176
15	6.9	0.47	7	0.14	9.66	0.075
20	5.8	0.6	7.9	0.06	7.83	0.14
25	7.5	0.64	8.49	0.13	7.82	0.14

In Figure 2.1, Vance et al. (2015) describe two ways in which yield stress can be determined, namely dynamic and static. Static yield stress refers to the value of stress that has to be overcome to initiate flow and dynamic yield stress is the stress required to maintain flow. However, the manner in which they are obtained differs. The fact that these two properties differ when measured for the same paste indicates the significant role that the microstructure in solid medium and liquid medium play in the rheology of cement pastes. The cementitious solution is hugely affected by the admixture used, water and cement content (Rubio-hernández et al., 2020).



(a

(b

Figure 2.1: Typical yield stress behaviour for cementitious materials: (a) typical flow onset as a function of strain for a cement paste (Brumaud et al., 2014); (b) variation of shear stress with time (Benaicha et al., 2015)

According to Saak et al. (2001), viscosity values are very high when the stress present in cementitious materials is minimal, but a decrease in viscosity results in the mix starting to flow. Nunes et al. (2011) indicate that the reduction of yield stress is directly related to the improvement of the fluidity of cement paste resulting from the increasing superplasticiser dosage.

But how this reduction in yield stress and viscosity impacts other properties of SCC is critical, as this influences the behaviour of concrete in its fresh and hardened state. Unwanted properties of SCC such as bleeding, segregation and cracks can be influenced by this.

It is understood from Libre et al. (2010) that there are minimum and maximum values of both yield stress and viscosity where a mix will be able to flow sufficiently, while segregation is maintained within an acceptable limit. This is what will ultimately determine the efficiency of yield stress and viscosity in cementitious systems. In their study investigating the relationship between fluidity and segregation of mortars, they found that the stability of the cement mortars was negatively impacted by an increase in fluidity. They further suggest the usage of water reducing admixtures to improve the flow properties as opposed to increasing the water content.

Despite numerous efforts with experiments to manage segregation in SCC systems, maintaining flow and stability in such systems remains a challenge. Abeyruwan et al. (2016) and Tregger et al. (2012) suggest that increasing the viscosity and value of yield stress helps to reduce segregation in an SCC.Their studies concluded that mixes with lower viscosity had segregated more than ones with higher viscosity and that increasing the viscosity and value of yield stress will help prevent segregation when bigger aggregate sizes are present in an SCC mix. Another investigation by Pichler et al. (2017) supports this finding: using the Stokes' law viscometer, they determined that an increase in viscosity will result in a decreased settling rate of particles.

Perrot et al. (2012), investigating the relationship between yield stress and bleeding, found that viscosity had a greater effect on bleeding while no relationship was determined between yield stress and bleeding. Therefore, the ability to investigate and estimate the yield stress of cementitious material will lead to the development of concrete mix designs that will increase the possibility of achieving the intended workability on site (Tan et al., 2017). Mortar plays an important role in this as all aggregates in the suspension system are suspended in the mortar. But more importantly, the segregation of any concrete system is dependent on the rheological parameters of the mortar within the system (Yahia et al., 2016).

2.4 Mortar in self-compacting concrete systems and effect on rheology

In masonry buildings, the quality of the end product is highly dependent and influenced by the quality of the mortar (Haach et al., 2011). In concrete, mortar plays a significant but totally different role. SCC mortar contributes to the decreasing of voids and increasing of strength by occupying the voids between the coarse aggregates, as illustrated in Figure 2.2.

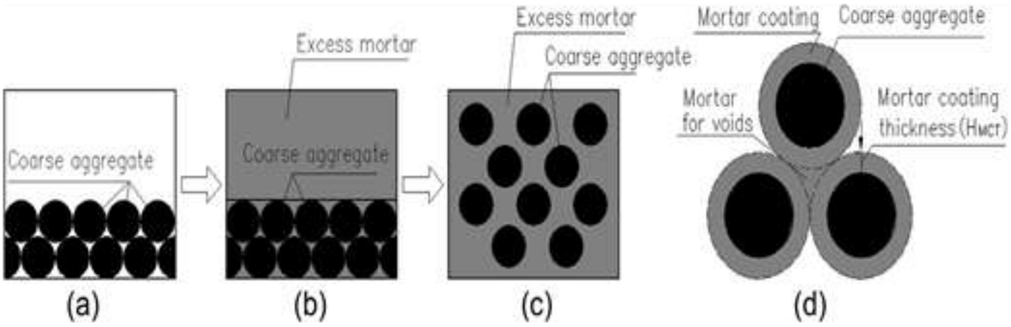


Figure 2.2: Aggregate dispersion model of SCC (Zhu et al., 2016)

Mortar also affects the flow properties because it sticks to the aggregate surface as the concrete flows, providing a smooth contact surface with other aggregates (Zhu et al., 2016).

Compared to the rheology of Newtonian fluids, cement mortars as non-Newtonian suspensions perform differently due to large amounts of different materials, chemical and physical reactions that are present in the suspension (Stolz & Masuero, 2018).

Stolz and Masuero’s study on the influence of the grading of aggregates on the rheology of mortars established that the interaction of the mortar with coarse aggregates was what positively impacted the flow of the mix. Figure 2.2(d) clearly indicates that the rheology of concrete is influenced by the integration and quality of aggregates while all the chemical reactions occur inside the cement paste.

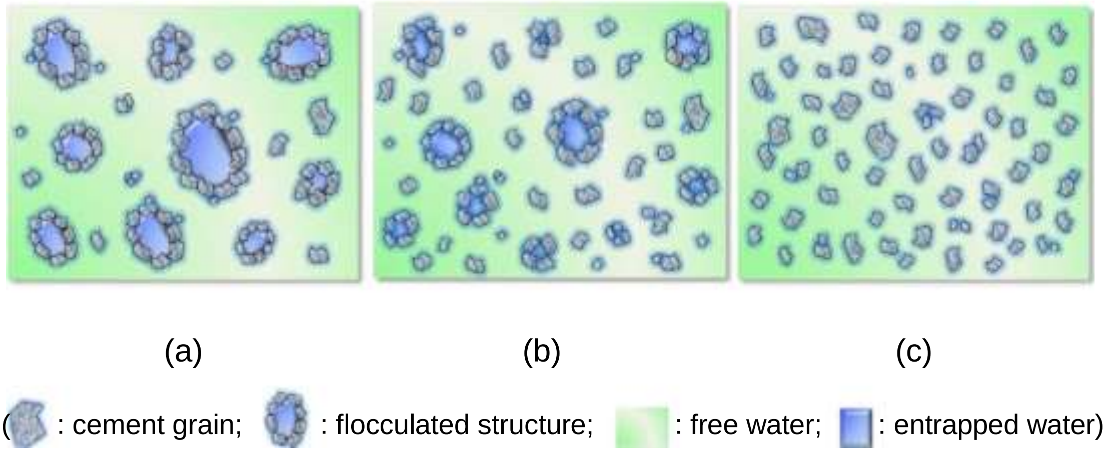
Cement mortar is thus commonly used for assessing the rheology in concrete systems because it is a colloidal solution, hydration occurs and it consists of sand particles which substitute as the stone content (Qian & Kawashima, 2016). Other researchers have also emphasised the importance of mortar in SCC as it makes up a large quantity; mortar, therefore, has a great effect on the rheology and stability of SCC.

Rubio-Hernández et al. (2013) confirm that the yield stress of mortar is the key influencing factor on the stability of the mix; viscosity plays a key role in preventing dynamic segregation. In addition to this, the ability of SCC to maintain great flowability and stability simultaneously is governed by the rheological properties of the mortar content within the mix (Yahia et al., 2005). A similar suggestion was made by Benabed et al. (2012) who indicate that the workability of self-compacting concrete and its ability to flow without segregating is dependent on and determined by an adequate mix design and altering of the rheological properties of the mortar within the mix. Evidence supporting these authors emerged from the study of Zhang et al. (2019), whose results showed that when the viscosity in the mortar is reduced to minimal values, the concrete system weakens, the mortar struggles to keep the aggregates in suspension as they settle rapidly and the mortar ultimately loses its stability.

Debates tend to arise regarding the relationship between mortar and paste or concrete and mortar studies of SCC as there are many variables to be considered. Correlation studies between mortar and concrete rheology, undertaken by Paiva et al. (2015), established that a strong correlation exists between concrete and mortar rheology with the quality of sand particles and quantity of water used greatly influencing this. Furthermore, the authors concluded that concrete rheology is dependent on the performance of the mortar. This was in accordance with earlier studies by Rubio-Hernández et al. (2013) who suggest that cement paste rheology is sufficient for estimation of cement mortar rheology and the same principle applied to the estimation of concrete rheology from mortar rheology. This suggestion, however, can only be considered based on the materials tested but cannot be made for all SCC mixes as different superplasticisers react differently with different materials, especially when related to rheology.

2.5 Effect of superplasticisers on rheological properties of SCCM

Superplasticisers are designed to disperse the solid particles and delay the hydration process when added to the cement solution, as shown in Figure 2.3(c). They affect the rheology of cement solutions by their ability to disperse cement particles through the solution while simultaneously being adsorbed onto these particles (Łaźniewska-Piekarczyk, 2013).



(a) (b) (c)

(▨ : cement grain; ▨ : flocculated structure; 🟩 : free water; 🟦 : entrapped water)

Figure 2.3: Schematic illustration of the effects of NSF and PCE superplasticisers on the microstructure of FCPs: (a) blank FCP; (b) FCP with NSF; & (c) FCP with PCE (Zhang & Kong, 2015)

Gołaszewski and Szwabowski (2004), investigating the influence of superplasticisers on the rheology of mortars, concluded that each superplasticiser had a distinctive effect on the rheology of mortar for each cement type used, even at the same dosages. This was in agreement with the findings in a later study by Antoni et al. (2017), whose study concluded that each type of superplasticiser had different effects when used with different types of cements, attributed to, amongst other factors, the difference in chemical composition of both cement and superplasticisers.

This makes cement and superplasticisers the key influencing factors affecting the fresh and hardened properties of SCCM or cement paste as these directly impact the yield stress and viscosity which also influence the stability and strength of cementitious systems. In cement mortar systems, high yield stress and viscosity in some cases exist because of the huge quantity of fines that are present in the mix (Westerholm et al., 2008). Westerholm et al.'s study proved that optimum usage of superplasticisers can improve the rheological properties of SCCM systems.

Contrary to this, an earlier study by Petrou et al. (2000) argued that only a minor improvement in the viscosity is obtained when water reducing admixtures are added to the mix, but excessive yield stress reduction was achieved. Robert et al. (2018) and Benaicha et al. (2019) supported Westerholm et al. (2008) as their results showed that both the yield stress and viscosity were increased when the superplasticiser dosage was decreased, and vice versa. A conclusion can then be drawn that this had an impact on the stability of the concrete because the compressive strength results indicated that the increase in the superplasticiser dosage produced poor results.

Alonso et al. (2013) supported the findings above that the reduction in water content and increased fluidity in cementitious materials due to the addition of superplasticisers improves the rheological properties which results from lowering the yield stress of the mixes. The researchers warn that certain factors affect the performance of superplasticisers when mixed with any cement: the quantity in the mix, the method used to add it in a mix, when it is added to the mix and their chemical and structural arrangements. The researchers raise concern, however, about the challenges that may arise between superplasticisers and cements if tests are not conducted to ensure that a compatible superplasticiser and cement combination is achievable.

This strengthens the case for ensuring that the compatibility materials are assessed thoroughly to prevent unwanted problems in SCC and to ensure optimum performance is achieved by understanding the interaction of materials in SCC systems. But this is also dependent on several other factors as indicated above due to the complex nature of SCC systems which affects their overall performance.

2.6 Factors which affect the performance, interaction and rheology of SCC

2.6.1 Optimisation of the superplasticiser

There is a superplasticiser dosage limit where no change will be evident in the flow of the mix regardless of how much additional dosage is added (Antoni et al., 2017). Similarly, a minimum dosage is required to initiate the flow; any dosage below this yields insignificant effects to the flowability. As a result, trial tests must be conducted to determine the optimum dosage for each superplasticiser with other materials. The study, investigating different superplasticisers to determine the optimum dosage for each one, reiterated that the performance of superplasticisers in concrete was directly influenced by its dosage.

Antoni et al., however, caution that different optimum dosages may exist for different cements due to the different chemical structure of each cement even if the same superplasticiser is used. The dosage demand of superplasticisers on mixtures with Portland pozzolana cement, as an example, is reduced significantly as a result of less adsorption that takes place between the superplasticiser and the cement particles (Antoni et al., 2017).

Li et al. (2020) suggest that an optimum superplasticiser dosage can be achieved in two ways, either at the minimum superplasticiser dosage when the water content is kept unchanged, or when the ideal workability is obtained at the lowest water content.

Contrary to normal experimental tests, Jimma and Rangaraju (2015) used a statistical model to establish the optimum dosage in their study, finding the method useful in the reduction of the number of dosages applied to each superplasticiser to determine the optimum mix which would significantly reduce the number of physical rheological experiments typically carried out to establish an optimum dosage.

2.6.2 Superplasticiser adsorption

Adsorption is a chemical process whereby some superplasticiser polymers are attracted to the cement particles forming new hydration products while simultaneously dispersing the cement particles to produce flowability in a cement system. The rest of the polymers remain floating within the solution without being adsorbed into the cement grains. The work by Flatt and Houst (2001) concluded that superplasticisers in cement solutions react chemically with the hydration products that are formed as a result of hydration, some adsorbed onto the surface of the cement particles and the rest remaining in the cement solution unutilised. The superplasticiser adsorbed by the cement grains was determined to be responsible for the forces that push the cement particles apart during the hydration process. Nevertheless, portions trapped inside the hydration products with water were unable to have this effect. Later studies by Marchon et al. (2019) agreed with these earlier findings, concluding that the adsorbed superplasticisers were the main cause in the delay of cement hydration.

A study by Alonso et al. (2013), focusing on the compatibility of cement and superplasticisers, reported that optimum adsorption occurs when the side chains of superplasticisers are chemically attracted to the cement particles and when the carboxylate concentration in the superplasticiser is high. As a result of this chemical attraction, the superplasticiser particles carry a force which pushes away the cement particles to prevent flocculation. The study also discovered that superplasticisers get adsorbed onto the grain particles of mineral additives in blended cements. Fly ash and blast-furnace slag adsorb a small portion of superplasticisers in cement paste while limestone blended cement results proved to adsorb much more than other minerals. This was confirmed by Dalas et al. (2015) who reported that the calcium aluminium sulphate minerals, and specifically ettringite, is very important in the study of adsorption as it adsorbs more than twice what calcium silicate hydrate adsorbs in a cement paste solution.

According to Zhang and Kong (2015), the delay in hardening and flowability retention that occurs in cement paste is a direct result of the polycarboxylate water-reducing admixtures that are adsorbed on the cement particles and also the portion of it that remains present in the solution. Matsuzawa et al. (2019), investigating the effect on the non-adsorbed molecules, reported that these increase the fluidity of the cement paste but warn that this was dependent on factors such as the chemical structure of the superplasticisers and proximity of particles in the solution. At microscopic scale, superplasticiser polymers are being adsorbed onto cement particles, as shown in Figure 2.4.

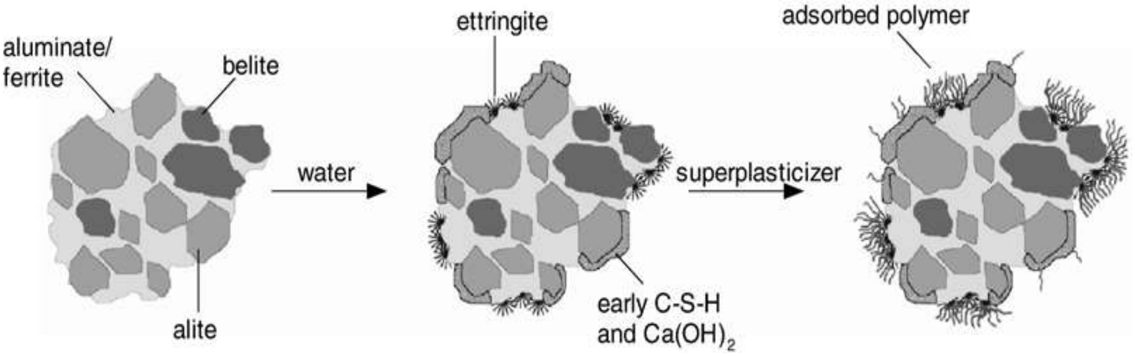


Figure 2.4: Hydrating cement grain with uneven polymer distribution on its surface leading to adsorption (Plank & Hirsch, 2007)

2.6.3 Superplasticiser and cement compatibility

Compatibility is the manner in which superplasticisers perform with cement to enable the mix to achieve ideal rheological and physical properties at minimal superplasticiser dosage while simultaneously maintaining stability (Hallal et al., 2010). Their study looked at compatibility from a rheological perspective. One of the superplasticisers used with Portland cement had better flow results an hour after the mixing compared to the flow results after five minutes, thereby validating their compatibility concerns.

Furthermore, the degree of fineness of cement, the phase structure and the quantity of each phase significantly influence the flowability of SCC. Phases such as tricalcium aluminate and sulphates play a critical role in compatibility with chemical admixtures, according to Alonso et al. (2013). The superplasticiser dosage amount, the method and time of addition to the mix, how the main chains and side chains are structured and chemical structure were determined to influence the compatibility of superplasticisers with cement.

Equally so, the quality of the particles, chemical characteristics, phases present in the clinker and amount thereof were suggested by Alonso et al. (2013) as influencing compatibility between the cement and superplasticiser. These researchers explain that compatibility between cement and superplasticisers is essential to eliminate all problems that could occur, such as undesirable and inconsistent flow properties and unpredictable or unexpected hardening times of concrete. Figure 2.5 is an example of the effect of a superplasticiser on the flowability of cement paste when used with different types of cements.

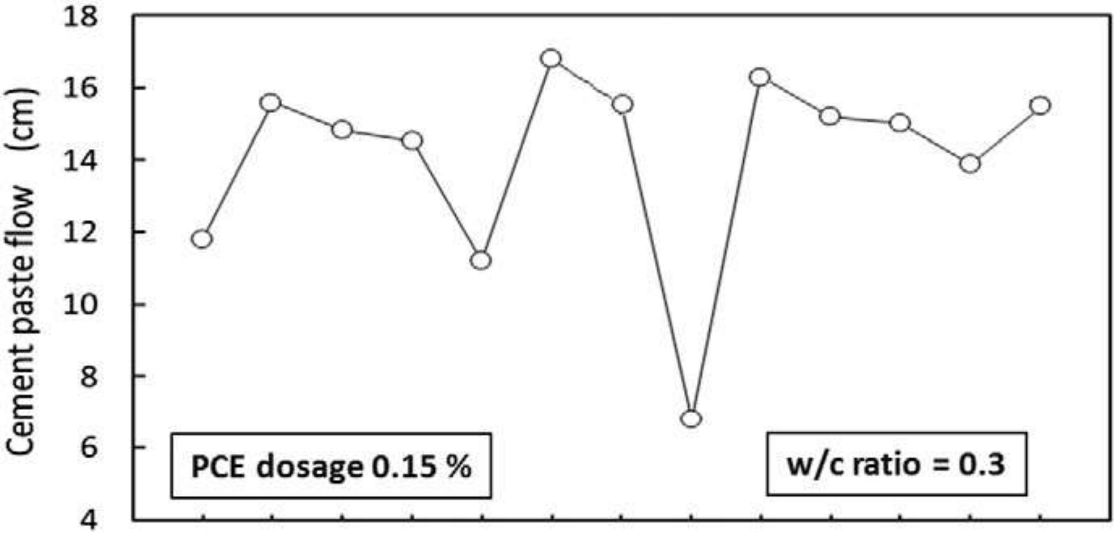


Figure 2.5: Flow behaviour of a PCE superplasticiser with 13 different cements (Plank et al., 2015)

Erdogdu (2000) concluded that the performance between a superplasticiser and cement was mostly dependent on the raw materials present in the cement itself rather than the quantity of cement in the mix design of concrete, while Bahurudeen et al. (2014) suggests that compatibility problems will result in poor properties of concrete in its fresh and hardened state. Therefore, proper selection of admixtures and cement must be assessed to minimise problems associated with incompatibility.

From a chemical perspective after concluding their investigations, the study by Plank et al. (2015) indicated that during the hydration process some of the sulphates present in the mix are dispersed and move freely around the solution. These sulphates were acknowledged as one of the factors of compatibility problems in a cement solution. Chemically, the sulphate molecules have positive and negatively charged ions which then attract and adsorb some of the superplasticiser in the solution. This reduces the cement adsorption which consequently minimises the steric hindrance and diffusion effect that superplasticisers have on the cement particles. Gypsum was also found to be a mineral additive that could improve the compatibility of some superplasticisers with cement if its dosage was slightly increased in the mix (Agarwal et al., 2000).

Material compatibility problems caused by a variety of factors discussed above have proven challenging. A great deal of research has been conducted to resolve incompatibility problems, but investigation must be ongoing as this is dependent on the chosen type of materials.

2.7 Physical interaction of particles in SCCM systems

A study on improving the cohesiveness of SCC by adding ground sand by Ling and Kwan (2015) found that the increase in dosage of water reducing admixtures improves the flow of SCC but causes the mixes to be less cohesive. The paste volume was kept to the minimum and ground sand increased to determine if this could help to improve the cohesion of the mixes. The addition of fine sand particles resulted in good particle interaction between fine and coarse aggregates within the mix which enhanced the cohesiveness and passing ability. However, the sand negatively influenced the flowrate and required more superplasticiser to improve the flowability.

Likewise, the aggregate shape has a significant influence on the mortar rheology. According to Westerholm et al. (2008), fine aggregates sourced from crushed aggregates will generally have a higher viscosity because of the interlocking between the irregular shaped particles during mixing or placing of mortar. The study by Erdoğan et al. (2008) confirmed the important role that the aggregate shape plays in cementitious suspensions, concluding that this will influence the viscosity more than the yield stress. In agreement with previous studies, round particles produce less friction when in contact with each other in a cementitious system resulting in the reduction of yield stress and improvement of viscosity in the mix (Alonso et al., 2017).

Kabagire et al. (2017) also reported that the shape and volume of sand particles affect the packing density in the mortar. This leads to a certain level of compaction during the interaction of particles which impacts the performance and behaviour of the mortar viscosity, which then impacts the stability of the SCCM. Furthermore, Alonso et al. (2017) indicate that concrete studies are designed practically with one size of aggregates while cement mortar comprises different particle sizes within the sand, different shapes and varying degrees of fineness which render rheology studies of mortar more sensitive than concrete.

2.8 Segregation in SCCM systems

Segregation occurs in two forms in SCC: dynamic segregation is the separation of the concrete materials which occurs when concrete is in motion; static segregation is caused by gravitational forces which pull the aggregates and solid particles down by their own weight and density (Shen et al., 2015).

The study by Roussel (2007) confirmed that segregation in SCC is governed and controlled by the mortar and paste mediums. Using polystyrene with cement paste to assess segregation, the results exposed the effect gravity has on cement suspensions irrespective of whether the solid particles have a greater or lesser density than the liquids. This was a significant finding as SCC uses smaller sizes of coarse aggregates as compared to traditional concrete which, according to the study, causes the mix to be highly susceptible to gravitational forces. Roussel indicates that two types of stability problems occur in concrete as a result of these forces, namely segregation and bleeding. Bleeding is the separation of the liquid from the solution and segregation is the separation of solid particles from each other in a mix.

Petrou et al. (2000) showed that normal concrete, contrary to SCC, does not flow and carry the aggregates in suspension during placing by itself, but requires mechanical vibration for this to occur because the yield stress and viscosity values of SCC are significantly lower than those of traditional concrete.

Tregger et al. (2012) considered the ability of SCC to remain homogeneous during mixing, placing and the setting period as one of the main properties that define SCC. Their study considered a mix that is able to distribute equal amounts of aggregate in all the areas of the concrete, as a mix with very good segregation resistance, as illustrated in Figure 2.6.

Their study also notes that increasing the viscosity and value of yield stress will help to prevent segregation when larger aggregate sizes are present in an SCC mix. Viscosity helps prolong the settling rate of aggregates but the setting rate of the concrete will proceed normal and keep the mix homogeneous. The researchers concluded that mixes with lower viscosity segregate more than ones with higher viscosity.

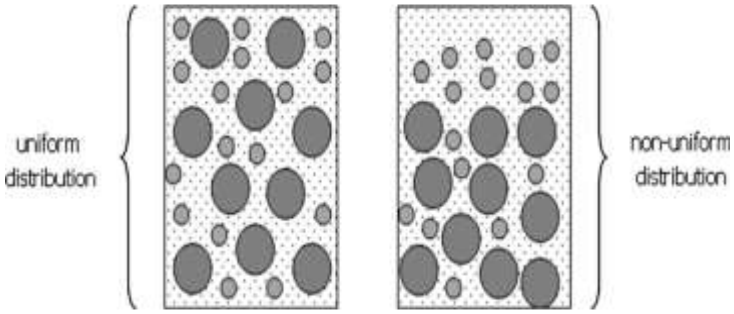


Figure 2.6: Simplified representation of segregation by sedimentation (Benaicha et al., 2015)

Nevertheless, this depended on a variety of factors including but not limited to, size, shape, amount of aggregate, powder content and other rheological related dynamics in a mix. Kabagire et al. (2019) agreed, but deemed all the factors that affect the stability and durability of SCC systems irrelevant if the materials used and quantities thereof are not properly selected and proportioned.

Having considered segregation elements, it is equally important to look at bleeding. Ji et al. (2017) concluded that in cement pastes, bleeding is a direct consequence of how much more or less the densities of the fines present in the mix weighed as compared to the liquid molecules. This is what causes the water to move to the top of the solution. Figure 2.7 demonstrates a typical model for bleed water. Excessive water to cement ratios in mixes are most likely the result of bleeding due to the unnecessary water that is present and moving through the mix. Massoussi et al. (2017) investigated bleeding in cement pastes as well, finding that at the lowest yield stresses bleeding is most likely to occur. The bleeding is reduced by an increase in yield stress, but the study warns that this may also risk a loss of the desired workability for the cement paste.

In accordance with earlier studies, this separation of water from the solid particles can also result in development of settlement cracks and is directly related to the chemical additive dosage amount, water content, mass of cement in the mix and packing density of solid materials (Ji et al., 2015)

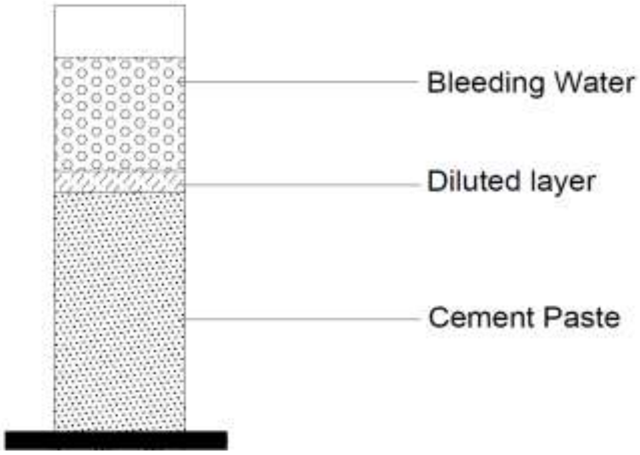


Figure 2.7: Typical bleeding for cement paste in fresh state (Ji et al., 2015)

Perrot et al. (2012), investigating segregation from a mix design perspective, argues that a concrete mix is not classified as SCC if the mix exceeds the recommended segregation limits resulting in the solid particles settling during placing or settling while the mix hardens in the formwork. They emphasise the importance of being able to establish the full extent of the relationship between yield stress and bleeding as it will allow the amount of bleeding in concrete mixes to be predetermined from the yield stress of that particular mix when doing a mix design. As their study found no relationship between yield stress and bleeding, this present study aims to determine the relationship between the rheological parameters and segregation.

2.8.1 Stokes' law

In static concrete systems, Stokes' law describes the settlement of particles as dependent on viscosity and density of the cementitious system, particle sizes and density (Sahai & Moghanloo, 2019). In order for Stokes' law to be valid, the following assumptions are made:

- Particles are to be spear-shaped and smooth.
- The law is applicable to static systems only.
- The settling velocity of a single particle is considered for calculations.
- The reduction in settling velocity is not considered.
- Effects due to inertia and turbulence in fluids are not considered.

Figure 2.8 presents segregation of particles due to Stokes' law.

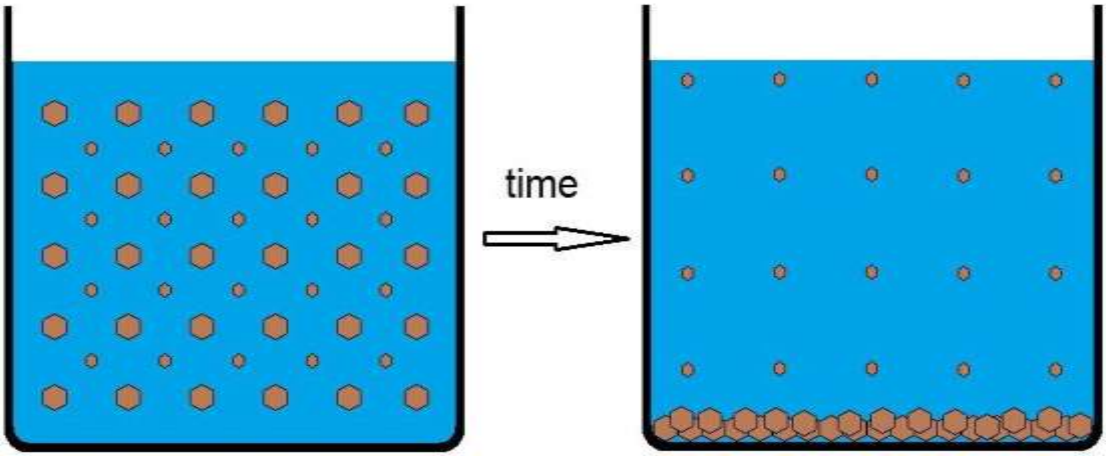


Figure 2.8: Segregation of particles according to Stokes' law (Batsanov et al., 2020)

The reason for this, according to Margarita et al. (2019), is that cementitious materials are comprised of different ingredients which have different densities; these densities play a significant role in the settling of aggregates to the bottom of the suspension and contribute to bleeding. This presents a challenge in achieving homogeneous mixes as the densities, forces, viscosity and velocity within a suspension will influence the static stability, according to Stokes' law.

Stokes' law indicates that viscosity plays an important role in static segregation in cement suspensions. Reducing the viscosity in cement suspensions will likely increase the settling velocity of the particles: the consequence of this will be segregation (Chen et al., 2019). Koehler and Fowler (2007) insist that SCCM viscosity must never be too high to prevent the required flowability or too low as this will lead to undesired segregation. Equation (2.1) presents the settle velocity of particles in a fluid based on Stokes' law.

$$v = Kgr^2 \frac{\gamma_p - \gamma_f}{\mu} \tag{2.1}$$

Where;

v is the settling velocity; K is the shape coefficient (for example, $K = \frac{1}{18}$ for the small spheres), γ is the specific gravity (the subscripts p and f mean particle and fluid, respectively); g is the acceleration of gravity; r is the radius of the particle; and μ is the dynamic viscosity of the fluid.

Stokes' law further suggests that the movement of particles in fluid suspensions is influenced by buoyancy, gravity and viscous drag (Yan et al., 2020). The viscous drag is affected by numerous properties in a suspension including particle size, velocity and the viscosity of that particular fluid. However, since cementitious suspensions behave as non-Newtonian fluids, their viscosity is linked to the plastic viscosity, yield stress and shear rate of its suspension. Therefore, segregation in SCC is also largely influenced by the yield stress as well, because the lower the yield stress the greater the risk of segregation due to the inability of the suspending medium to carry the coarse particles (Yan et al., 2020).

In contrast, traditional concrete viscosity is more critical than yield stress where segregation is concerned (Navarrete & Lopez, 2017). The difference in particle sizes causes the particles to settle downwards in a static system in accordance with Stokes' law.

2.9 Mix design and materials for self-compacting concrete mortar (SCCM)

2.9.1 Mix design

Self-compacting concrete is a mixture that consists of water, cement, sand, coarse aggregates and admixtures. Mix designs are prepared to ensure the desired performance outcomes of a mix and to prevent undesirable effects. When preparing cement paste or mortar, the same principle applies. Rößler et al. (2008) acknowledge the important for concrete manufacturers who undertake the mix designs to be cognizant of how much workability is expected in a mix beforehand when producing SCC and UHPC.

According to Shi et al. (2015), a mix design is the process of selecting the individual materials used in concrete and apportioning them into portions that will ensure that the concrete mix achieves the purpose it was intended for, before and after hardening occurs. Their study explores several design methods for SCC that were developed over the years by other researchers, categorising them by method:

- *Design based on empirical information* – This method uses observations and experience to design the mix. Trial mixes are conducted and the necessary alterations to the mix made.
- *Design based on compressive strength* – The basis of the mix design is the predetermined or required concrete strength and all the other concrete ingredients then get developed from the design strength. Consequently, when modifications are made to the trial mix, each mix component is affected.
- *Design based on close aggregate packing* – The amount or degree of compaction which can be achieved for that particular aggregate is the basis of this method. Once the packing density of the aggregate is determined, the void

content will be known and the amount of paste required to fill these gaps can be calculated. Figure 2.9 shows a flow diagram of this design method.

- *Design based on statistical factorial design* – This is a mix design to ensure that the key properties of SCC are met by taking into account the effects of each concrete constituent. Quantities are determined using the same method for normal concrete to conform to the typical SCC criteria, but more tests are required for the assessment of raw materials.
- *Design based on rheology of the paste* – This method adopts the principle that the stability of a mix and its fresh properties are directly influenced by and dependent upon paste rheology. As this is applicable to a specific aggregate size distribution, minimum values for both rheological properties must be surpassed to prevent any form of segregation.

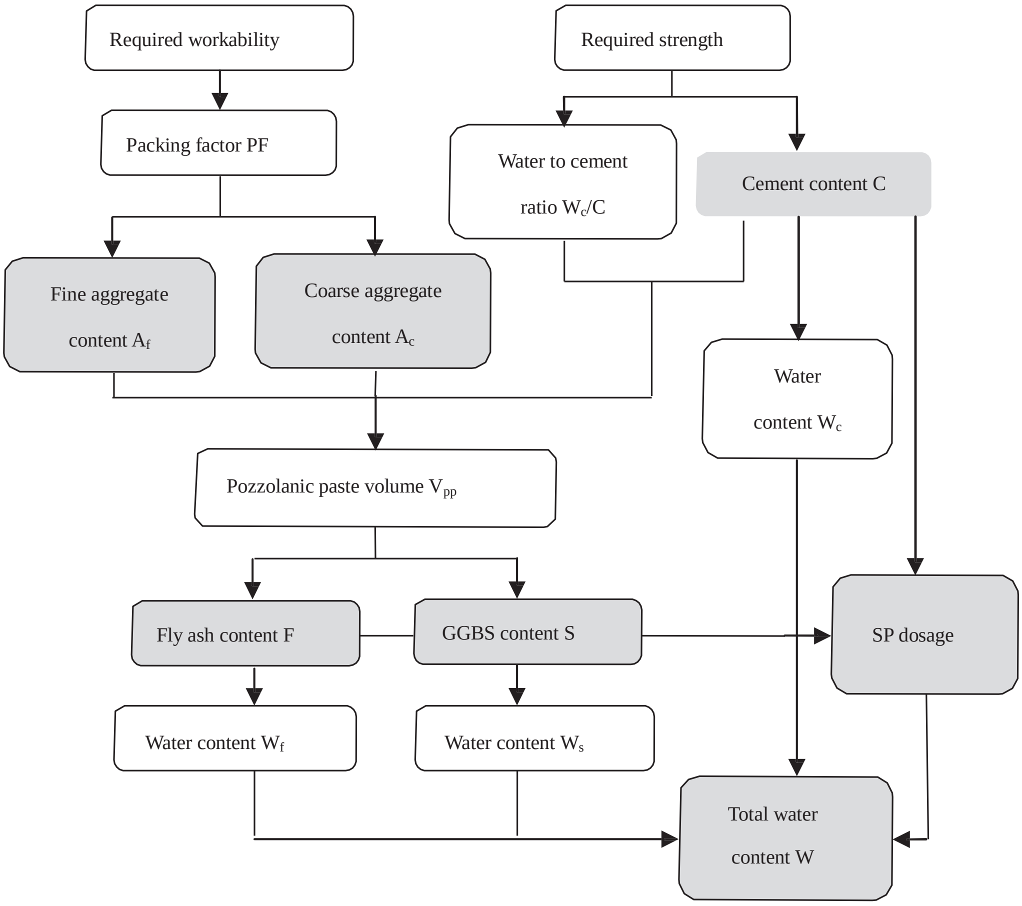


Figure 2.9: Proposed mix design method (Shi et al., 2015)

Saak et al. (2001) conducted research based on a theory that flow behaviour of cement paste will be the key indicating factor of how good the workability of the mix will be and also how well the mix will respond to segregation. Material properties and flow behaviour form the basis for this design approach. It is advantageous because fewer materials are used, fewer experiments are required, it provides opportunity for further research on paste materials and it reduces the number of variables which eliminates sub-standard work.

The European guidelines establish limits and criteria for classification of concrete as SCC meaning that design mixes must fall within a particular range or limits. The guidelines concluded that there are various ways of designing an SCC mix.

2.9.2 Polycarboxylate-based superplasticiser

There is a notable demand for concrete to retain its workability for longer periods during placing while still achieving compressive strength results (Kjeldsen et al., 2006); this encouraged the development of enhanced superplasticisers for concrete mixes. Their study determined that superplasticisers significantly increase the life-span of concrete structures because they reduce the mixing water demand which reduces the porosity of the concrete and thereby enhances the strength.

The aspiration to construct buildings and other concrete structures in minimal time became a reality through the use of admixtures in concrete (Aggoun et al., 2008). Superplasticisers are admixtures that can be used to adjust and regulate the properties of freshly mixed SCC (Burgos-Montes et al., 2012), thus rendering SCC favourable to construct any form of structure.

The study by Janowska-Renkas (2013) discovered that maleic-based superplasticisers with high molecular weight and long side chains indicate good compatibility in terms of performance with cement particles and enhance the flow properties of the mixes. This behaviour of the maleic-based superplasticiser is contrary to the behaviour of polycarboxylate-based superplasticisers as shorter chains and high molecular weight are more effective, as shown by Kong et al. (2016) in their study on the effects of the molecular structure of polycarboxylate-based superplasticisers.

The molecular composition of each type of polycarboxylate superplasticiser varies from one product to another, but a typical chemical structure comprises a main chain and side chains which vary in weight and length (Antoni et al., 2017a).

Apart from the chemical structure, Felekoğlu et al. (2011) credit the exceptional performance of superplasticisers to how efficiently they are absorbed onto cement molecules, the mass of their molecules and their ability to disperse cement particles which initially delays the setting time by preventing the cement particles from agglomerating into large masses. Superplasticisers therefore temporarily delay the chemical reaction between cement particles and water through the adsorption process caused by positively and negatively charged particles, as shown in Figure 2.10.

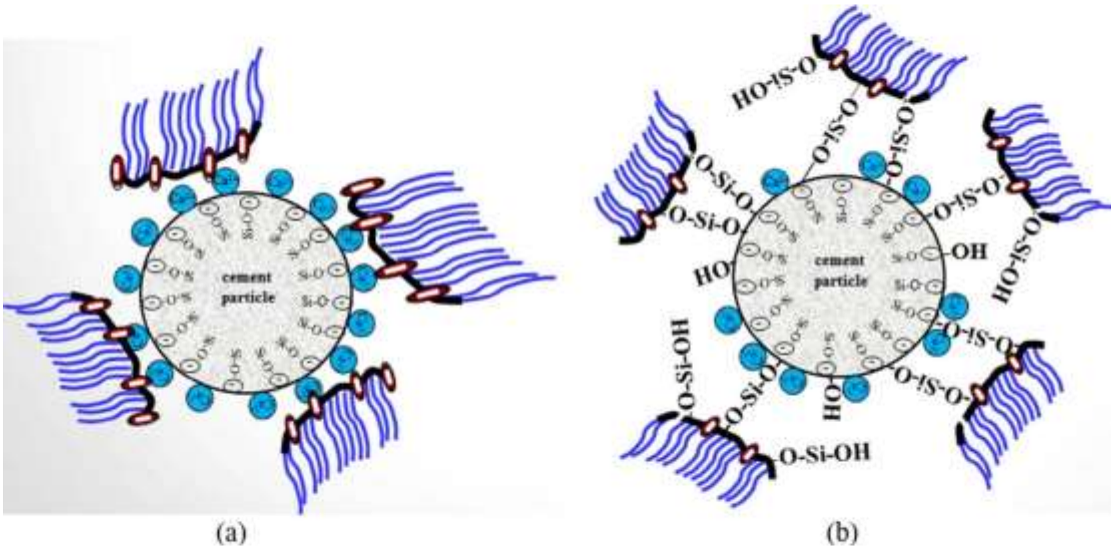


Figure 2.10: Adsorption mechanisms of polycarboxylate superplasticisers: (a) typical polycarboxylate, P(PEG1-AA2); (b) organosilane-modified polycarboxylate polymers, P (HPEG- co-AA-co-MAPTMS) (He, Zhang & Hooton, 2017)

However, with all the good qualities and benefits superplasticisers display, some disadvantages do exist. For example, several different cements were mixed with one superplasticiser and the rheology results were significantly different for all mixes (Plank et al., 2015). This indicates the sensitivity of the chemical reactions between superplasticisers and cement particles. Plank et al.'s study also concluded that superplasticisers do not reach their optimum performance in the presence of clay minerals from the cement particles but still influenced the development of high strength and slump retention of SCC.

2.9.3 Fine aggregate (sand)

A large volume of fine material is required to ensure that self-compacting mortar maintains the required flow and remains homogeneous with no segregation (Benabed et al., 2012). Benabed et al. suggest that sand with a high powder content can reduce the fines content in the overall mix and thereby reduce the total cementitious amount of material in the mix. The cost of using fillers in SCC would therefore be reduced. The study further concluded that an increase in strength and a decrease in void content are advantages of using sands with a high powder content because they provide additional fines to the mix.

Fine sand thus improves the performance of concrete and results in favourable hardened properties (Bonicelli et al., 2015). According to Bonicelli, the performance of fine sand (with particles sizes ranging from 0.25-0.35 mm for type 1 and 0-3 mm for type 2) in pervious concrete demonstrate that the addition of sand produced better quality where disintegration of particles and strength are concerned.

According to Zeghichi et al. (2014), sand also provides a smooth transition between the fine cement particles and the coarse aggregates. Zeghichi et al. determined that round sands require more fine materials to fill the voids that exist because of their particle shape, while sands with irregular shapes affect the workability of concrete. The results proved that dune sand has a greater and positive impact on the rheological properties of the mix as a direct result of the particle shape which resulted in less friction generated between the coarse particles in the concrete mix and the sand particles. The results for the assessment of fresh and hardened state of SCC indicate that a mixture of a fine and coarse sand yields better results.

The source of the sand, robustness, shape, surface characteristics and particle size distribution are factors that influence rheology and strength of SCCM (De Schutter & Poppe, 2004). De Schutter and Poppe argue that the performance outcomes or behaviour of mortar is significantly governed by the kind of sand used in the mix, with the particle size distribution playing a vital role. Their study concluded that both the fresh and hardened properties of mortar are dependent on the type of sand used in a mortar mix.

The impact of sand on the yield stress and viscosity in SCCM was studied by Westerholm et al. (2008) whose study concluded that fine aggregates increase the yield stress of mortar and water demand. Due to the fine aggregate shape, the viscosity of mortar is also increased because of intense friction amongst the sand particles. The study classified the shapes according to their F-value which defined the shape of a particular aggregate according to the results obtained using formulas shown in Figure 2.11 as follows:

- 0 6 ≤ F-value < 0.25: very elongated;
- 0.25 ≤ F-value < 0.50: elongated;
- 0.50 ≤ F-value < 0.75: cubic; and
- 0.75 ≤ F-value ≤ 1: circular.

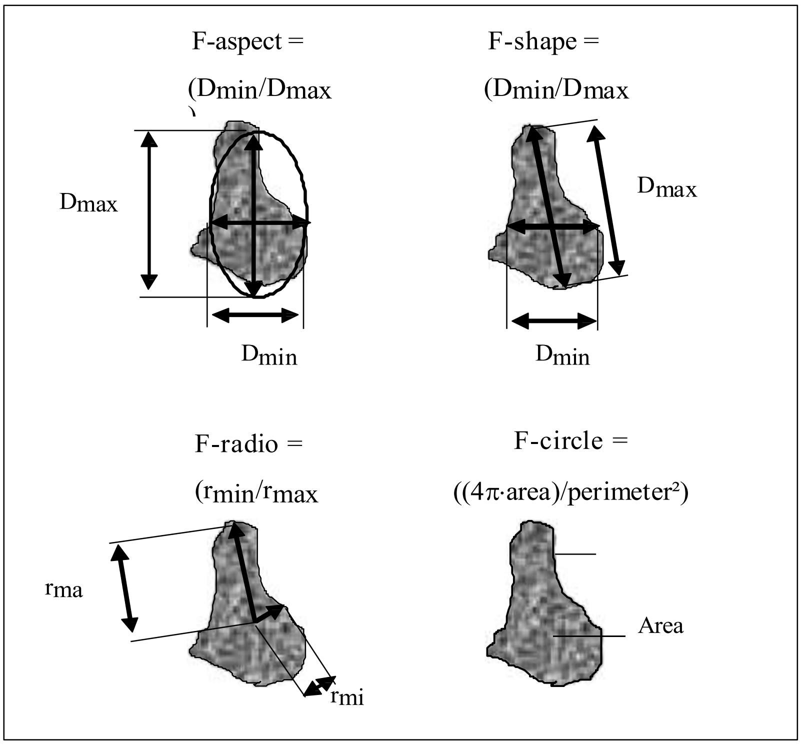


Figure 2.11: Various F-parameters for characterisation of the roundness of particles (Westerholm et al., 2008)

Other alternatives to sand have also been explored in SCC studies. An unconventional sand which is a direct product of crushed rock sediment, known as manufactured sand, is one alternative explored for use in concrete (Nanthagopalan & Santhanam, 2011). This type of sand has a substantial volume of fines which produce undesirable rheology results in SCC, with a high-water demand making it more expensive. Similarly, other environmentally-friendly materials were used successfully to replace sand in SCCM (Safi et al., 2015). Research has used sea shells, for example, to partially and totally replace sand in SCCM with acceptable results when compared to other traditional concrete and SCC results.

2.9.4 Main phases of cement

Wesselsky and Jensen (2009) insist that each individual phase has to be synthesised in a controlled environment in order to clearly understand the specific role and influence each phase has on the hydration process because as readily available cement from suppliers has all phases already merged, this complicates cement related research.

Main phases such as C_3S, C_3A, C_2S, C_4AF and other minor phases such as calcium oxide, periclase, thenardite and arcanite are major contributing elements chemically and physically during the hydration process (Erdoğan, 2013). However, the manner in which each phase is proportioned between the sand and coarse aggregate within the mix will have a significant effect on the microstructure.

Bogue’s equation is often used for the estimation of phases composition of cement in the clinker and widely used and accepted in the cement industry. Crumbie et al. (2006) recommended that Bogues’s equation be used with other methods such as the X-ray diffraction (XRD) analysis with Scanned Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) microanalysis in order to have accurate data on the cement phases. Stutzman et al. (2014) suggested that the calculations come with some form of inaccuracy and their study investigated the degree of these uncertainties by conducting an error analysis. By looking at the effects of the inaccuracy of the bulk chemical analysis and chemical composition, they concluded that a standard deviation of approximately 9.6% existed for the Tricalcium Silicate and Dicalcium Silicate phases while 2.2% and 1.4% for the Tricalcium Aluminate and Tetracalcium Aluminoferrite phases respectively. The following equations are used to estimate these phases;

$$C_3S = (4.071CaO) - (7.600SiO_2) - (6.718Al_2O_3) - (1.430Fe_2O_3) - (2.852SO_3) \qquad (2.2)$$

$$C_2S = (-3.075CaO) + (8.608SiO_2) + (5.703Al_2O_3) + (1.071Fe_2O_3) + (2.154SO_3) \qquad (2.3)$$

$$C_3A = (2.65Al2O3) - (1.692Fe2O3) \qquad (2.4)$$

$$C_4AF = (3.043Fe_2O_3) \qquad (2.5)$$

A recent study by Li et al. (2020) established that calcium silicate hydrate, which is a hydration product, was largely credited to the chemical reactions of the tricalcium and dicalcium silicate phases with silicon dioxide and calcium hydrate in the cementitious solution. Figure 2.12 shows the phase composition of a cement in a study using an SEM and X-ray image.

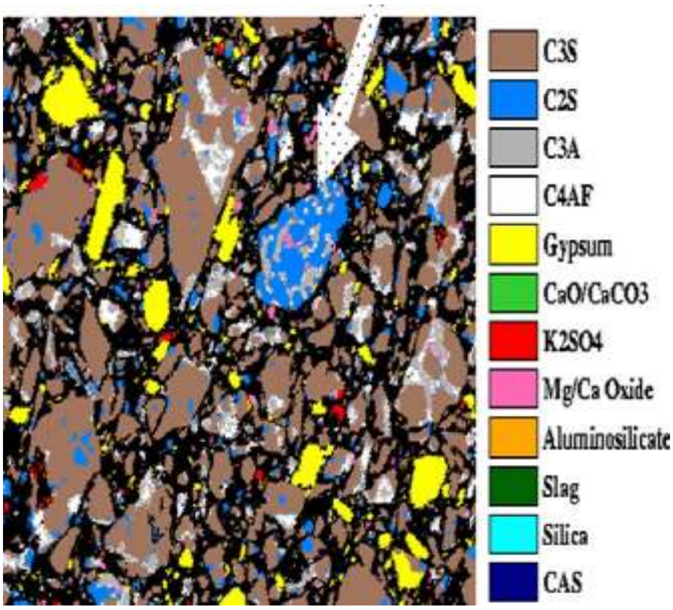


Figure 2.12: SEM/X-ray image of cement used in the study by Erdoğan (2013)

Tricalcium Silicate (C_3S)

In industry, Portland cement is made up of 50-70% tricalcium silicate. According to Stark (2011), there are immediate reactions that occur at the start of mixing followed by reactions at the end of mixing after a couple of minutes lapse during hydration of this phase. Bullard et al. (2011) support these finding and indicate that for approximately six hours after mixing has ended, this phase undergoes minimal reactions in the cement paste solution. Due to its effect on early strength development of cement, Tavakoli and Tarighat (2016) consider it a significant phase while Wesselsky and Jensen (2009) suggest that the triclinic pure form consists mainly of CaO and SiO_2 .

Tricalcium Aluminate (C_3A)

The study by Liu et al. (2015) determined that a greater amount of superplasticisers is adsorbed by this phase and only a maximum of 10% make up Portland cement (Tavakoli & Tarighat, 2016). This phase is regarded as one of the top two phases that play a critical role in the hydration process during the early stages (Bullard et al., 2011). Aluminate has a very high chemical reaction rate which causes cement to harden very quickly so Gypsum is frequently used when manufacturing cement to delay the hardening of cements. CaO and AL_2O_3 are the two main elements used for the synthesis of this cement phase (Wesselsky & Jensen, 2009).

Dicalcium Silicate (C_2S)

This phase makes up to 30% of Portland cement and is the main contributing phase to the strength of concrete from 28 days onwards (Tavakoli & Tarighat, 2016). Wesselsky and Jensen (2009) insist that this phase can collect a small percentage of ions from other phases during the cement manufacturing process in cement plants. In lab environments, it can be produced using silicon oxide and calcium nitrate.

Tetracalcium Aluminoferrite (C_4AF)

Tavakoli and Tarighat (2016) estimate that up to 15% of commonly found Portland cement is made up of tetracalcium alumina ferrite. The study suggests that this phase has a negligible influence on cement strength and plays a less critical role than the silicate phases.

2.10 Yield stress estimation from the mini-slump flow

Roussel et al. (2005), linking yield stress to cement paste spread diameter through an empirical formula, also stress the importance of tension effects in very low yield stress cement pastes. Yields stress of cementitious materials is determined based on the relationship between the cone volume, density of the material and flow spread and consideration of the gravitational force. This relationship can be expressed as follows:

$$V = \int_0^{2\pi}\int_0^{R} h(r)\, r dr d\theta = \frac{8\sqrt{2\pi}\,\tau_y \frac{1}{2} R^{\frac{5}{2}}}{15\,(\rho g)^{\frac{1}{2}}} \tag{2.6}$$

The above was simplified further. Yield stress could be estimated based on the relationship above which also allowed for the prediction of the final flow diameter using:

$$\tau_y = \frac{225\,\rho g\, V^2}{128\,\pi^2\, R^5} \tag{2.7}$$

Despite the ability to determine yield stress using Equation (2.7), it still did not allow for any surface tension effects. However, Roussel and Coussot (2005) argue that surface tension effects can be ignored and Equation (2.7) can be used to estimate yield stress, provided the following condition is met:

$$\tau_y \gg \frac{3\,(1 - cos\theta)\, A_1}{R} \tag{2.8}$$

If the results fail to comply with the above condition, then surface tension effects based on Thomas Young's equation for surface wetting has to be considered and Equation (2.9), based on Figure 2.13, becomes applicable:

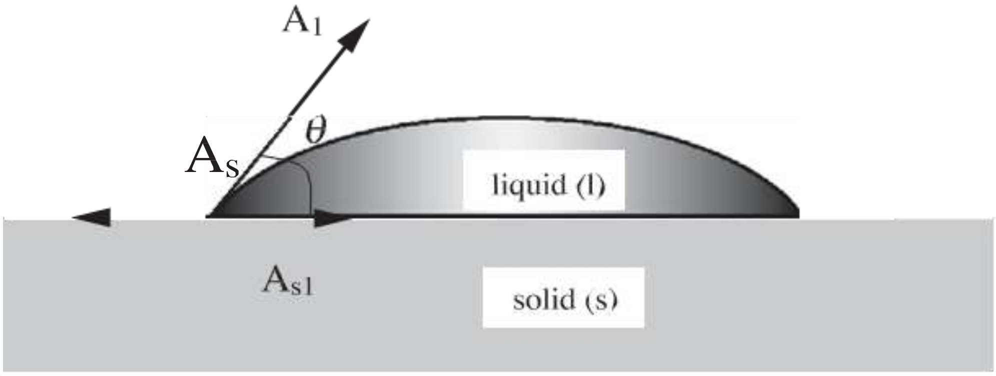


Figure 2.13: Description of the wetting state: A_s is the surface tension of the solid; A_1 is the surface tension of the liquid; A_{s1} is the interfacial tension solid/liquid (Roussel et al., 2005)

$$A_{sl} - A_s + A_1 cos\theta = 0 \tag{2.9}$$

By taking the cementitious material's flow spread and height to factor in surface tension at equilibrium conditions, Roussel et al. (2005) developed Equation 2.10:

$$\pi R^2 h \rho g \frac{dh}{2} + 2\pi R dR\,(A_{sl} - A_s + A_1) = 0 \tag{2.10}$$

If the spread shape is anticipated to have an oval cross-section using Equation (2.9) in Equation (2.10), the stoppage depth could be expressed as follows:

$$h = \sqrt{2\,\frac{A_1}{\rho \mathrm{g}}\,(1 - cos\theta)} \tag{2.11}$$

The depth of the material decreases at a faster rate for cement paste with low yield stress (Abeyruwan, 2016). When taking surface tension effects into consideration, the contact angle can then be estimated by Equation (2.12):

$$\tau_y = \frac{225\,\rho \mathrm{g}\,V^2}{128\,\pi^2\,R^5} - \frac{constant}{h} \tag{2.12}$$

Using the material volume, the above is then simplified to:

$$\tau_y = 1.747\,\rho\,V^2\,R^{-5} - \lambda\,\frac{R^2}{V} \tag{2.13}$$

Where;

ρ *is* density of sample (kg/m³); τ_y is yield stress (Pa); V is volume of cone (m³); R is spreading diameter (mm); λ is coefficient of the fluid and the unknown angle; h is stoppage depth (mm); g is gravitational acceleration (m/s²); ρ is yield stress (Pa); A_1 is surface tension of the material (nM/m); and ϑ is wetted contact angle (ºC).

2.11 Tests for fresh properties of SCCM

2.11.1 Rheometer measurements

Saak et al. (2001) explain that numerous models have been used over the years to determine the yield stress based on the flow curve results obtained from rheometers. According to the authors, the Bingham and Herschel-Bulkley rheological models are the two models preferred by many researchers. The authors indicate that the type of rheometer used and the degree of workmanship when conducting and obtaining the data are fundamental to the authenticity of the results. As an example, the study concluded that the speed of rotation applied in the vane method had a significant impact on the results.

A recent study by Bala et al. (2019) compared methods used to determine the yield stress of SCCP. Using four different rheological models to estimate the yield stress of cement paste, they found that the Bingham model is the preferred model to use because the Herschel-Bulkley model seems to underestimate the actual yield stress of the SCCP while the Modified Bingham model overestimates the yield stress. This was contrary to the earlier studies by Feys et al. (2013).

The mixing device, allocated mixing time, quantities in terms of volume and the addition time of the admixture must remain constant for all mixes (Schwartzentruber et al., 2006). The following conclusions relating to rheology tests are proffered by Vance et al. (2015):

- The distance between the two plates in a parallel plate rheometer reduce the viscosity while intensifying the yield stress in a cement paste mix.
- Plastic viscosity was found to be significantly reduced when the mixing rate was increased as a result of the mixing method used when conducting experiments.
- When comparing the results, the Casson model produced the most accurate yield stress results, but were totally unreliable and out of range to the model criteria. The Bingham model was found to over exaggerate the results obtained for yield stress while the Herschel-Bulkley produced results which were very low.

The study used the three rheology models as shown in Equations (2.14 -2.16):

Bingham: $$\tau = \tau_y + \eta_p \dot{\gamma} \tag{2.14}$$

Herschel-Bulkley: $$\tau = \tau_y + K\dot{\gamma}^n \tag{2.15}$$

Casson: $$\sqrt{\tau} = \sqrt{\tau_y} + \sqrt{\eta_\infty}\sqrt{\dot{\gamma}} \tag{2.16}$$

Where;

τ is shear stress (N/m²); τ_y is yield stress (N/m²); η_p is plastic viscosity (N/m².);
$\dot{\gamma}$ is shear rate (s^{-1}); K is consistency index; n is flow behaviour index; and
η_∞ is the viscosity at an infinite shear rate (N/m²s).

Flow curves are used for the estimation of rheological parameters for cementitious systems. Many researchers have used the downward flow curve data similarly to Ricardo et al. (2020) whose study used shear rates ranging from 0.1 to 100 s^{-1} which are widely acceptable. The study further suggests that the step duration on each point be 30 seconds in order to obtain reliable results. Li et al. (2020) used downward shear rates from 100 to 1 s^{-1} for their investigations of rheological parameters. Bala et al. (2019) used similar shear rate ranges as they deemed the ranges sufficient to obtain reliable measurements of cement paste irrespective of whether the yield stress is high or low. This supported early findings by Vance et al. (2015) who concluded that a shear rate range between 0.1 and 100 s^{-1} is sufficient in obtaining reliable yield stress measurements.

The researchers above all measured their samples at a fixed temperature of approximately 25ºC ±1ºC irrespective of the instruments used for the rheological measurements, except Bala et al. (2019) who adhered to a temperature of 20ºC. According to Vance et al. (2015), who used the parallel plate configuration for testing, the gap between the parallel plates is dependent on the largest particle size in the paste, shear rate range and the serration of the two plates. Their study used a gap of 1 mm citing that increasing the gap above this value results in an increase in yield stress. Ferraris et al. (2001) recommend the parallel plate system only for cement paste rheology measurements due to its variable geometry and used a gap of 0.4 mm for their investigations.

There are, however, no agreed criteria or set standard methods for conducting rheology tests of cement paste and mortars, according to Nunes et al. (2011). These researchers claim that the benefit of testing rheology at cement paste scale is that the methods are not complicated and eliminate the need for excessive material usage (stone and sand). However, the requirement of skilled operators to conduct the tests and the expensive equipment were two highlighted disadvantages. The study further established a direct relationship between the flow spread and flow time results with the yield stress and viscosity results obtained from a rheometer. A later study by Ricardo et al. (2020), investigating different methods of evaluating yield stress of cement pastes and supporting the findings of the relationship between the mini-slump test and the measured yield stress by a rheometer, compared both the rheometer and mini-slump test to evaluate the correlation between the results obtained by the two methods. They caution against the use of the mini-slump test alone to assess fresh properties of cement pastes that have a large variance in flowability.

2.11.2 Mini-slump flow test

Apart from the advanced technology of rheometers, there are other empirical methods that can be used to assess fluidity and flowability characteristics of cement paste, mortar and SCC. Antoni et al. (2017) prescribe the mini-slump cone test as a sufficient flow test to obtain an optimum mix when conducting mortar experiments as an alternative to rheometers. The shape of the mini-slump cone is similar to the traditional slump cone used for concrete, but with much smaller dimensions, as shown in Figure 2.14.

The study by Ferraris et al. (2001) concurs with the researchers above by pointing out that this method is widely available, easily accessible and economically affordable. They warn, however, that the marsh cone and mini-slump tests produce less reliable data when compared to rheometer measurements.

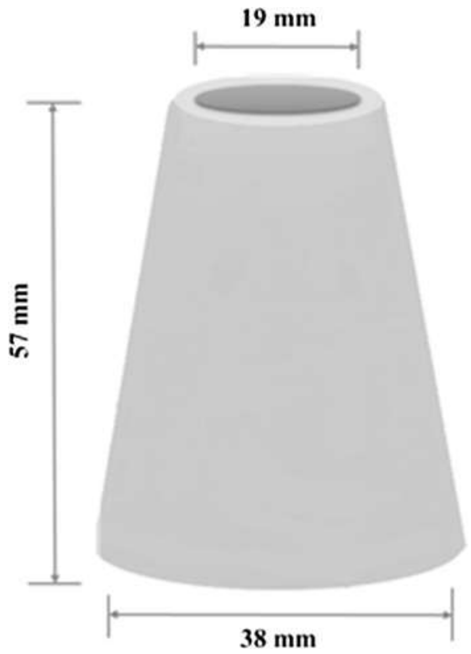


Figure 2.14: Mini-slump cone with dimensions (Tan et al., 2017)

Roussel and Coussot (2005) disagree with this and support the credibility of the mini-slump test. They argue that it is not financially sustainable to have an advanced instrument like a rheometer just for determining yield stress when the mini-slump cone test is reliable, not complex and a cost-effective alternative. Their study, however, suggests that high yield stress values associated with low slump flow need additional factors such as surface tension and contact angle of the materials to be taken into account.

The study by Tregger et al. (2012) supports Roussel and Coussot (2005), showing that this method is useful for visually observing and assessing segregation elements, specifically bleeding. A later study by Kabagire et al. (2019) also determined that a strong relationship exists between the mini-slump of SCCM and rheological measurements of SCC.

The mini-slump test, while the most commonly used method to assess the workability of SCCP, lacks a common standardised method for conducting the test to guarantee credibility of results (Tan et al., 2017).

2.11.3 Segregation

EFNARC (2005) prescribes the sieve segregation test method for determining the amount of segregation in SCC. However, this method is only applicable for concrete mixes and not for SCC mortar or paste mixes.

From 2010, several researchers embarked on studies to establish a method of testing segregation in SCC mortar mixes. Libre et al. (2010), using the mini-column segregation method to assess segregation of mortars with a column model apparatus, found that a maximum SI value of 15% was suitable for SCCM while up to 30% was deemed acceptable, but warn of stability concerns for any SI greater than 30%. Mahdikhani and Ramezanianpour (2015), using the same test, recommended that the SI of SCC mortars be less than 15%. The apparatus used for the test is presented in Figure 2.15.

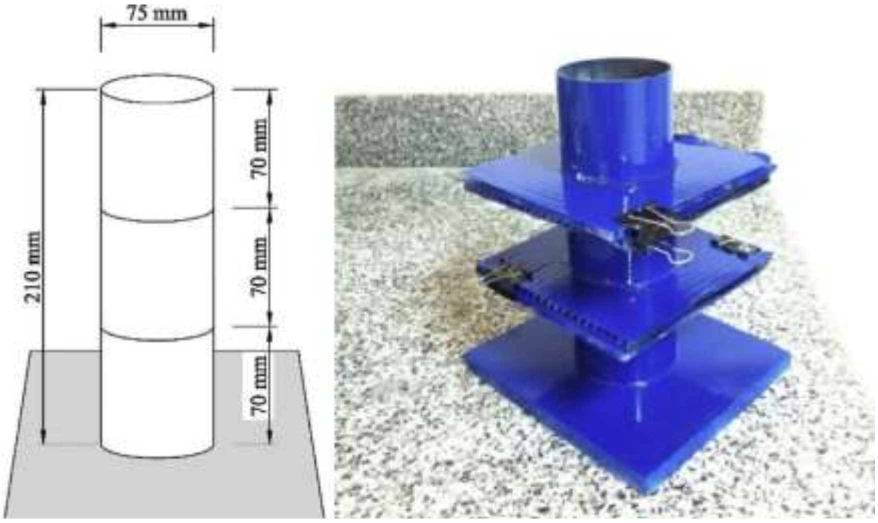


Figure 2.15: Mini-column segregation apparatus (Mahdikhani & Ramezanianpour, 2015)

Libre et al. (2010) and Mahdikhani and Ramezanianpour (2015) focused on static segregation investigations while the aggregate distribution method using the slump flow test was used by Tregger et al. (2012) to determine SCC ability to withstand segregation under flowing conditions. The method sampled concrete from three different areas of the baseplate, sieving the samples to determine the aggregate content in each section. Moreover, this study assessed any signs of bleeding by visual inspection during the slump flow test.

Furthermore, Shen et al. (2015) concluded that dynamic and static segregation need to be measured individually for each mix because they do not always occur simultaneously and therefore, methods of testing the two will always be different. The researchers listed the column, sieve, image analysis and penetration test methods as some methods for evaluating segregation in mixes that are not flowing.

Bleeding is another equally important property of segregation in cementitious materials. The study by Perrot et al. (2012) suggests that bleeding takes additional time and effort to assess. Nonetheless, bleeding is important and cannot be overlooked as it affects the mechanical properties of SCC. A simple method using a cylinder with a predetermined amount of paste to determine the speed of bleeding was carried out by Ji et al. (2017). Measurements of the volume of bleeding water were then taken at various time intervals to determine the bleeding rate and velocity in a mix.

2.11.4 Adsorption test

Absorption is a process where the molecules of one substance enter or penetrate into another substance. Adsorption is the chemical process where the superplasticiser molecules get attached onto the surface of cement particles (He et al., 2017). Adsorption tests are conducted to determine two things: how much of the superplasticiser polymers are adsorbed onto the cement particles and how much is left free flowing in the cement solution. Figure 2.16 illustrates this.

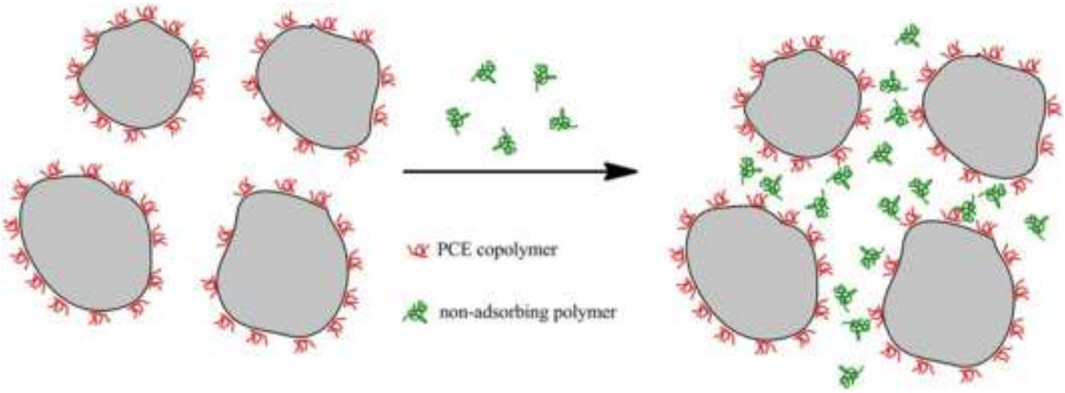


Figure 2.16: Adsorbed and non-adsorbed molecules model (Lange & Plank, 2016)

The superplasticiser that remains floating in the solution helps reduce the friction between the cement particles and thus contributes greatly to the fluidity of the cementitious system (Lange & Plank, 2016). However, the efficiency of this is dependent on the water-to-cement ratio of each mix. The study used the depletion method and the total organic method (TOC) to evaluate adsorption.

Perrot et al. (2012) indicates that cement also contains some carbon. Due to the presence of these small amounts of carbon in the pure cement mix, when doing the TOC tests, Zhang et al. (2015) advise that this very small amount should be factored in when calculating the adsorption of superplasticisers on cement particles.

This research used two superplasticisers, blending them together to form the superplasticiser used for testing and investigation. According to Bessaies et al. (2014), the inadequacy of the TOC test to distinguish the amounts adsorbed on the cement from each admixture when two different polymers are used in a single solution is one of its limitations. They concluded that adsorption competitiveness arises in similar cases between the different admixtures.

The TOC appears to be the popular and most frequently used method to determine the amount of adsorption.

2.12 Conclusion

The main focus of this study was on the rheology and stability of SCC mortar. The microscopic chemical and mechanical reactions which occur in SCC mortar mixes are however what will influence the outcomes of these two properties in the fresh and hardened state: from the adsorption process, which is highly dependent on the chemical composition of the superplasticiser, to the hydration reactions, which are governed by a variety of factors especially the phase composition of the cement. These factors and others will impact the overall stability and rheology of SCC mortar.

This research conducted mortar and paste scale tests. Based on the literature, most researchers deemed SCCP and SCCM tests as accurate, reliable, economically beneficial and assisting in eliminating compatibility issues in SCC, as shown in Table 2.2, which highlights some of the key findings from the literature.

Table 2.2: Summary of some key elements relevant to this study

Description	Author(s)	Year	Summary
SCC, SCCP and SCCM rheology	Bouvet et al.	2010	Flow behaviour of SCC is a direct consequence of the rheology of SCCM and flow behaviour of SCCM is governed by the SCCP within the mix
	Rubio-hernández	2020	Flow behaviour of SCCM or SCC is governed by the rheological properties of the SCCP
	Abeyruwan	2016	Cement paste rheology is what governs the rheology of SCC in cementitious solution
	Schwartzentruber	2006	Cement paste rheology is what influences the rheology of the entire concrete system
	Asghari et al.	2016	Rheological properties of SCCP are accurate and relevant to SCC
	Ferraris et al.	2001	The rheology of paste can differ from that of SCC due to different dynamics that aggregates add
	Massoussi et al.	2017	Yield stress of SCCM was extremely higher than SCCP due to the influence of the addition of sand
	Paiva et al.	2015	A strong correlation exists between SCC and SCCM rheology
	Rubio-Hernández	2013	SCCP rheology is sufficient for estimation of SCCM rheology and SCCM rheology for SCC rheology
Mini-slump Test	Winnefeld et al.	2007	Suggested that rheometers be used with the mini-slump for SCCM as the stability of the aggregates can influence the slump flow results

	Roussel et al.	2005	Supported the test and linked yield stress to cement paste spread diameter through an empirical formula
	Roussel & Coussot	2005	Supported the credibility of the mini-slump test
	Abeyruwan	2016	Favoured the testing method and formula used to obtain yield stress from the mini-slump diameter
	Ricardo et al.	2020	Compared the mini-slump test to the rheometer and found a correlation existed between the two methods
	Antoni et al.	2017	Deemed the mini-slump cone test as being a sufficient test as an alternative to a rheometer
	Ferraris et al.	2001	Suggested that the mini-slump test produced unreliable data compared to a rheometer
	Kabagire et al.	2019	Compared the mini-slump test to the rheometer and found a correlation existed between the two methods
Segregation Assessment Test	Libre et al.	2010	Used the mini-column segregation method
	Mahdikhani & Ramezanianpour	2015	Used the mini-column segregation method
	Tregger et al.	2012	Used the aggregate distribution method
	Perrot et al.	2012	Used the cylinder method for bleeding assessment
	Ji et al.	2017	Used the cylinder method for bleeding assessment
Adsorption Test	Perrot et al.	2012	Conducted the TOC test
	Lange & Plank	2016	Depletion method and TOC tests
	Bessaies et al.	2014	Conducted the TOC test
Rheometer Share Rate Range	Ricardo et al.,	2020	0.10 to 100 s−1
	Li et al.	2020	0.00 to 100 s−1
	Bala et al.	2019	0.01 to 100 s−1
	Vance et al.	2015	0.10 to 100 s−1
Rheometer Attachments	Vance et al.	2015	Used a parallel plate with 1.0 mm gap
	Ferraris et al.	2001	Used a parallel plate with 0.4 mm gap
Rheological Models	Vance et al.	2015	Used the Casson, Herschel-Bulkley, Bingham
	Saak et al.	2001	Preferred the Bingham and Herschel-Bulkley models
	Bala et al.	2019	Bingham model was the preferred model

The literature shows that the type of sand used in SCC significantly affects the rheological properties, workability and strength, with particle size distribution, shape and size being the major influencing factors of the performance of SCC mortar. Yield stress and viscosity are also affected by the degree of sand fineness due to the water demand, making sand selection an important factor effecting the rheological outcomes.

A polycarboxylate-based superplasticiser was used for this study. Based on the literature review, these are by far the most utilised superplasticisers for SCC. Their performance will be highly dependent on their chemical structure and compatibility with the type of cement used. The most challenging part in SCCM, SCCP or SCC, is finding highly compatible cements and superplasticisers which are able to produce the desired flowability without negatively affecting the stability of the system. When compatibility issues are not verified, performance of the superplasticiser is compromised and segregation is one of the undesired effects which results. Optimisation of materials thus becomes very important because for each type of superplasticiser used with a different cement, a conclusion can be drawn based on the literature that there is a possibility that different optimum values can be obtained for each different cement.

From a mix design perspective, SCC has a range of methods which can be used but must conform to the criteria set in the standards and specifications for SCC. Flowability results obtained from the mini-flow test for a mortar system are beneficial to SCC mix designers and ready-mix suppliers as they have some correlation to those of concrete and give some indication of the concrete properties. This implies the need for a proper mix design for SCC mixes.

Yield stress and viscosity are vital parameters of SCC because they are linked to the flowability and influence the fresh properties which determine the performance of SCC. While Ferraris et al. (2001) question the reliability of mini-slump test, the test has been widely used and validated by numerous researchers. Segregation in mortar mixes are broken down into two categories: static and dynamic. Keeping SCC materials evenly distributed around the cement suspension to achieve desirable results in a hardened state is critical in SCC production (Libre et al., 2010). This can be influenced by many factors including the entrapped water moving upward (bleeding) and separation of aggregates from the mortar or settlement of aggregates due to gravitational forces (segregation). This is why this study focused on static segregation.

The reviewed literature identifies contributing factors to segregation such as the aggregate size, weight of solid particle and water content. Previous research has demonstrated the influence that the plastic viscosity of a suspension phase has on a mono or poly-dispersed system. However, less attention has been given to the yield stress that inter-particles of the suspension phase should experience within the cement mortar system to prevent instability. This study, then, investigated the relationship between yield stress and viscosity with segregation and bleeding, which are seen as crucial characteristics in ensuring the production of high-quality SCC. The purpose of this study was to correlate the rheological parameters to the stability of SCCM to obtain clearer understanding, as it is possible that these parameters can affect the segregation of the mortar system as the system becomes more rigid, which will either prevent or allow the suspended materials to sink through the system.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

This chapter describes the methodology and experiments to acquire and analyse the data. Physical and chemical characteristics of materials used in this research were presented. The sand used for the mortar was optimised to meet the SANS 1083:2017 requirements. The mix design of cement mortars is described. Cements were characterised using an XRF for oxide assessment, the Blaine for finesses determination and superplasticiser details were provided by the manufacturer. The admixtures were optimised by varying their SP content from 0.5%-1.1% at a rate of 0.1% in cement mortar with a constant w/c ratio set to 0.4. The same dosages were used for both SP1 and SP2. The optimum dosage was the concentration of the SP above which the spread values were no longer affected. The two superplasticisers were thereafter blended at the determined optimum dosage in different proportions varying from 0-90% at a rate of 10%. In this research, the dosages of the blended SP3 were expressed in terms of SP2 fraction in order to design mortars with cement paste of different yield stress values.

Testing measurements used in this research were discussed and clearly explained for repeatability. Experimental trials were done firstly at cement mortar level to determine the rheology and the stability of samples. Rheological and TOC measurements were assessed at cement paste scale to determine the relationship between the rheology of cement paste, that of cement mortar and its stability.

3.2 Research design

The tests were conducted in three phases using a 30 MPa mix design that met the required criteria for SCC as set out in the European Guidelines. All tests were carried out in a controlled laboratory environment. The objectives of the study were achieved using experimental research techniques.

3.3 Mix design method

SCC has no standard mix design method, but there are a variety of methods as indicated by the European Guidelines for SCC. This study used the mix design method proposed by Su et al. (2001) which is one of the methods recommended in the European Guidelines for SCC. The method was used as a basic guide to develop the design but certain steps were omitted and other conventional concrete formulas were used to suit this particular study.

Table 3.1 indicates the typical recommended quantities of materials for SCC by the European Guidelines. Table 3.2 displays the mortar mix design used for this study. The cement paste and mortar mix design at optimum dosage are shown in Tables 3.3 and 3.4.

Table 3.1: Typical range of SCC mix composition

Constituent	Typical range by mass (kg/m^3)	Typical range by volume (litres/m^3)
Powder	380 - 600	
Paste		300 - 380
Water	150 - 210	150 - 210
Coarse aggregate	750 - 1000	270 - 360
Fine aggregate (sand)	Content balances the volume of the other constituents, typically 48-55% of total aggregate weight	
Water/Powder ratio by Vol		0.85 – 1.10

Table 3.2: Mix design for each superplasticiser dosage

SSCM MIX DESIGN							
RD			3.14	2.65	2.65		
Units	**kg/m³**						
Mix	**w/c**	**Water**	**Cement**	**Stone**	**Sand**	**Superplasticiser**	**SP Dosage (%)**
SCC M	0.40	183	463	0	922	2.313	0.50%
SCC M	0.40	182	463	0	922	2.775	0.60%
SCC M	0.40	182	463	0	922	3.238	0.70%
SCC M	0.40	181	463	0	922	3.700	0.80%
SCC M	0.40	181	463	0	922	4.163	0.90%
SCC M	0.40	180	463	0	922	4.625	1.00%
SCC M	0.40	180	463	0	922	5.088	1.10%

Table 3.3: Mix design for the cement mortar at optimum dosage using SP3

SSCM MIX DESIGN						
Units	**w/c**	**Water**	**PPC Cement**	**Stone**	**Silica Sand**	**Admixture**
kg/m³	0.40	181	463	0	922	4.163

Table 3.4: Mix design for the cement paste

SSCP MIX DESIGN				
Units	**w/c**	**Water**	**PPC Cement**	**Admixture**
g	0.4	11.7	30	0.09

3.4 Research materials

3.4.1 Sand

Two crystalline silica sands that were manufactured and supplied by Consol Industrial Minerals were blended to obtain a balance in the particle size distribution of the materials, as suggested by Zeghichi et al. (2014). Each sand was supplied in 25 kg paper bags from the supplier already washed and sterilised which simplified the grading analysis. The blended sand had a bluff colour and was inert when mixed with water. The sand used is generally suitable as a construction material and sand blasting amongst other things. However, one limitation is that the aggregate size used (less than 2 mm) is very light as compared to concrete aggregate sizes (approximately around 10 mm and above), which may result in greater segregation compared to the SCCM. Table 3.5 shows the chemical analysis of sand no. 1 and no. 2 respectively and Table 3.6 presents the physical properties of the sand.

Table 3.5: Sand no. 1 and no. 2 chemical compositions

Type	Sand no. 1	Sand no. 2
	%	%
$Si0_2$	99,75	99,62
$Al_2 0_3$	0,07	0,15
$Fe_2 0_3$	0,023	0,034
$Ti0_2$	0,024	0,037
$Zr0_2$	0,005	0,009
Ca0	0,003	0,018
Mg0	Traces	0,002
L.O.I	0,12	0,13

Table 3.6: Sand no. 1 and no. 2 physical and chemical properties

Class	Crystalline Silica
Appearance	Sand
Odour	Odourless
Boiling point	2230 C
Melting point	1703 C
pH	5.59
Density	2,65 g/ml
Coefficient water/oil distribution	No applicable

A sieve grading analysis was carried out. The objective of this test was to determine the particle size distribution of the two sands that were used for this research and to collect materials as predetermined for each sieve size. The study by Haach et al. (2011) deemed this test a significant factor impacting the fresh properties and physical performance of mortars due to particle distribution within the mortar. The particle size distribution of the materials was used as a point of reference because the sand required blending to ensure proper distribution of the particles and also to fall within the acceptable criteria for the grading limits as per Table 1, SANS 1083:2017. The test was conducted as set out in TMH 1 Method A1 with quartering, boiling, washing and drying of sand the only deviations from the procedure.

According to the reviewed literature in Chapter 2, sand particle sizes generally ranged from 0-6 mm for SCC mortar including the study by Benabed et al. (2012) which investigated mortars produced using different types of sands. However, the European guidelines for SCC defined mortar as the component of SCC with fines not larger than 4 mm. The sand used in this study conformed to the European Guidelines for SCC.

According to the grading curves shown in Figure 3.1, the sands fell outside the acceptable grading limits as required in Table 1 of SANS 1083:2013. The graph shows that Sand no. 2 was located above the upper limit indicating the degree of fineness of the material, and Sand no. 1 below the lower limit and partially between the two limits. Through this analysis it became evident that the sand had to be blended to ensure that the sand was used optimally and evenly distributed in terms of fine and coarser sand particles, similar to the study by Zeghichi et al. (2014).

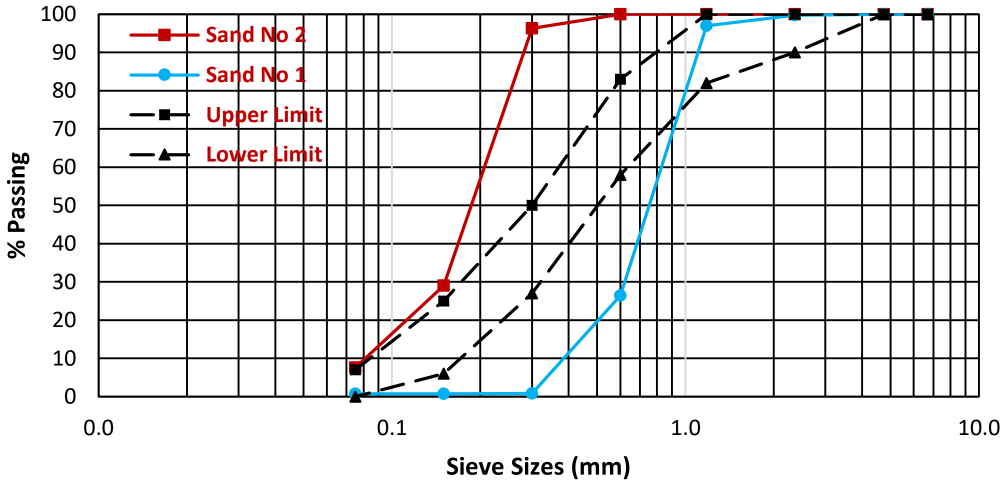


Figure 3.1: Grading curves for sand no. 1 and no. 2 in relation to the grading limits

The two sands were therefore blended with a 50% split for all sand fractions which fell within the grading limits, as shown in Figure 3.2. The procedure and formulas used to determine the ideal combined sand grading curve from the 50% split can be found in Appendix B. Consequently, each mass to be retained in each sieve was pre-calculated; all that had to be done in the lab was to sieve the materials to obtain the required mass for each individual sieve size.

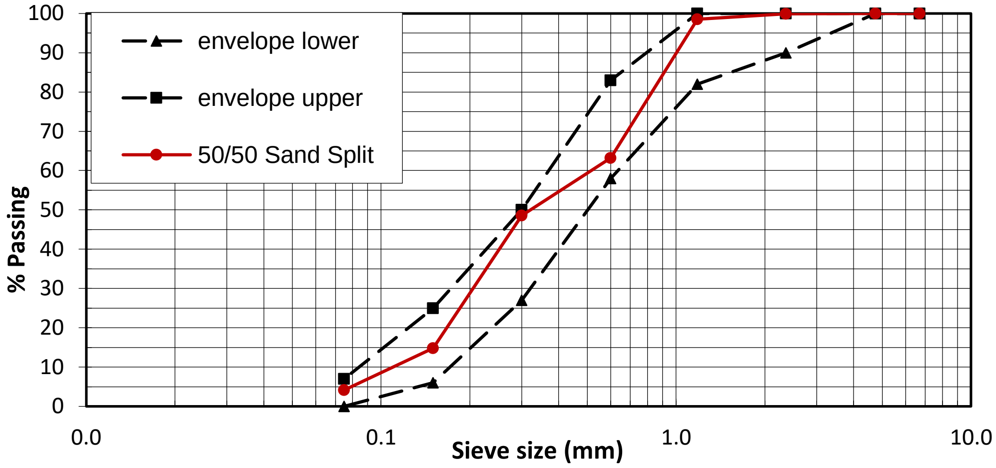


Figure 3.2: Combined sand grading of sand no. 1 and no. 2 in relation to the grading limits

3.4.2 Cement

Table 3.7 displays the chemical analysis of the cements obtained through an X-ray fluorescence analysis (XRF) method and Table 3.8 displays the physical properties for the three different cements used for this research. All cement types used in this study were CEM 1 52.5N with varying mineral and phase compositions that were sourced from Pretoria Portland Cement Company (PPC) but from three different plants. The Bogue equation was used to estimate the Tricalcium Silicate and Tricalcium Aluminate phases.

Table 3.7: Chemical analysis of the cements

PARAMETER	**INFORMATION**		
	Sample Reference		
Chemical Oxides (%)	**CEMENT A**	**CEMENT B**	**CEMENT C**
SiO_2	23.14	20.28	20.40
Al_2O_3	3.28	4.26	4.55
Fe_2O_3	4.43	2.55	2.68
Mn_2O_3	0.13	0.12	0.57
TiO_2	0.41	0.28	0.31
CaO	62.64	64.80	61.79
MgO	1.50	0.91	2.51
P_2O_5	0.07	0.11	0.04
SO_3	2.46	2.45	3.35
Cl	0.00	0.00	0.00
K_2O	0.47	0.67	0.20
Na_2O	0.26	0.16	0.05
LOI	1.75	2.25	3.43
Total	**100.5**	**98.8**	**99.9**
C_3S	50.73	77.37	62.07
C_3A	1.20	6.97	7.52

Table 3.8: Physical characteristic of the cements

PARAMETER		INFORMATION		
Physical Testing		Sample Reference		
		CEMENT A	CEMENT B	CEMENT C
Relative Density		3.09	3.15	3.12
Specific Surface Ares, m^2/g		0.663	0.691	0.748
d (0.1) µm		4.766	4.582	4.365
Standard Consistency, %		28.0	27.6	28.0
Initial Set	Min	180	130	100
Final Set	Min	195	150	120
32 µm Residue, %		15.6	23.2	9.5
45 µm Residue, %		5.3	10.3	2.2
90 µm Residue, %		0.2	0.8	0.1
212 µm Residue, %		0.0	0.1	0.0

3.4.3 Superplasticisers

Tables 3.9 and 3.10 show the two types of superplasticisers sourced from Mapei which were used in this study: Dynamon SP1 (referred to as SP1 in this study) is the older type and costs slightly less compared to the Dynamon SR3 (referred to as SP2 in this study). They were initially used separately to obtain the optimum dosage and later combined at different dosages for the formation of SP3 to see how it would perform.

Table 3.9: Physical and chemical characteristics SP1

SUPERPLASTICISER SP1	
Consistency	liquid
Colour	amber
Density according to ISO 758 (g/m^3)	1.08 ± 0.02 at +20°C
Main action	increased workability/reduction of mixing water and rapid development of mechanical strengths at early ages and at T > 15°C
Classification according to EN 934-2	high range water reducing, hardening accelerating, superplasticiser, Tables 3.1,3.2 & 7
Classification according to ASTM C494	type F and type C
Classification according to ASTM C1017	type I
Chlorides soluble in water according to EN 480-10 (%)	< 0.1 (absent according to EN 934-2)
Alkali content (Na_2O equivalent) according to EN 480-12 (%)	< 3.0
pH content according to ISO 4316	6.5 ± 1.0
Molecular weight	42.000

Table 3.10: Physical and chemical characteristics SP2

SUPERPLASTICISER SP2	
Consistency	liquid
Colour	amber
Density according to ISO 758 (g/cm³)	1.07 ± 0.02 at +20°C
Main action	increase workability/reduction of mixing water and slump retention over long periods
Classification according to EN 934-2	set retarding, high range water reducing, superplasticiser, Tables 11.1 and 11.2
Classification according to ASTM C494	type G
Classification according to ASTM C1017	type II
Chlorides soluble in water according to EN 480-10 (%)	< 0.1 (absent according to EN 934-2)
Alkali content (Na_2O equivalent) according to EN 480-12 (%)	< 2.5
pH according to ISO 4316	6.0 ± 1.0
Molecular weight	40.000

According to the manufacturer, SP1 is used more for precast as it is not a retarder and reacts slower with some cements. SP2 retards setting due to the polymers in the SP. The admixtures were both from the polycarboxylate-based group of polymers which are designed to produce effective steric hindrance when mixed with cement and longer slump retention (Felekoğlu et al., 2011). While both these properties are ideal for SCC mortar, this brought uncertainty to the results as SP1 had longer side chains and a higher molecular mass and SP2 had the opposite according to information received from the manufacturer, as shown in Tables 3.9 and 3.10. Therefore, not only did the different polymers compete in relation to their chain lengths, but their different molecular masses had an impact on the adsorption and was affected by the different phase composition in the three cements. This was verified by Fourier Transformed Infrared Spectroscopy (FTIR) similarly to the study by Janowska-Renkas (2013). One equation was used in two ways to estimate the hydrophilicity of the side chains of SP1 and SP2. Equation (3.1) calculated the hydrophilicity using the absorbance values of the initial intensity over the transmittances obtained from the FTIR readings, while Equation (3.3) used the peak areas to determine the hydrophilicity of the side chains.

$$\text{Hydrophilicity} = A_{ET}^{1080} / A_{ES}^{1640} \qquad (3.1)$$

Where; A_{ET}^{1080} is the absorbance of the second peak of the Ether group and A_{ES}^{1640} is the first peak of the Ester group. Where the absorbance is determined as follows;

$$A = -\log \frac{P_T}{P_O} \qquad (3.2)$$

Where; A is the calculated absorbance; P_0 (%) is the baseline (initial intensity) and P_T (%) is the transmittances.

$$\text{Hydrophilicity} = \text{Area}_{ET}^{1080} / \text{Area}_{ES}^{1640} \qquad (3.3)$$

Where; A is the calculated absorbance; Area_{ET} (%T.cm^{-1}) is the Ether group area of the peak and Area_{ES} (%T.cm^{-1}) Ester group area of the peak. Figure 3.3 and 3.4 shows the infrared spectrum results at wavelengths of 4000 cm^{-1} – 900 cm^{-1} and 2000 cm^{-1} – 900cm^{-1} respectively.

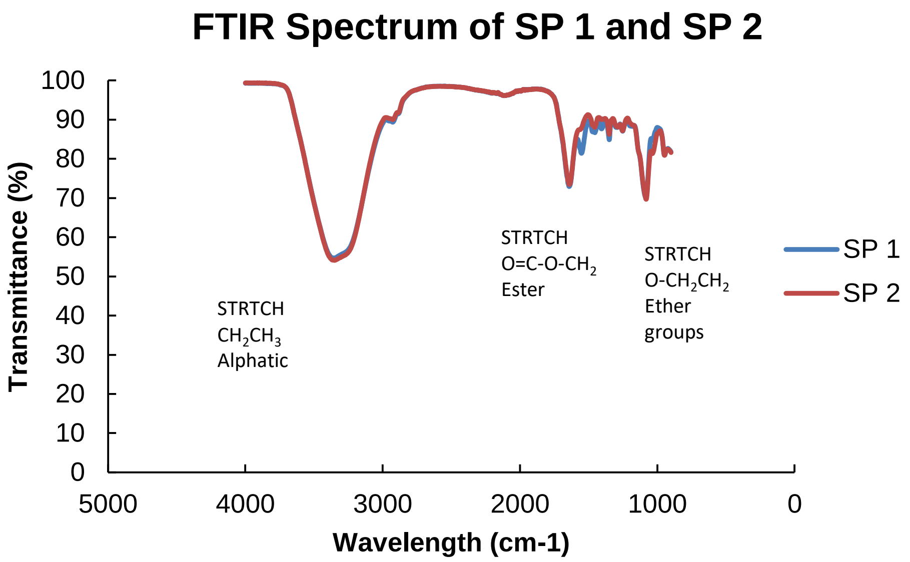


Figure 3.3: Infra-red spectrum of SP 1 and SP 2 (4000 cm^{-1}- 900 cm^{-1})

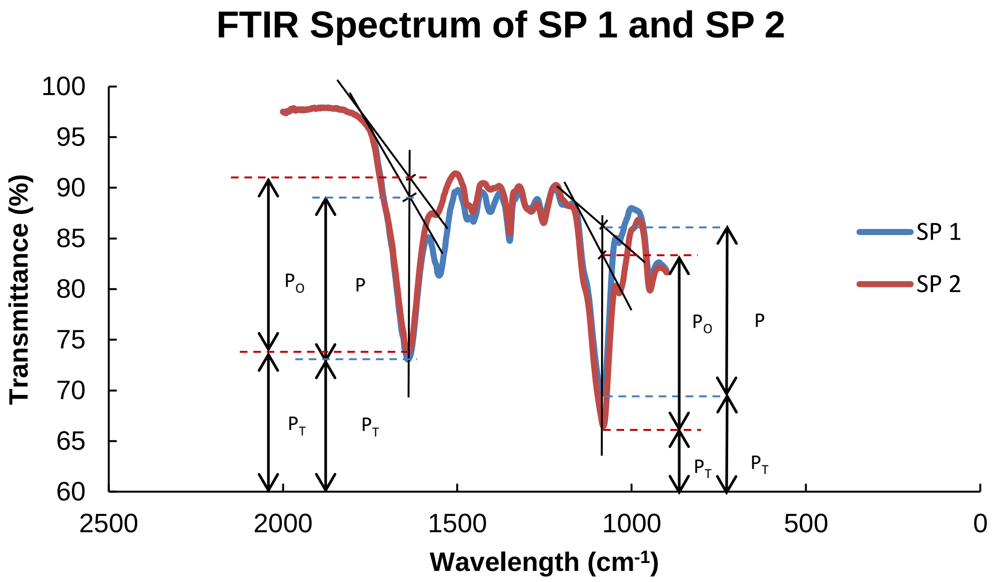


Figure 3.4: Infra-red spectrum of SP 1 and SP 2 (2000 cm^{-1}- 900 cm^{-1})

The results shown in Table 3.11 and 3.12 indicated that the hydrophilicity of the side chains of SP1 were greater than SP2 which was in agreement with the information received from the supplier.

Using Equation (3.1);

Table 3.11: Hydrophilicity of the side chains using the ratio in absorbance relative to a baseline

SP Type	A (First peak 1640 cm^{-1} /Ester group)	A (Second peak 1080 cm^{-1} /Ether group)	Hydrophilicity ($A_{ET}^{1080}/A_{ES}^{1640}$)
SP1	0.086	0.089	1.035
SP2	0.09	0.093	1.033

Using Equation (3.3);

Table 3.12: Hydrophilicity of the side chains using the peak areas

SP Type	Area of peak 1640 cm-1 (%T.cm-1)	Area of peak 1080 cm^{-1} (%T.cm^{-1})	Hydrophilicity ($A_{ET}^{1080}/A_{ES}^{1640}$)
SP1	715.79	1023.68	1.43
SP2	1899.57	910.28	0.479

3.5 Testing methods

3.5.1 Mini-slump flow test

According to Tan et al. (2017), there is no prescribed and agreed standard method for conducting this test. The study highlighted this as one of the disadvantages of the mini-slump test. However, other researchers including Bouvet et al. (2010) have endorsed the reliability of this method. A mini slump cone with 20 mm upper inside diameter (30 mm outer side) and 44 mm bottom diameter (54 mm outer side) and a height of 60 mm was used in this study. The mini cone was positioned at the centre of the baseplate as displayed in Figure 3.5. The sample temperature was measured and monitored to ensure that there was not a huge variance in the temperature of the mixes as this can affect the rheological properties, as demonstrated in the study by Fernàndez-Altable and Casanova (2006).The sample was then poured into the cone and left to rest for 30 seconds to settle. The cone was lifted slowly. The time taken for the flow to come to rest was noted, the two perpendicular diameters were measured and the average diameter recorded. As in the study by Benabed et al. (2012), visual observations for any signs of bleedings or segregation were done.

Figure 3.5: Mini-slump cone on top of baseplate before pouring the sample

The yield stress was then calculated from the results obtained from the mini-slump cone test using Equation (2.7) as set out by Roussel and Coussot (2005). Only four mixes when using Cement B and C did not meet the criteria established by the researchers for the above formula, which meant that surface tension effects needed to be taken into consideration for these mortar mixes. A recent study by Mantellato et al. (2019) determined that the surface tension coefficient could be assumed to be 0.005 for cement suspensions. This study used the suggested value to determine the surface tension effects for these SCCM mixes, but for this study the surface tension effects were ignored due to low negative values obtained when surface tension was included.

Another significant factor was the conclusion reached in earlier studies. According to Roussel et al. (2005), mini-slump diameters less than 350 mm would have negligible surface tension effects and for this study the values were all less than 350 mm.

3.5.2 Mini-column segregation test

The ASTM C 1610/C 1610M-06 standard guidelines were followed as reference for the manufacturing of the column mould, as shown in Figure 3.6. The size of the apparatus and the sieve used for washing the materials, however, were different from the specifications as they were significantly smaller. The dimensions used were in accordance with the measurements specified in the study by Libre et al. (2010).

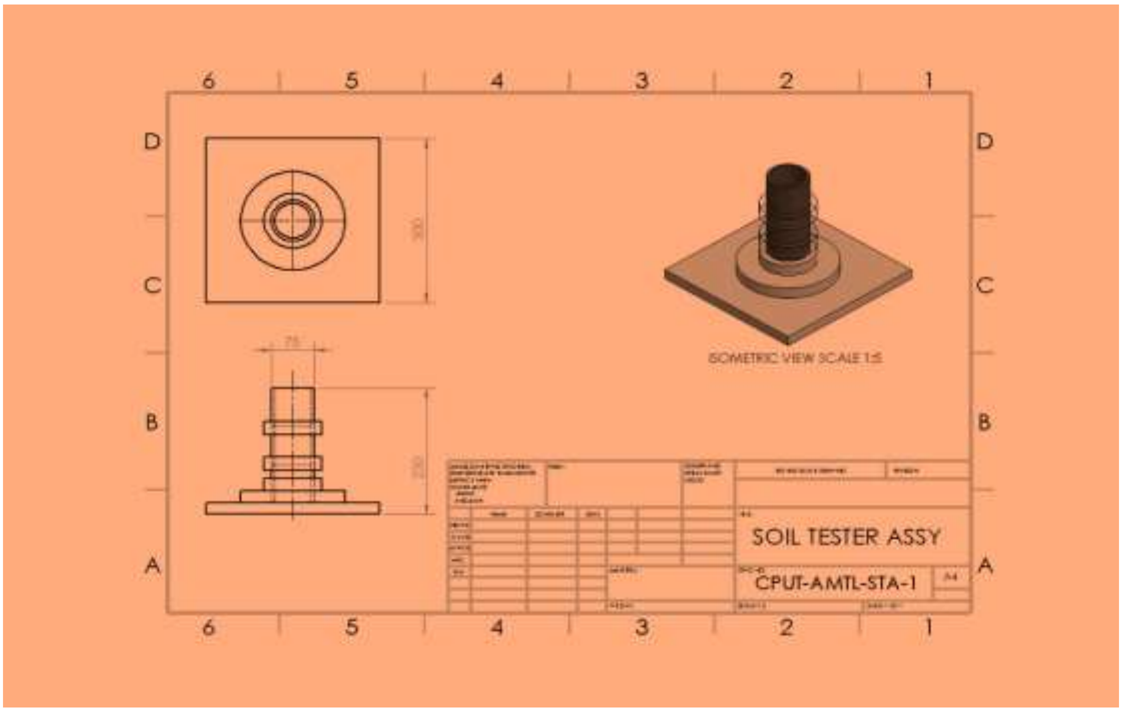


Figure 3.6: Mini-column segregation apparatus design drawings

The mini-column apparatus was assembled, oiled and placed on a level table and the sample poured to full height. The mortar was left for 15 minutes to settle. The top column mould was removed after 15 minutes and washed in a 300 µm sieve for 1 minute, followed by the mortar in the bottom column mould. The material retained in the sieves was then labelled in containers and placed in the oven to dry for 24 hours at 110°C. All the steps after the resting period were done within 20 min as prescribed by the ASTM C 1610/C 1610M-06 standard. The static segregation was then calculated using Equation (3.4), similar to the study by Mehdipour et al. (2013).

$$SI \ = 2\left[\frac{M_{bot} \ - \ M_{Top}}{M_{bot} \ + M_{top}}\right] \times 100 \qquad (3.4)$$

Where; SI is segregation Index (%); M_{bot} is oven dried mass of the top sample (g); and M_{Top} is oven dried mass of the bottom sample (g).

3.5.3 Bleeding test

Ji et al. (2017) and Ji et al. (2015) used a cylinder for evaluating the degree of bleeding in cement pastes. This study used the same apparatus and conducted the test as set out in the ASTM C 940-98a standards. A labelled 1000 ml cylinder was oiled and placed on a level table. Approximately 800 ml (± 10 ml) of the sample was poured into the cylinder. The sample volume and time of pouring were recorded. The top of the cylinder was covered with filter paper to prevent evaporation. Mortar volume measurements were recorded at 15-minute intervals for the first hour and at 60-minute intervals thereafter for three hours from the first reading. The bleed water present in the cylinder was measured at the end of three hours. The degree of bleeding was then calculated using the Equation (3.5) (Ji et al., 2015):

$$B = \frac{V_w}{V_1} \times 100 \tag{3.5}$$

Where; V_1 is volume of the mortar sample at the start of test (ml); V_w is volume of bleed water at the end (ml); and B is final bleeding (%).

3.5.4 Total organic carbon (TOC) test

The chemistry when combining two polymers prompted the study to further explore the adsorption dynamics. The adsorption of the superplasticisers by the cement particles which resulted in the dispersion of the cement particles was the reason for the change in fluidity of the SCCM. The testing process for the total organic carbon in each mix began by centrifuging the cement mortar sample to obtain the supernatant for each mix (Perrot et al., 2012). The objective of the centrifuge process was to separate the solids from the liquid phase in the paste solution to extract the liquid solution for testing the total organic carbon present.

The cement paste sample was mixed by hand similar to Matsuzawa et al. (2019), but for two minutes only, and poured into a labelled centrifuge tube thereafter. The tube was placed in the SIGMA centrifuge machine with another tube with water to balance the machine opposite the sample. The speed limit was set at 5000 rpm and switched off automatically after eight minutes, slightly less than in the study by Perrot et al. (2012) due to the type of machine used. After the machine switched off, the sample was removed and the supernatant was poured into a labelled glass sample container. This procedure was repeated for each mortar sample tested at each superplasticiser dosage. All the sample containers were then placed in a sample holder in preparation for the TOC test.

The supernatants of all mixes, the neat cement paste together with the plain superplasticiser and water solution were then mixed with deionised water to ensure the mixes were within a range the machine could detect (Zhang & Kong, 2015). The samples where tested using the DR 3900 VIS spectrophotometer machine to determine the amount of superplasticiser adsorbed by the cement particles in the cement mortar for each sample. The adsorption was then calculated by the subtracting the amount of carbon left in the supernatant (the amount of carbon in the cement paste sample less the amount of carbon present in the cement only sample) from the original amount of carbon (amount of carbon present in the superplasticiser solution only), as indicated by Lange and Plank (2016) and Zhang and Kong (2015).

3.5.5 Rheometer measurements

An Anton Paar MCR 51 rheometer equipped with two sandblasted parallel plates was used to evaluate the rheological properties of the three cements with superplasticiser SP3 at cement paste scale using an alternative method to the mini-slump test. It is understood that the sand does not react with the water, cement or with the superplasticisers (Rubio-hernández et al., 2020); hence studying the paste rheology was critical. This enabled the study to evaluate the flow behaviour of the paste in relation to the cement mortar tests which were done using the mini-slump test by determining the yield stress and viscosity of the paste. According to the study, the paste within the solution in self-compacting concrete systems is what keeps the aggregates in suspension while flowing under its own weight. An earlier study by Roussel (2007) suggested that the stability of SCC is dependent on its mortar and paste. The aggregates flow within the paste, so understanding the behaviour of the yield stress and viscosity of the paste and how it relates to the stability of the mortar is an essential part of this study.

The cement, water and superplasticiser for a particular sample were weighed in different sample containers. The parameters on the rheometer software were set to pre-shear the samples for 10 s at 100 s^{-1}. The test duration was a total of 300 seconds (150 up and 150 down), measuring for 10 seconds per point. The shear rate for both the downward and upward curves was 0.1 s^{-1} to 100 s^{-1}. These were still within the range adhered to in the study by Asghari et al. (2016) which prescribed any range between 10 s^{-1} and 100 s^{-1} using a similar instrument. The study by Vance et al. (2015) concluded that shear rates from 0.1 s^{-1} and 100 s^{-1} were adequate to provide reliable yield stress results. When the rheometer was fully calibrated, cleaned and ready for testing, the water was added to the superplasticiser and mixed for 30 seconds. The temperature was also set at approximately 25ºC. The liquid mixture was then added to the dry cement and mixed vigorously for two minutes.

After mixing and when the sample was homogeneous, the 50 mm diameter plate was lifted to 75 mm for testing and the sample was placed at the bottom plate using a spoon. The rotating plate was then lowered to 0.6 mm above the sample and the excess paste was trimmed where necessary before commencing the shearing process. This was in accordance with the study by Vance et al. (2015), who suggest that the gap should be significantly greater than the largest grain size of the solid particles used. All cements used had grain sizes not larger than 0.1 mm.

The shearing process was then initiated and the raw data and graphs plotted by the software. The downward flow curve results were used with the Bingham model to estimate the yield stress and viscosity of the SCCP. Equation (2.14) shows the formula for the Bingham model. After testing, the rotating plate was again lifted to 75 mm above the sample and removed for cleaning. The bottom plate of the instrument was also cleaned thoroughly to avoid contamination. This process was repeated for each mix at all dosages for all three cements.

3.6 Mixing procedure

Procedures of mixing cement paste has a direct effect on the rheological properties (Han & Ferron, 2015). It is necessary to ensure that the best suitable method of mixing is used in order to meet their objectives. Two different mixing methods were carried out during this investigation. The mixing procedure used for the paste samples and used for the centrifuge (TOC tests) and rheometer measurements were intensely mixed by hand for two minutes as in the study by Lei and Struble (1997) and Juilland et al. (2012) in a glass container using deionised water, similar to the studies of Han and Ferron (2015) and Ricardo et al. (2020) .

In contrast, for the mini-slump, bleeding and mini-column segregation tests the mixing of the mortar was done using an industrial 10 l capacity dough mixer with a three-speed function. A similar approach was adopted in the study by Bahurudeen et al. (2014), Toutanji et al. (2015) and Ling and Kwan (2015) for mixing. But to ensure there were no delayed reactions and adequate mixing was achieved, this study mixed the superplasticiser with the water at once and extended the mixing times to make the total mixing duration nine minutes.

To obtain SP3, SP1 and SP2 were mixed in varying fractions and left in the laboratory for 24 hours. The cement was poured first into the mixing bowl and then the sand was gradually poured into the mixer, allowing the dry materials to first mix for three minutes at low speed. The superplasticiser and water were also mixed for one minute and left to settle for another minute while the mixer was mixing the dry materials. After four minutes, the mixer was stopped and the superplasticiser content was poured into the bowl. The mixer was switched on for 10 seconds at low speed and then changed to high speed and mixed for three minutes. After three minutes of additional mixing, the bowl was removed and the unmixed cement was scraped off by hand. The mixer was switched on for an additional two minutes and stopped after nine minutes for sampling and testing.

3.7 Equipment and instrumentation

A list of all the equipment and instruments used during the laboratory experiments can be seen in Table 3.13. Compliance to standards was the defining criteria for laboratory equipment and instruments and thus SANS approved sieves were used for the grading test. A mechanical shaker with a timer was used due to the degree of accuracy required for SCCM and the large quantities of materials that had to be sieved to suit the grading limits. An industrial 10 L capacity, three-speed dough mixer (Westerholm et al., 2008) was used for all the tests. For all the mixes and tests, a basic laboratory thermometer ranging from -20 to 100ºC was used to measure the mortar temperature. All tests were conducted at an ambient temperature of 25ºC ± 2ºC. A digital timer was used to record time and separate timers were used for mixing and testing. Electronic scales with calibration certification were used to weigh all materials. Gilson pipettes were used for accurate measuring of the supernatants and other chemicals. A DR 3900 spectrophotometer and a SIGMA centrifuge machine were used for the TOC tests.

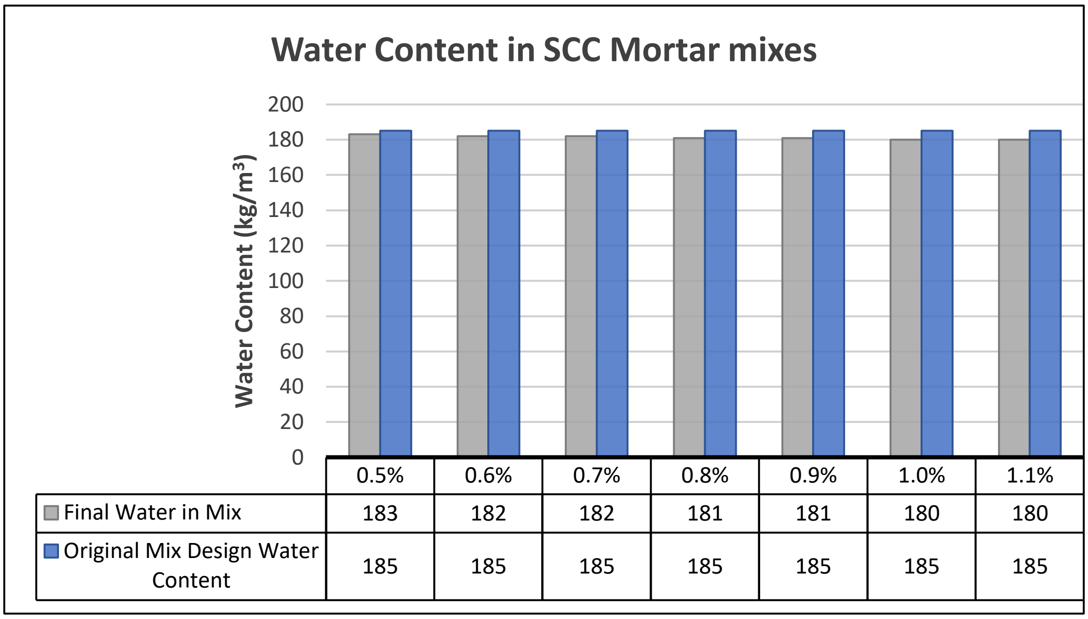
Table 3.13: Apparatus and instruments used for testing

Grading Test	Mini-Slump Flow Test	Bleeding Test	Segregation Test	TOC,FTIR & Rheometer Test
Weighing Scale		Weighing Scale	Weighing Scale	Weighing Scale
	Thermometer	Thermometer	Thermometer	Cylinders
	Mixer	Mixer	Mixer	Reactor
Timer	Timer	Timer	Timer	Timers
Sieve Shaker	Mini-cone	Measuring	Column Mould	Erlenmeyer Flask
Wire Brush	Baseplate	Filter Paper	Collector Plate	Magnetic Stirrer
Soft Brush			0.3 µm Sieve	Pipettes and Tips
Plastic Bags			Oven	Stir Bars
				Lint Free Wipes
				MCR51
				PN Spectrum two
GENERAL ITEMS - Spatulas, bowls, mortar mixer, scooping trowels, bucket containers, cleaning cloths, syringes, marker, test tube rack, glass beakers and small sample containers were general items used in the lab when conducting the tests. Deionized water was used for the adsorption tests.				

3.8 Testing phases

Performance of superplasticisers is highly dependent on the dosage (Antoni et al., 2017) and therefore optimisation of the materials had to occur prior to assessing other properties to ensure that an optimum dosage was used for both superplasticisers with the three different cements. In all mixes the w/c ratio was kept constant and the water only varied slightly in the first phase of the optimising stage because of the water content within the superplasticisers which affected the overall water content, as shown in Figure 3.7. It was, however, kept constant in Phases 2 and 3.

3.8.1. Phase 1: Superplasticiser optimising



	0.5%	0.6%	0.7%	0.8%	0.9%	1.0%	1.1%
▨ Final Water in Mix	183	182	182	181	181	180	180
■ Original Mix Design Water Content	185	185	185	185	185	185	185

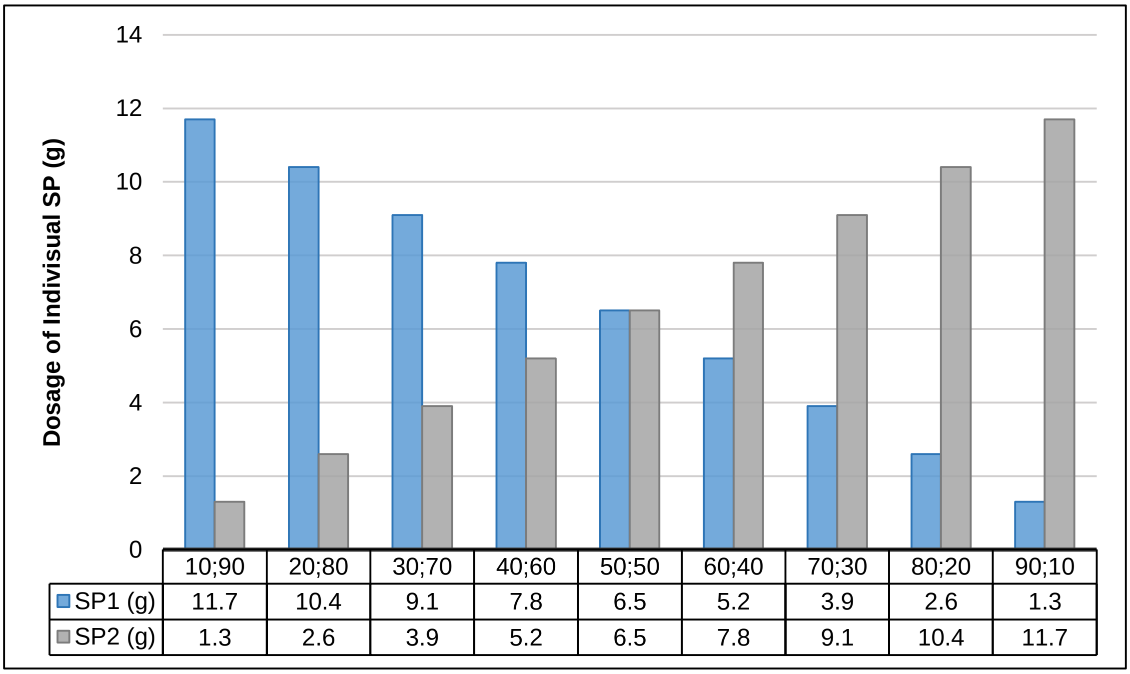
Figure 3.7: Difference in water content after superplasticiser water content has been taken into account for Phase 1

After the grading analysis of the sand, Phase 1 focused on optimising the mix design by preparing mixes for each superplasticiser with each cement. This was in accordance to the recommendations made by Antoni et al. (2017) that an optimum dosage be established for superplasticisers as this directly affects their performance. The lowest concentration was 0.50% which was thereafter increased by 0.1% to a highest value of 1.10%. SCCM using SP1 with each of the three cements were prepared at different superplasticiser concentration, and the same was done with SP2. The mini-slump test was the assessment method for evaluating the flowability and to establish the optimum dosage, similar to the study of Bouvet et al. (2010) and later studies by Robert et al. (2018).

3.8.2. Phase 2: Rheological properties and measurements

Phase 2 used SP3 (a combination of SP1 and SP2 in varying fractions) at the fixed optimum dosage of 0.9% obtained from Phase 1. The dosage portions of each type of superplasticiser in SP3 varied in the overall superplasticiser dosage of 0.9% for each mix. For example, mix 1 consisted of 90% SP2 fraction and 10% SP1, Mix 2 80% SP2 fraction and 20% SP1 etc., but at the fixed dosage amount of 0.9%. This was done to assess the change in rheological properties when the two superplasticisers were mixed at different ratios. The study intended to assist the manufacturer of the superplasticisers to evaluate the performance of combining an older type of superplasticiser with a newer one. According to Bessaies et al. (2014), two different polymers used together in one solution will affect the rheology of cement paste due to the competitive adsorption behaviour of the polymers. For assessment, the mini-slump flow test was carried out for the SCCM and the results of the mini-slump flow were converted to yield stress. Rheometer measurements to obtain the yield stress and viscosity of the SCCP were also done at paste scale to obtain accurate viscosity and yield stress values from the flow curves.

The fraction of each superplasticiser on the overall SP3 dosage amount for each mix is indicated in Figure 3.8.



	10;90	20;80	30;70	40;60	50;50	60;40	70;30	80;20	90;10
🟦 SP1 (g)	11.7	10.4	9.1	7.8	6.5	5.2	3.9	2.6	1.3
⬜ SP2 (g)	1.3	2.6	3.9	5.2	6.5	7.8	9.1	10.4	11.7

Figure 3.8: SP1 and SP2 fractions in SP3 at the optimum dosage of 0.9%

3.8.3. Phase 3: Stability assessment and adsorption

Phase 3 used SP3 with the three cements at varying SP1 and SP2 fractions (Figure 3.8) with the aim of assessing the stability of the mortars at varying yield stress values. SCC is always faced with a challenge of having to produce good flowability without segregating. An over dosage of the superplasticiser could lead to these undesired effects (Antoni et al., 2017). This challenge requires the stability of SCC to be assessed along with other properties.

The mini-slump flow, mini-column segregation and bleeding test were used to assess the fresh mortar properties. The superplasticiser ratio was the only variable in this phase and the dosage was kept constant throughout. The w/c ratio, water content, sand and cement were also kept constant and the stability of each mix was then investigated using the bleeding and mini-column segregation tests.

As a result of the complexity of the cement and superplasticiser chemistry and to attempt to understand and interpret the results obtained, an adsorption study was done using the TOC method.

3.9 Testing sequence for SCCM

As indicated above, a three-phase experimental approach was carried out. Phases 1 and 2 tested the flowability of the cement mortar and paste through the mini-slump test and rheometer measurements. Phase 3 used the mini-slump test to detect any visible segregation in conjunction with the mini-column segregation test and bleeding tests to assess the stability of the mortar (Tregger et al., 2012) while simultaneously assessing the adsorption behaviour of the three cements at each superplasticiser dosage. The mixing procedure was as follows:

- Start mixing – 0 minutes;
- Stop mixing – 9 minutes; and
- Start mini-slump test – 12 minutes.

All the mini-slump cone tests were done between two and five minutes after completing mixing. This had to remain constant throughout and adhered to because if the times extended beyond the indicated times then the yield stress results could also be affected by hydration. The hydration age has a direct impact on the yield stress of cement paste regardless of the superplasticiser dosage (Panchal et al., 2018). On the other hand, the stability testing sequence was carried out as indicated:

- Start mixing – 0 minutes;
- Stop mixing – 9 minutes;
- Start bleeding and segregation test – 10 minutes; and
- Start bleeding measurements, wet sieve washing and oven drying – 25 minutes.

The test times indicated were the planned times, but the actual times varied slightly. The starting time of these tests was between one to nine minutes after stopping the mixer for all tests. Consequently, the start time for the interval measurements for bleeding and washing of materials for the segregation tests varied as these are dependent of the start times for testing.

3.10 Data collection

The sand grading analysis data of the two sands enabled appropriate selection of sand and further allowed the determination of the percentage of material that needed to pass each sieve for the sand to fall within the required grading limits.

The mini-slump cone flow results were used to assess fluidity of the cement mortar and determine the optimum dosage for the two chosen admixtures. Calculations were then done to convert the flow spread to yield stress. The yield stress and viscosity measurements of the cement paste were obtained from the flow curves obtained from the rheometer and applying the Bingham model.

Visual assessment of the mortar mixes from flow spread, bleeding test and column segregation test of each mix were also recorded and formed part of the data as excessive segregation and bleeding which could be seen in some mixes. The column segregation test was used to calculate the segregation index to evaluate the stability of the SCC mortar mixes. The bleeding test simultaneously produced results which indicated the extent of bleeding of each mix at the same yield stress values. The stability of each mix could then be fully investigated based on the data obtained.

Chemical and mineral composition of each cement was also determined by testing a representative sample of each cement. The TOC test provided evidence of the adsorption dynamics of the different cements to support the rheological behaviour of the SCCM.

3.11 Analysis and presentation of results

The optimum dosage was assessed and analysed by the flow behaviour of each mortar mix through yield stress, slump flow and viscosity presented individually as a function of the superplasticiser dosage. Similarly, bleeding was presented as a function of the superplasticiser dosage.

The yield stress, slump flow and viscosity were also presented as functions of superplasticiser SP3 dosage on the SCCM, but presented as functions of the segregation and bleeding for all the mortar mixes for the last phase, which allowed the assessment of their relationship. This was done for each cement type because each cement had a different phase composition. The rheology and stability results for each cement type were then compared to make an informed analysis of the results obtained. The yield stress and adsorption were also presented as functions of the superplasticiser dosage to evaluate the relationship between adsorption and yield stress in the SCCM.

The analysis of results was based on pre-established conformity and criteria used to conduct each test as set out in the European Guidelines, SANS and ASTM standards.

3.12 Experimental flowchart

The testing procedure followed for this study is shown in Figure 3.9.

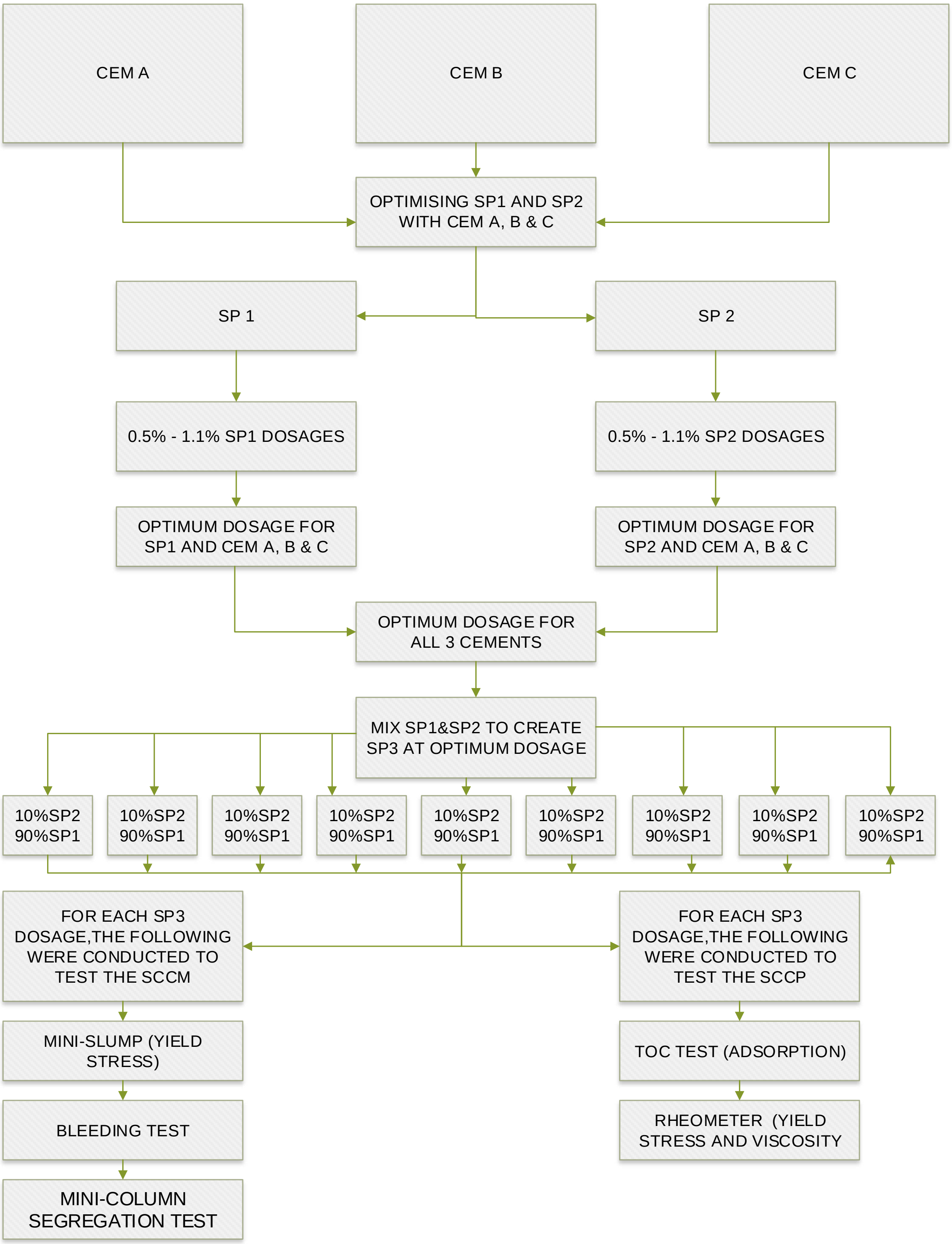


Figure 3.9: Experimental flowchart

3.13 Conclusion

This study investigated the effect of yield stress and viscosity on static segregation of SCC mortar. The tests and methodology discussed above enabled the study to determine this effect for the all the mixes and materials used.

The mix design method was satisfactory and conformed to the criteria established by the European Guidelines for SCC. The methodology used to obtain the sand materials provided good quality control and even distribution of particles for the sand. The mini-slump flow, rheometer, bleeding and segregation tests allowed workability and stability properties to be assessed adequately.

All tests were conducted in a laboratory environment and required two to five people to conducted them accurately, with time keeping, sequence and identical procedure for each mix playing an important role. As a quality control measure, all test results in Chapter 4 were conducted twice and repeated if there was a variance greater than 20% for the stability assessment and 1 cm for the mini-slump tests in accordance with earlier studies by Libre et al. (2010). An average of the data sets was used and an error analysis was also done for the test data by calculating the standard deviation over the square root of the number of tests. All test data is presented in Appendix D.

CHAPTER 4

RESULTS

4.1 Introduction

In this chapter, the results obtained from the previous chapter (Chapter 3) are presented in detail. The research consisted of demonstrating the effect of the cement paste rheology on the stability of cement mortar. Firstly, the effect of the individual superplasticisers on the three cements were assessed to obtain the optimum dosage. SP3 (the product of two superplasticisers) with different fractions of the two superplasticisers at the set optimum dosage on cements was assessed by measuring the spread flow of cement mortar samples. Secondly, the stability of mortar with the blended SP3 product was evaluated. Results pertaining to cement paste microstructure behavior in terms of rheological properties and SP adsorption are presented.

4.2 Sand grading analysis

The grading for the 50% split in quantity of the two sands still needed to be blended further to obtain uniformity throughout, as seen in Figure 3.2. A desired grading curve which fell within the limits and had a fair distribution of all particle sizes available, was plotted and the mass required to be retained in each sieve was determined. The different fractions were separated through sieving and later combined to get the desired quantity of sand for each mix. A uniformly graded sand can be seen falling between the grading limits to conform with the specified criteria for sand as required by SANS 1083. The combined grading curve is presented in Figure 4.1. The grading curve shows that the amount of material needed passing the 0.075 µm sieve was approximately 4%. This number seemed small but these fines affected the workability of the mortar as more water was needed to produce adequate workability in the mixes to compensate for these fines, as suggested in the study by Benabed et al. (2012).

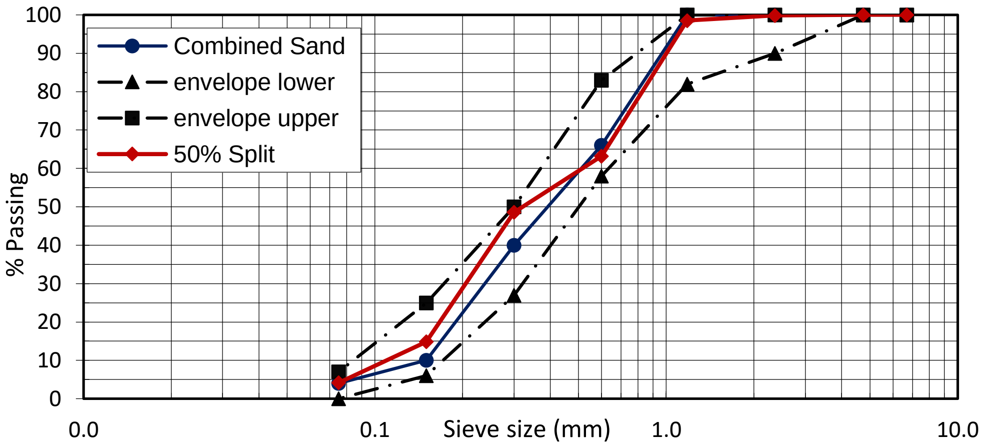


Figure 4.1: Sand grading analysis

4.3 Optimisation of the superplasticiser

This section deals with the optimisation of the superplasticisers. Two different superplasticisers, SP1 and SP2, were used with three different cements which were manufactured at different plants to assess the optimum admixture dosage for each cement. The mini-slump results were used to estimate the yield stress of the SCCM using Equation (2.13).

4.3.1 Effect of SP1 with Cement A on the flowability of SCCM

Graphical representation of the yield stress and mini-slump flow are shown in Figures 4.2 and Figure 4.3. It was observed that the first three dosages, Cement A displayed a steady decrease in yield stress and increase in slump flow with an increase in superplasticiser dosage until an optimum dosage was reached between 0.7% and 0.8%.

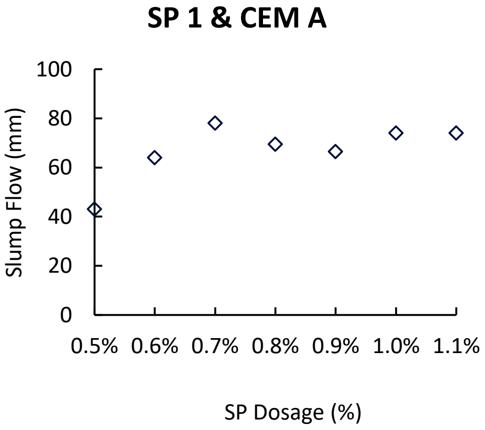


Figure 4.2: Mini-slump flow results

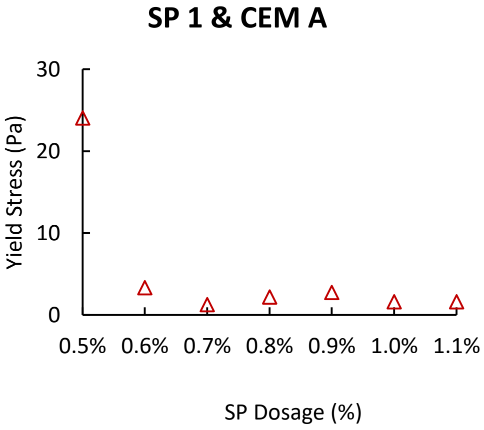


Figure 4.3: Yield stress results

4.3.2 Effect of SP1 with Cement B on the flowability of SCCM

The results obtained using SP1 with Cement B in Figures 4.4 and 4.5 show no significant change in flow or yield stress from 0.8% to 1.1%, which is where the optimum dosage was for this particular cement.

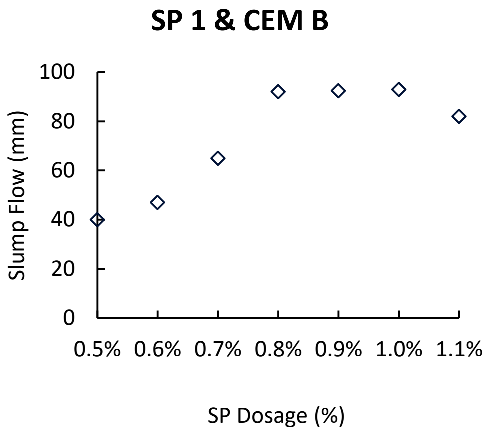


Figure 4.4: Mini-slump flow results

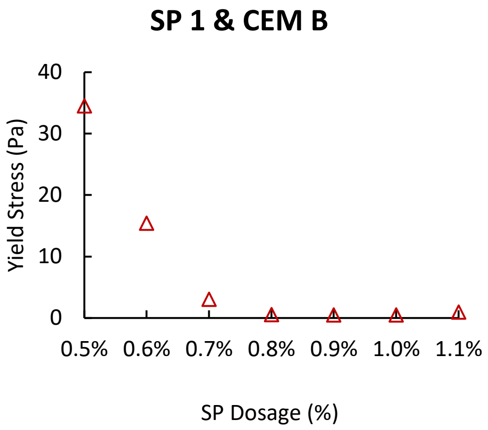


Figure 4.5: Yield stress results

4.3.3 Effect of SP1 with Cement C on the flowability of SCCM

SP1 and Cement C showed a similar trend to the other two cements, as illustrated in Figure 4.7. Figure 4.6 indicates that the mini-slump flow increased with an increase in superplasticiser dosage from 0.5 to 0.7% before declining. The optimum dosage was between 0.7% and 0.9%.

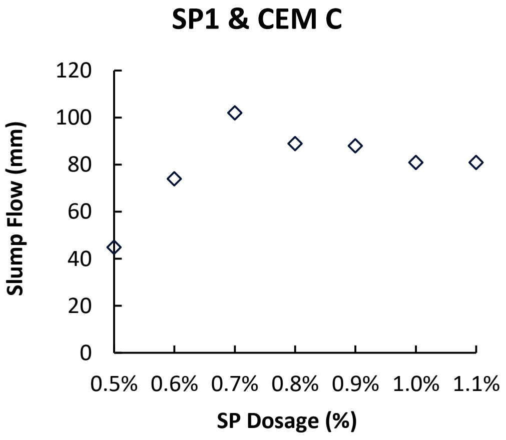


Figure 4.6: Mini-slump flow results

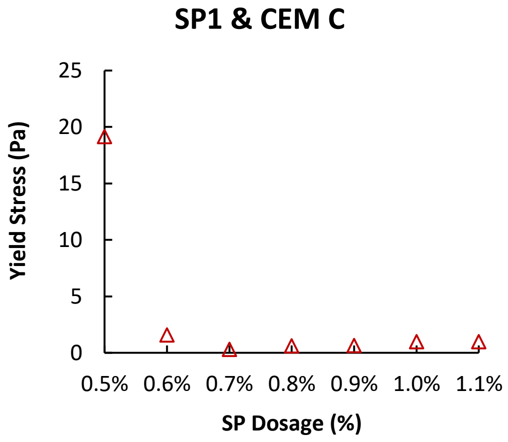


Figure 4.7: Yield stress results

4.3.4 Effect of SP2 with Cement A on the flowability of SCCM

A decrease in yield stress with an increase in superplasticiser dosage is seen for Cement A. Any dosage above 0.9% resulted in a decrease in flow and increase in yield stress, as seen in Figures 4.8 and 4.9. The selected optimum dosage was between 0.8% and 0.9%.

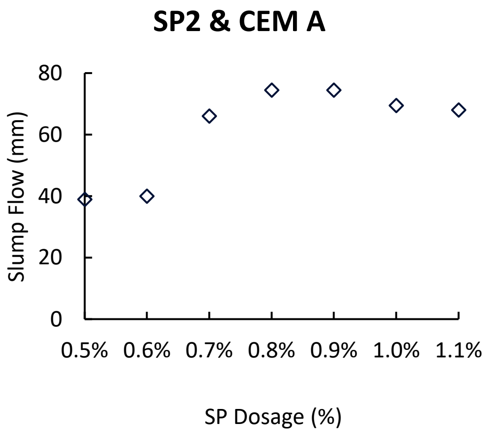


Figure 4.8: Mini-slump flow results

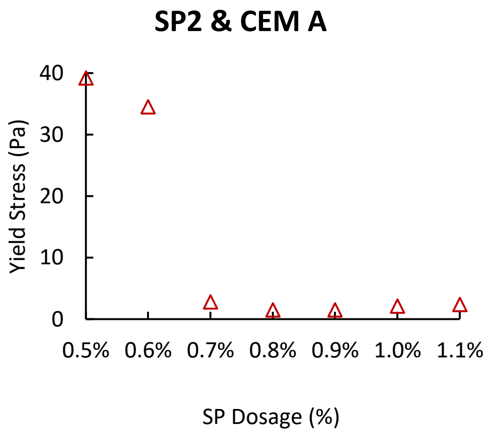


Figure 4.9: Yield stress results

4.3.5 Effect of SP2 with Cement B on the flowability of SCCM

The results presented in Figures 4.10 and 4.11 show that the reduction in yield stress continued over the whole range of SP concentrations. An optimum dosage was not that easy to determine and was selected as between 0.8%-1.1%.

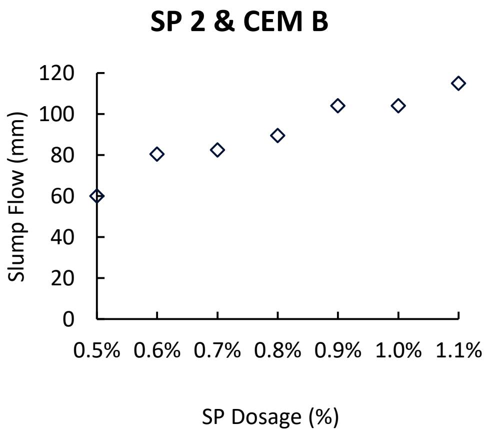


Figure 4.10: Mini-slump flow results

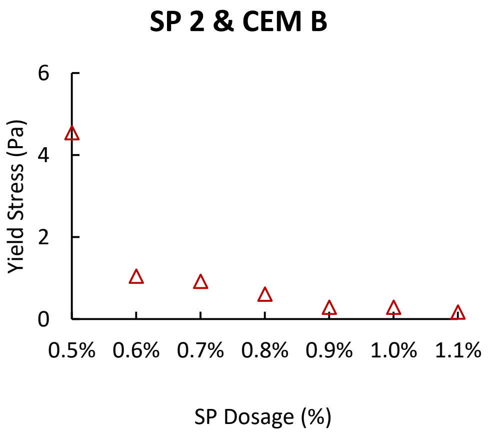


Figure 4.11: Yield stress results

4.3.6 Effect of SP2 with Cement C on the flowability of SCCM

Cement C and SP2 yielded the highest flow spread diameters when compared to all the other cements up to a value of 145.5 mm. At 0.7% superplasticiser dosage and beyond, the yield stress remained fairly constant, as shown in Figure 4.13. The optimum dosage therefore selected to be between 0.7% and 1.1%.

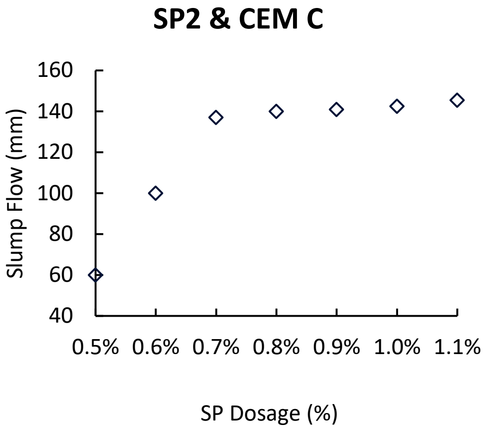


Figure 4.12: Mini-slump flow results

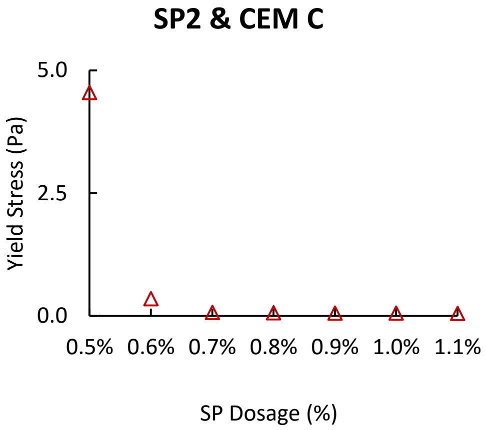


Figure 4.13: Yield stress results

4.4 Effect of SP3 with Cements A, B and C on the flowability of SCCM

SP3 was a combination of SP1 and SP2 mixed in varying fractions. The main aim of blending the two superplasticisers was to evaluate SP3 performance and to clearly establish if there would be any significant change in the rheological behaviour and flowability of the results compared to when they were used individually with each cement in Phase 1. SP1 was the older type of superplasticiser. The horizontal axis on all graphs shows SP2's fraction in SP3 dosage only, as per split dosage shown in Figure 3.8. The yield stress results for all cement mortars are shown in Figure 4.14.

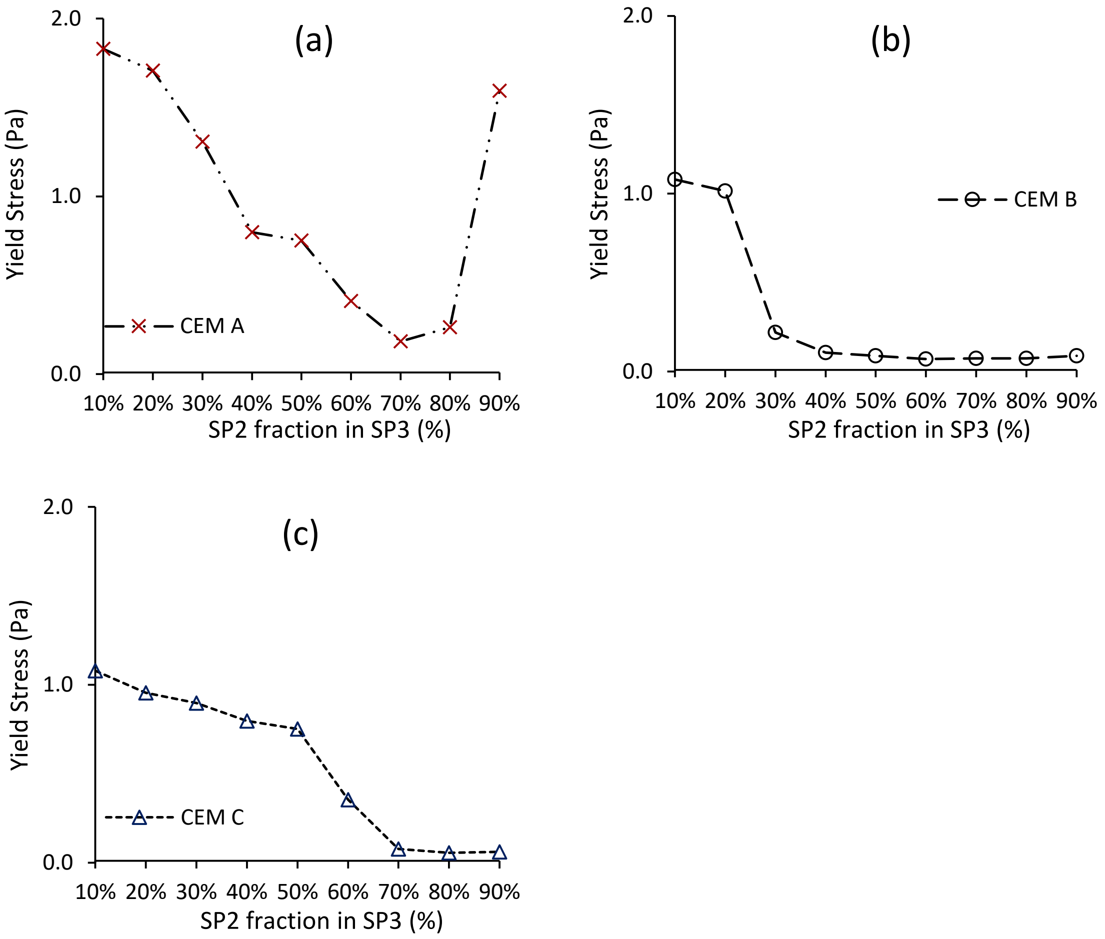


Figure 4.14: SCCM yield stress of different cement with blended superplasticisers expressed in SP2 fraction: (a) Cem A; (b) Cem B; (c) Cem C

Figure 4.14 (a) presents the yield stress results which were obtained by using Equation (2.13) from the mini-slump flow results for all cements. The pattern observed for Cement A shows that an increase in SP2 fraction within SP3 reduces the yield stress until reaching a plateau at approximately 70% SP2. The mortar behaviour displayed an unexpected increase in yield stress from 80% SP2 onwards for Cement A.

The data presented in Figure 4.14(b) shows that the combined superplasticiser becomes effective at 50% dosage split; anything beyond that does not improve the flowability of the mortar.

A decrease in yield stress with an increase in the fraction of SP2 in SP3 is evident in Figure 4.14(c). SP3 becomes effective at 70% SP2 where the yield stress is lowered significantly.

4.5 SCCM stability assessment for Cements A, B and C

The SCCM is the medium which supports the aggregates in the SCC suspension. It is, however, important to assess the stability of the mortar system without the stone aggregates as this dictates the strength of mortar systems. This section shows the stability results of the SCCM in relation to the yield stress values obtained at the combined superplasticiser dosage concentrations.

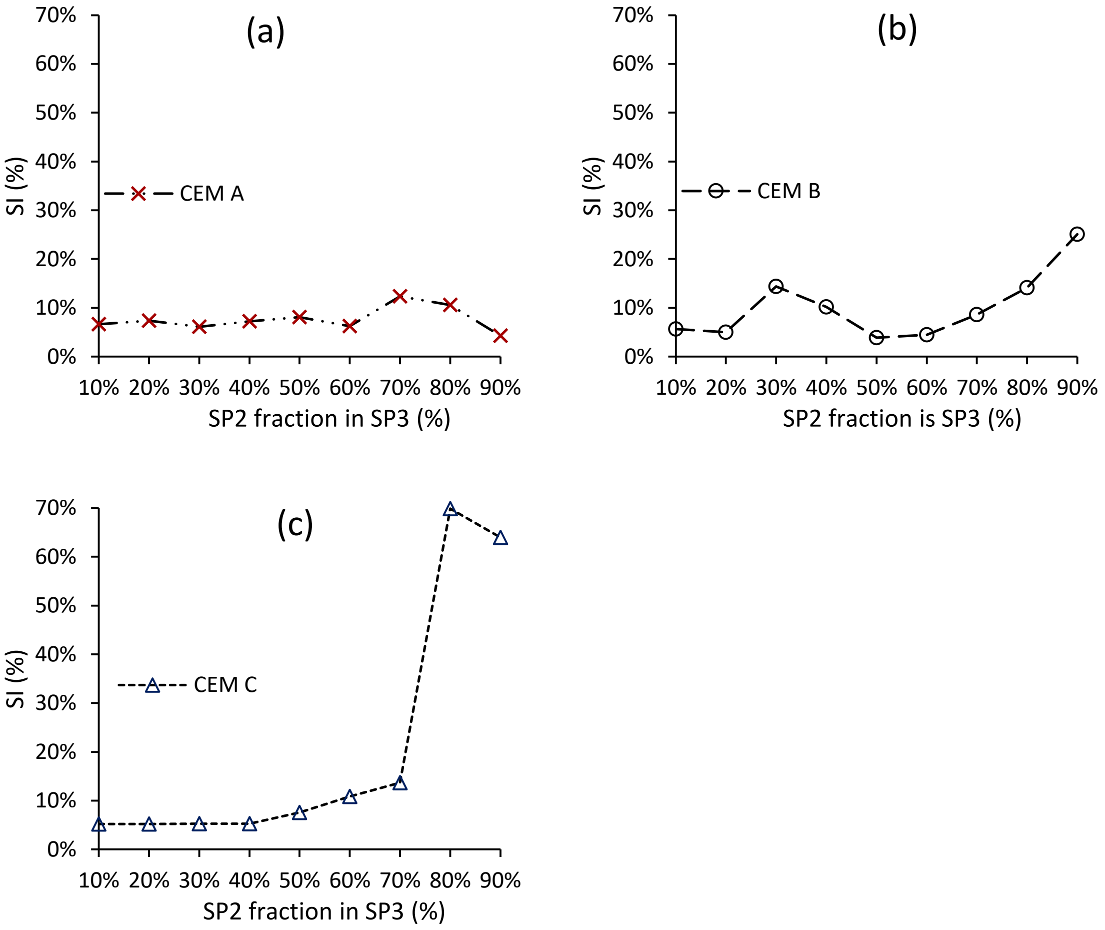


Figure 4.15: Segregation Index of different cement with blended superplasticisers expressed in SP2 fraction: (a) Cem A; (b) Cem B; (c) Cem C

According to Figure 4.15(a), the segregation index of the mortar remained stable between 6% and 8% from 10% to 60% SP2 when Cement A was used. An increased fraction of SP1 brought stability by preventing any major changes in the segregation index of the mix. The mixes were all stable between 10% and 60% SP2; however, at 70% SP2 the SI increased by 6%, double the previous mix. It can be seen that where the SI for the mortar was the highest, the yield stress was also the lowest, as shown in Figure 4.14(a).

It was interesting to see that none of the mixes tested had any bleeding for Cement B even though the lowest yield stress values were similar to Cement C and yet the segregation also still remained below 30%. Between 50% SP2 and 90% SP2 dosage in Figure 4.15(b), the SI increases continuously.

Cement C results are shown in Figure 4.15(c): the SI remained constant at 5% from 10% SP2 to 40% SP2, then increased moderately by 3% when SP2 fraction was increased between 40% SP2 to 70% SP2, and then spiked between 80% and 90% SP2.

The more dominant SP1 became, the less segregation and bleeding was observed. The rest of the cement mortars from 10% to 70% SP2, however, had an SI of less than 15% indicating good segregation resistance. Bleeding was also reduced to zero below 70% SP2, as seen in Figure 4.16.

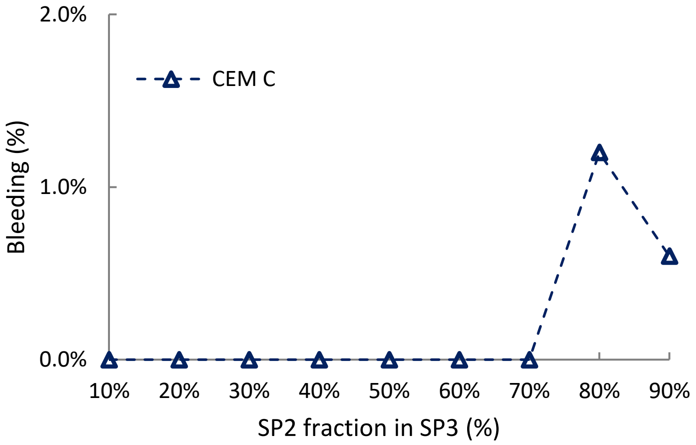


Figure 4.16: Effect of blending superplasticisers on the bleeding of cement mortar designed with different cement

4.6 SCCP yield stress and viscosity measurements for Cements A, B and C

After concluding the SCCM investigations, the study further investigated the cement paste rheological performance of the cements and SP3 samples at paste scale. The SCCP is the medium which supports the aggregates in the suspension; therefore, the strength of the cement paste can affect the suspension. This is why it is important to study the rheological behaviour of the paste only, to understand the effectiveness and strength of the net that keeps solid particles in suspension. The results show the rheological behaviour of the SCCP.

It was evident that a decrease in the SP1 fraction in SP3 led to a decrease in the yield stress for all the cement pastes (Figure 4.17) except for Cement A at 90% SP2 as shown in Figure 4.17(a).

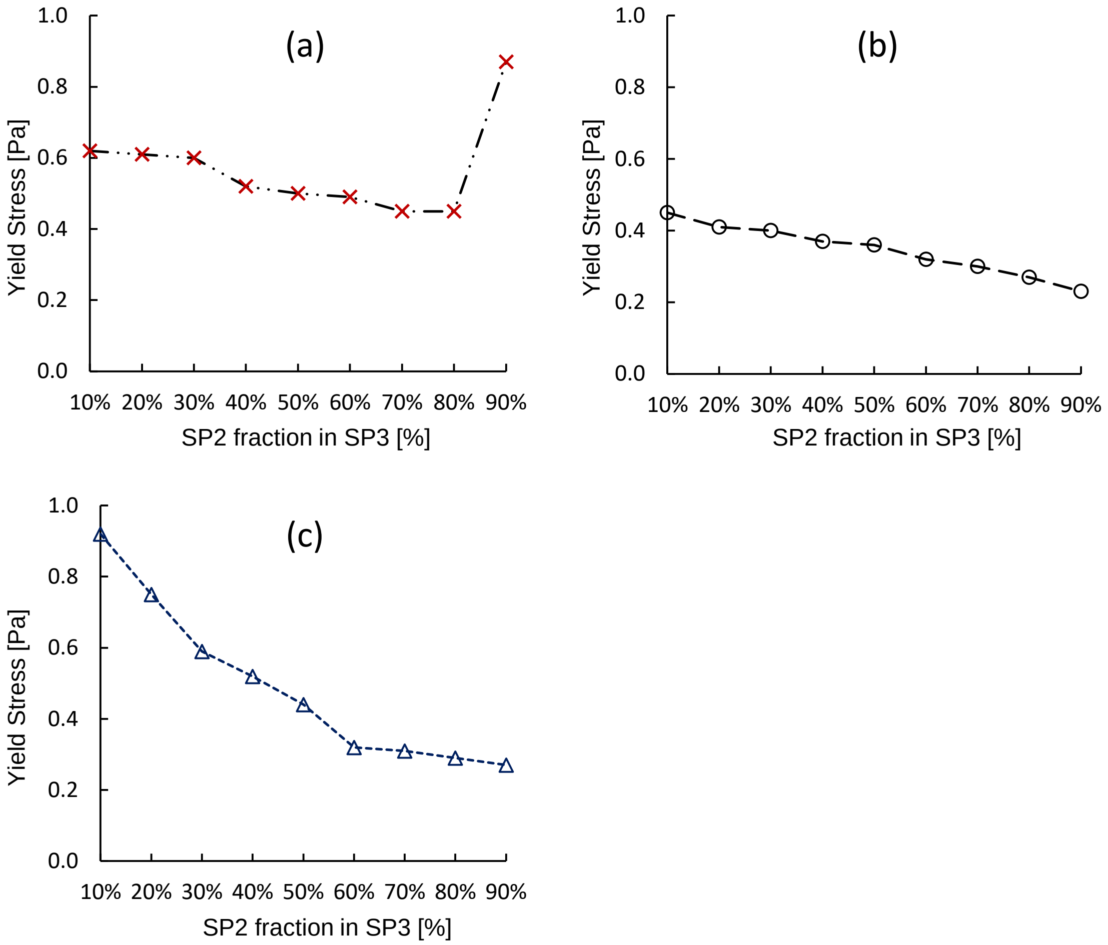


Figure 4.17: SCCP yield stress at different fractions of SP2: (a) Cem A; (b) Cem B; (c) Cem C

Similar to the yield stress, the viscosity results were obtained from the flow curves and applying the Bingham model. The data in Figure 4.18 show a constant increase in the viscosity of the cement paste with an increase in SP2 fraction. In Figure 4.18(c), it is also noticeable that Cement C viscosity results were slightly higher compared to Cements B and A. Cement A had the lowest viscosity measurements with one outlier at 80% SP2, as shown in Figure 4.18(a).

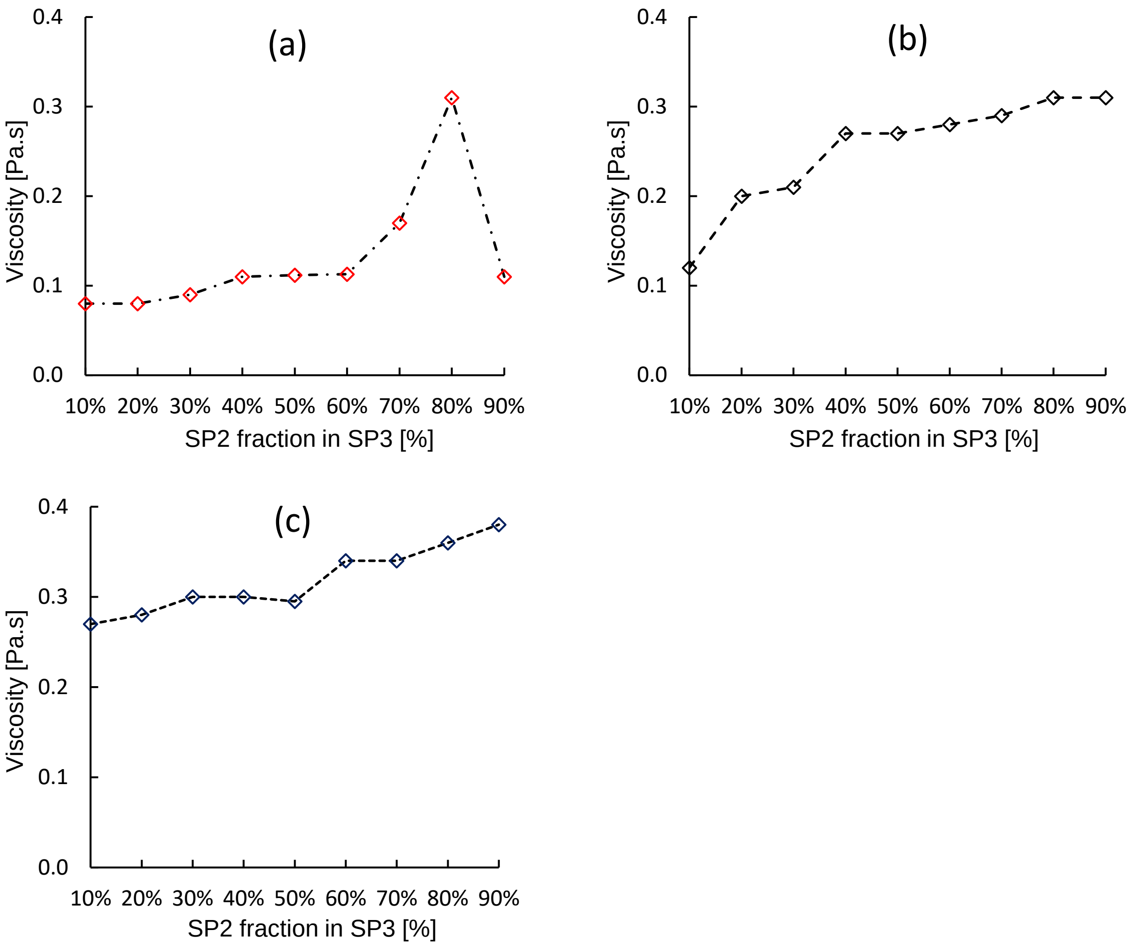


Figure 4.18: Cement paste viscosity at different fractions of SP2: (a) Cem A; (b) Cem B; (c) Cem C

4.7 Adsorption of superplasticisers with Cements A, B and C

The effectiveness of superplasticisers depends mostly on the adsorption of the superplasticiser on the cement particles. This is why the adsorption of SP3 on the cement particles was investigated to obtain further clarity on the adsorption behaviour of the superplasticisers with the three cements.

A similar trend could be seen for all three cements. From Figure 4.19, it was observed that cement particles adsorbed more of the superplasticiser at lower yield stress values judging from the yield stress results in Figure 4.14. The TOC results indicate that less adsorption occurred between the cement particles and the superplasticisers polymers at higher yield stress values which resulted from a high concentration of SP1 fraction within the superplasticiser solution. Figure 5.7 illustrates this relationship between yield stress and adsorption.

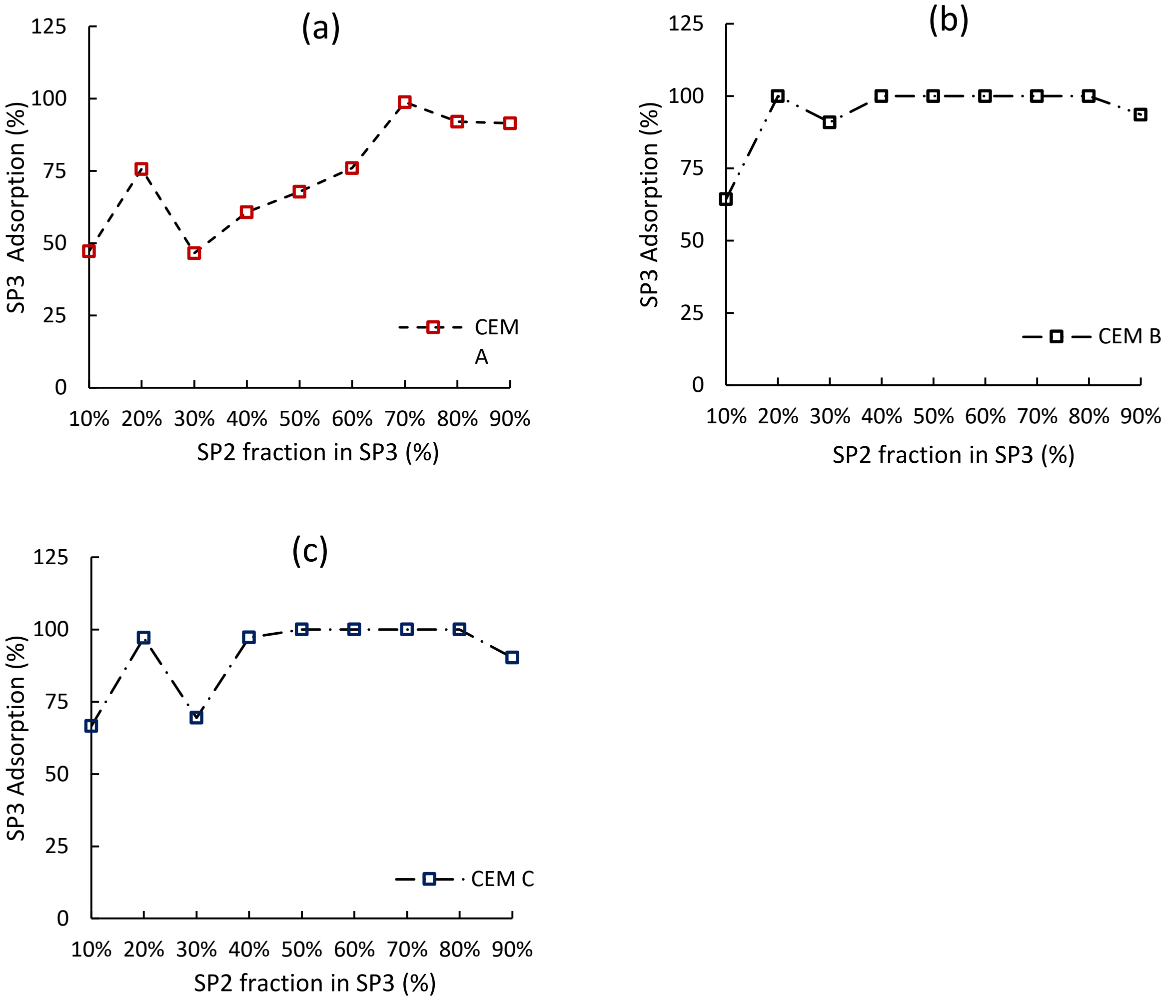


Figure 4.19: Adsorption behaviour of SP3: (a) Cem A; (b) Cem B; (c) Cem C

An increase in the adsorption with an increase in the SP2 fraction in the superplasticiser concentration is evident for all cements. This demonstrated SP2 effectiveness in dispersing the cement grains in all three cements. However, it can be seen that Cement A had the lowest adsorption rate of the three cements. On the other hand, Cements C and B had almost 70% of SP3 dosages being adsorbed a 100% by the cement particles.

CHAPTER 5

DISCUSSIONS

5.1 Introduction

The findings are discussed in detail in this chapter. Descriptive and comparative approaches are both used to rationally interpret the results. The compatibility between cements and individual SPs at their optimum content is analysed. Mini-slump flow values of cement mortar are compared to those of calculated yield stress values as highlighted in Chapter 3. Thereafter, the effectiveness of the blended SP3 with different proportions of the two superplasticisers expressed in terms of SP2 fraction are assessed. The sensitivity of cement characteristics on the superplasticisers is carefully evaluated. The effect of the blended SP3 (expressed in terms of SP2 fraction) at optimum dosage on stability of cement mortar is discussed. The stability of mortar samples is further correlated to the rheology of the cement pastes. Finally, the rheology of cement paste is discussed in terms of the adsorption characteristics of the blended SP3. Figures 5.1 to 5.4 verify the effect of SP1 and SP2 on cements.

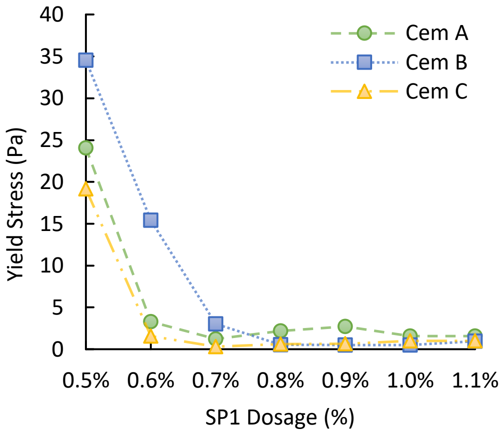


Figure 5.1: Yield stress results for SP1

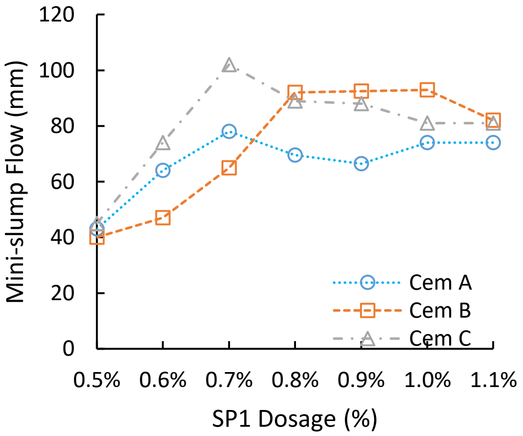


Figure 5.2: Mini-slump results with SP1

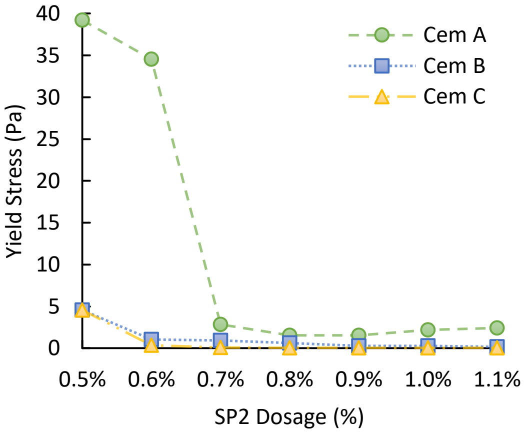


Figure 5.3: Yield stress results for SP2

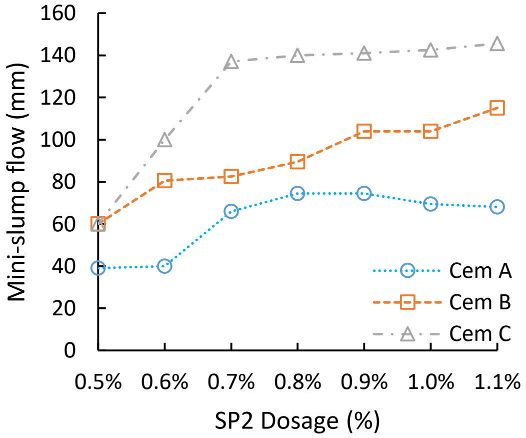


Figure 5.4: Mini-slump results with SP2

5.2 Effect of SP1 and SP2 on Cements A, B and C

It can be seen that the cements are more sensitive to SP2 compared to SP1. This is displayed by the difference in flowability as seen in Figures 5.2 and 5.4. Cement A is the only cement which the mini-slump flow did not reach 100 mm with SP2 whereas with SP1 only Cement C reached. This indicated that good compatibility existed between SP2 and the cements according to the findings by Mardani-Aghabaglou et al. (2013) which accredited such behaviour to the increased number of side chains of the superplasticiser.

The change in behaviour of the yield stress is another factor indicating the sensitivity of the cements to SP2. Figure 5.3 shows a drop in the initial yield stress by 35 Pa between the cements compared to the 11 Pa drop in yield stress with SP1 at 0.5% dosage. This suggests that more adsorption between the cement particles with SP2 occurred when Cements B and C were used and vice versa for Cement A. This led to increasing displacement of cement particles in each mortar mix or that the free flowing superplasticiser molecules within the solution reduced the yield stress, as suggested by Matsuzawa et al. (2019).

All cements show a reduction in yield stress and increase in slump flow with an increase in superplasticiser dosage as expected and reported by Bessaies et al. (2014) and Alonso et al. (2013). Cement C generally had the lowest yield stresses for SP2 as seen in Figures 5.4 and 5.2, but only up to 0.7% when used with SP1, while Cement A has the highest yield stress values. This signifies the importance of testing each superplasticiser with each cement because of the chemistry dynamics involved which originate from the raw materials used in cements (Erdogdu, 2000).

Cements A, B and C achieved an optimum dosage between 0.7% and 1.1%, respectively, when superplasticiser SP2 was used, while with SP1, the optimum dosage was between 0.7% and 0.9%. The results prove that it is possible to obtain different optimum dosages using the same superplasticiser (Gołaszewski & Szwabowski, 2004; Hallal et al., 2010). After the optimum dosage was reached, the yield stress of some SCCMs increase while some decrease. In this study, the optimum dosage was selected based on the flowability and visual assessment results from the mini-slump flow test. However, an average of the three optimums was obtained for each cement. SP1 had an average of 0.73% while SP2 was 0.9%. Subsequently, the optimum dosage for both superplasticisers with all three cements ranges between 0.73 to 0.9%. The higher optimum dosage range (0.9%) was then selected as the optimum to proceed to the next phase to be within the optimum range for all cements. This was based on the fact that the change in yield stress of cement pastes is more critical before the optimum dosage point and less relevant beyond that (Robert et al., 2018). The wide optimum dosage range existed because two different superplasticisers with 3 different cements were used for the study. Taking this into consideration, it was assumed that any dosage between 0.8% and 0.9% would have minimal impact to the results as the optimum on average would have been reached at 0.73%, hence the choice of the higher optimum dosage.

5.3 Effect of SP3 on Cements A, B and C

After obtaining the optimum dosage and assessing the effect of the individual superplasticisers (SP1 and SP2) on cement, the two superplasticisers were blended to form SP3 with the intention of improving the rheological properties of the SCCM. This section compares the blended superplasticisers at an optimum dosage of 0.9%.

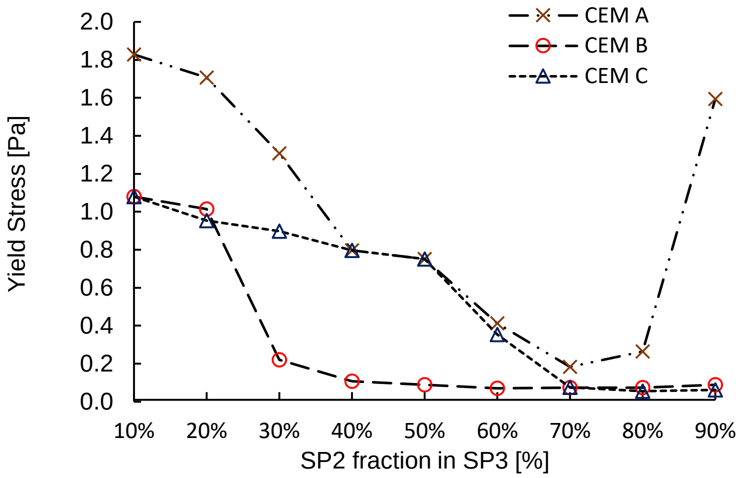


Figure 5.5: Combined SCCM yield stress results for SP3 with all three cements

Blending the admixtures increased the chemical complexity of the mix as the chemistry involved between the admixtures and cements was much greater as each superplasticiser had a different chemical structure while all cements differed chemically and physically. This affected the rheological behaviour of cement pastes as highlighted in a study by Nunes et al. (2011).

A similar trend was evident for all cements as shown in Figure 5.5. In all the cements, an increase in SP2 fraction in the cementitious materials led to a constant decrease in the yield stress. This meant that SP1 reduced the flowability and fluidity of the mixes. Long side chains in superplasticisers led to less adsorption by cement particles (Kong et al., 2016). The chemical structure of the admixtures used for SP3 also differed. SP2 had shorter side chains compared to SP1. It is assumed that the high efficiency displayed by SP2 was a result of this chemical structure which reacted positively with the cement particles.

Generally, Cement A had the highest yield stress values followed by Cement C and with Cement B with the lowest yield stress values. Cement A had the lowest specific surface area and a lower C_3A content. It is believed that this led to a slower rate of adsorption as shown in Figure 5.11(a) and reduced the rate of hydration; hence, the high yield stress values for Cement A (Li et al., 2020). This observation was true in earlier studies by Vikan et al. (2007) who discovered that superplasticisers get adsorbed less onto cements particles with a low surface area and a low tricalcium aluminate phase content.

The ability of Cement B to achieve lower yield stress values without any stability concerns can be attributed to the combination of high C_3S content and second highest C_3A content present in the cement phases based on the rheology results. The C_3S phase has been discovered to produce quick reactions; cement hydration happens very fast (Choudhary et al., 2015). Cement B had the lowest yield stress for the mortar tests, suggesting that SP3 actually reacted at a slower rate with Cement B, but due to a high C_3A content, greater adsorption of the superplasticiser was achieved and this minimised the fast reaction effects of the C_3S. This led to low yield stress values caused by the effective dispersion of cement particles as seen in the adsorption data in Figure 5.11(b).

5.4 Performance of the superplasticisers on cement in SCCM

Data in Figures 4.3, 4.10 and 4.14 make evident that the lowest yield stress results for Cement A were improved slightly when SP1 and SP2 were mixed in different ratios as compared to when used separately. They were reduced by 15% and 12% for SP1 and SP2, respectively. This was assumed to be due to the high concentrations of SP2 within SP3 which reduced the yield stress. SP2 had a slower electrostatic adhesion to cement due to shorter side chains which resulted in delayed hydration (Marchon et al., 2019).

The reduction in yield stress can be attributed to the addition of SP2 which has a chemical structure with very short and thick steric chains that produce greater adsorption on cement particles (Winnefeld et al., 2007). SP3 performed much better compared to the individual superplasticisers with Cement B (Figures 4.5, 4.12 and 4.14) with only higher yield stress values at 80% SP1 and 90% SP1. This high yield stress was attributed to the high dosage of SP1 polymers as they tend to decrease the flowability of the mortar and increase the viscosity. SP3 showed an improvement for all results with Cement B and had a lowest yield stress value which was a significant improvement from the lowest obtained for SP2 and SP1. For Cement C, the improvement was not better than when using SP2 when SP3 was used. This can be attributed to SP2's performance. SP2 produced greater fluidity with Cement C. Moreover, SP2 had shorter side chains compared to SP1. The shorter side chains produced greater steric hindrance which reduced the yield stress (Feng et al., 2018).

5.5 Effect of rheological behaviour on stability of SCCM

Mortar is a suspension with two mediums: liquid and solid. The liquid medium is concentrated with paste while the solid is concentrated with the fine aggregates. The available literature associated the stability of the suspension to the viscosity of the suspension (Pichler et al., 2017). The effect of yield stress on the static segregation of a SCCM suspension medium within a concrete system was another focus area. This section assessed how the rheological parameters affected the stability of the SCCM for each cement. Figure 5.6 includes the recommended ranges for the segregation index. The aggregate size used in this study was approximately 1mm in size and therefore as a limitation it must be noted that the results could vary when larger aggregates are used and could result in major instability for a similar mix design.

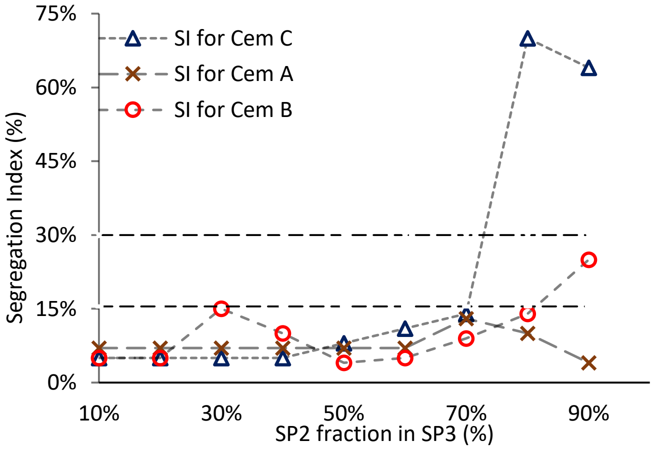


Figure 5.6: Segregation index range in relation to recommended criteria

According to Libre et al. (2010), a segregation index (SI) less than 15% was deemed satisfactory for SCC. However, to achieve stable SCC mixes, an SI below 30% was suggested as still acceptable for SCC mortar. The researchers also indicated that SI values between 30 and 130% were likely to experience some sort of segregation and any value above 130% would result in unwanted segregation in the mix.

Based on the stability results, it is evident that combining the sands also contributed to the stability of the SCCM due to improved compaction (Nécira et al., 2017). The SI results of all mixes were considered stable with the exception of two. The results obtained and presented in Figure 5.6 show that 89% of the results in this study had a segregation index of less than 15% which led to little or no bleeding in the SCCM. It was also observed that no bleeding occurred in Cements A and B by both visual observations during testing as well as by the bleeding tests conducted. Figure 5.7 shows that reducing the yield stress of SCCM will result in higher segregation in SCCM.

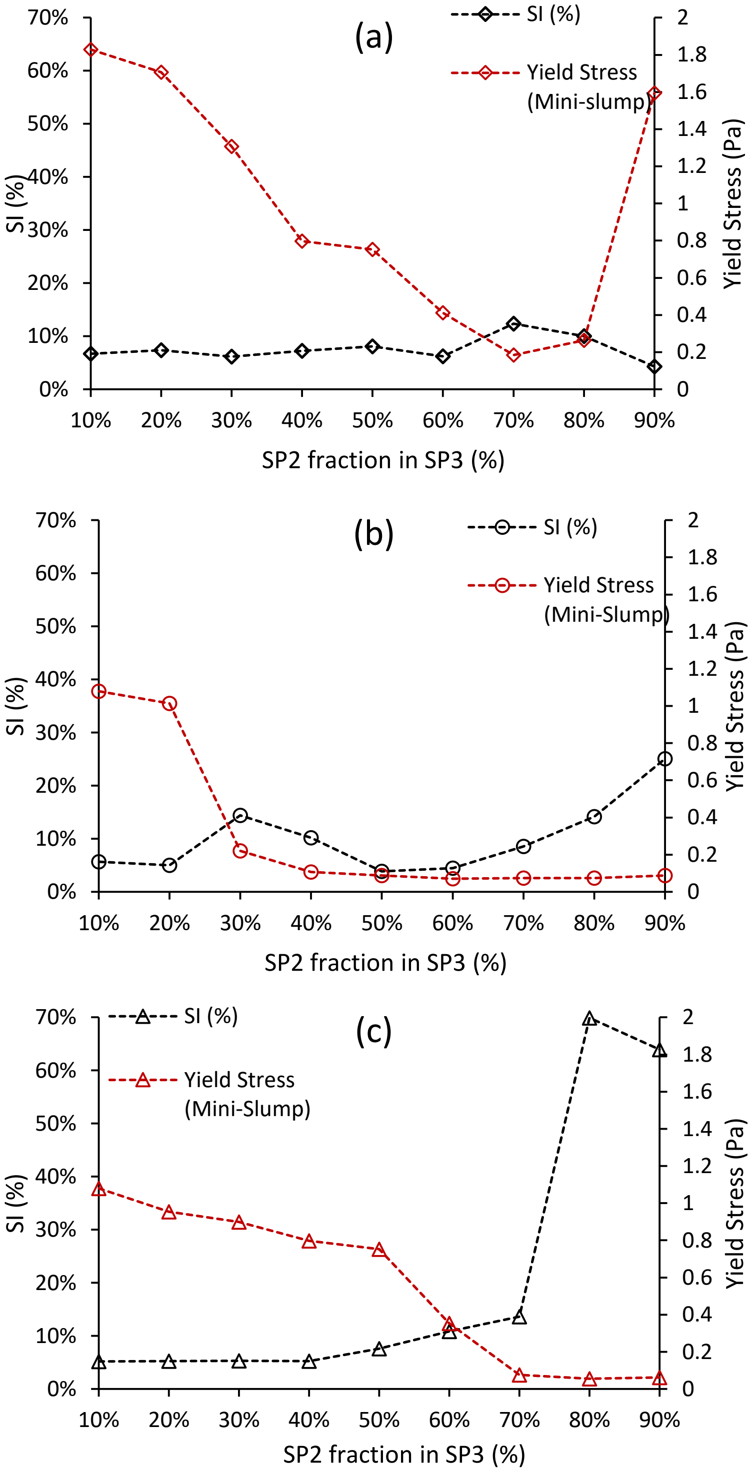


Figure 5.7: Relationship between yield stress and segregation of SCCM

High fluidity was the contributing factor to the segregation in the SCCM according to findings by Libre et al. (2010). The results shown in Figure 5.7 indicated an increase in segregation with the reduction of the yield stress. The reduction in yield stress appeared to be the result of adsorption of superplasticiser polymers onto the cement grains as shown by the adsorption results in Figure 5.11. This also meant that the cement paste within the mortar solution was not strong enough to keep the sand particles in suspension at these low yield stresses. Hence, segregation occurs because all solid particles are suspended in the cement paste thereby making cement paste the carrier of solid particles in the cement suspensions (Rubio-hernández et al., 2020).

Where the yield stress was the lowest, the SI was also the highest for all cements. An equally significant contributing factor to this was the superplasticiser adsorption which was also at 100% for Cements A, B and C at the lowest yield stress value. The adsorption of superplasticisers played a key role in reducing the yield stress which resulted in greater fluidity by delaying the hydration, as concluded by He et al. (2017).

Cement C showed high fluidity caused by the low yield stress values at 80% SP2 and 90% SP2 which resulted in a high degree of segregation, findings consistent with Abebe and Lohaus (2017). An increase in SP1 fraction increased the stability of the mix by constantly reducing the segregation of the SCCM, while increasing the SP2 fraction increased the yield stress of the mortar.

Because each SCCM mix contained a varying fraction of SP2 and SP1, all mixes had different yield stress values. It was evident that there is a certain range where the change in yield stress has no effect in the segregation. For Cement A it is between 10% and 60% SP2 fraction, with the SI ranging between 6% and 7%. This range is between 30% and 60% for Cement B and between 10% and 50% for Cement C. In this range, irrespective of the change in yield stress of the SCCM, the segregation is not affected much as the SCCP which acts as a net keeping the solid particles suspended was strong enough to prevent the particles from sinking through the SCCP.

In this same range between 10% and 60% SP2 fraction, Figure 5.10 shows that the viscosity was also constant. This implies that viscosity was effective at these dosages and significantly influenced the outcomes of the segregation for Cements A and C as suggested by Jiao et al. (2017).

There was also a range where irrespective of the high and increasing SI index, the yield stress remained fairly unchanged. This was between 70% and 80% for Cement A and 70%-90% for Cements B and C. The adsorption was also very high at this range, suggesting a great dispersion of particles due to the superplasticiser molecules being adsorbed onto the cement particles resulting in high fluidity and low yield stress; hence the high segregation (He et al. 2017). Bleeding followed a similar but opposite trend to the yield stress of the SCCM, as seen in Figure 5.8.

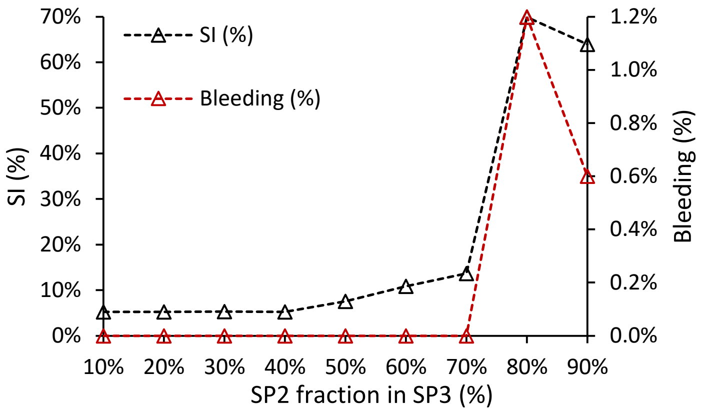


Figure 5.8: Bleeding and SI in relation to SP3 dosage for Cement C

The bleeding tests for Cements A and B indicated no was bleeding present in all mortars and only two mixes for Cement C had bleeding. This was verified by the visual inspections carried out during testing, in agreement with the results and findings by Perrot et al. (2012). The study determined no correlation between bleeding and yield stress. High instability existed with lower yield stress values, with the bleeding index going beyond 1% at 90% SP2 and 80% SP2. The increase in bleeding was a result of the dominance of the SP2 fraction which resulted in higher adsorption of the polymers. This suggested that more bleeding occurred due to greater adsorption which resulted in greater fluidity, in agreement with earlier studies by Petrou et al. (2000) who reported that bleeding in cement mortar was caused by gravitational forces which attracted the solid particles to the surface and pushed the liquid upwards. Moreover, this is in line with Stokes' law, as according to Margarita et al. (2019), cementitious materials have different densities and these densities play an important role in the settling of aggregates to the bottom of the suspension and pushing the liquid upwards which contributes to bleeding.

The constant decrease in yield stress and increase in viscosity at each dosage reduced the bleeding to an absolute minimum from 10% SP2 to 70% SP2. The increased fluidity reduced the yield stress in the SCCM which resulted in high segregation and bleeding. In this case, the viscosity increased and the yield stress increased; hence the greater segregation and bleeding resistance seen from 70% SP2 downwards.

5.6 Effect of SP3 on Cements A, B and C on SCCP

After conducting the mortar tests, cement paste rheological behaviour and superplasticiser adsorption studies were conducted. SCCP is the liquid medium in SCCM which sustains and carries the solid medium (sand aggregates). Therefore, the rheology of the SCCP influences the SCCM rheological outcomes. The solid particles are inert and therefore do not react with the superplasticiser nor the cement, thus making SCCP the medium where the chemical reactions and adsorption occurs in SCCM solutions. The SCCP results show that Cement C had highest initial yield stress and highest rate of change. From 30% SP2, Cement A had the highest yield stress values followed by Cement C and Cement B with the lowest yield stress values on the SCCP (Figure 5.9). For all three cements, the yield stress decreased with an increase in SP2 fraction. Cement A spiked sharply at 90% fraction similar to the SCCM results. The reason for this is not clear.

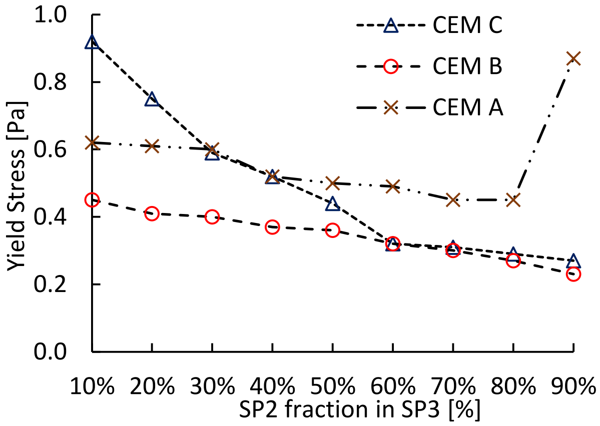


Figure 5.9: Combined SCCP yield stress results for SP3 with all cements

Based on the findings by Massoussi et al. (2017), it was expected that the SCCM yield stress results would be slightly higher than the SCCP results because of the effect of the sand on the fluidity. The results showed this to be true for Cements A and C only with Cement B having SCCP results higher than the SCCM. This could have been attributed by the two different methods used to mix the cement paste and the mortar, as mechanical mixing provides much more intensity compared to hand mixing.

It is interesting to note that Cement C had the highest viscosity and Cement A had the lowest viscosity results of all three cements (Figure 5.10). This could be attributed to the particle sizes of Cement A. According to the particle size distribution analysis, Cement A had the lowest specific surface area. It is assumed that this led to the less adsorption which resulted in higher yield stress and lowest viscosity values compared to the other two cements, similar to what was shown in the study by Li et al. (2020). Mahdikhani and Ramezanianpour (2015) concluded that cement mortar binders with large surface areas would result in higher viscosities.

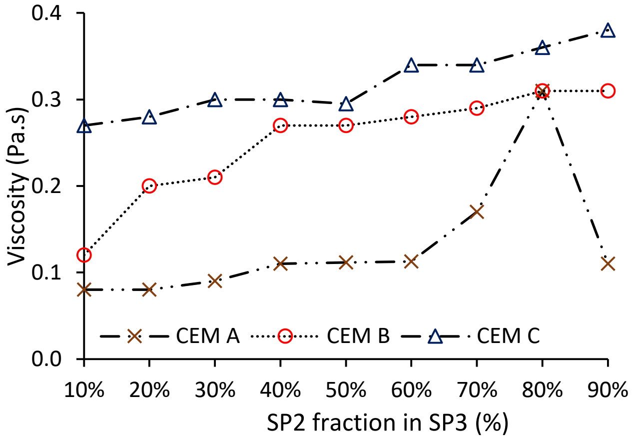


Figure 5.10: Combined SCCP viscosity results for SP3 with all cements

Cement C also had the second highest C_3S content, the highest C_3A content and the largest surface area of all three cements, yet Cement B had much better fluidity than Cement C. This could only mean that the phase composition and particle sizes in this study were not the only determining factors of the rheological results. This was in agreement with the conclusion reached by Bogner et al. (2020) that the change in rheological properties was not caused only by the interactions and changes of the cement phases and their particles. The study determined that the ability to disperse and then attract the cement particles in cement pastes, which is caused by the formation of C-S-H and other hydration products, was also a major contributing factor. C_3S transformed into C-S-H after the quick early reactions (Choudhary et al., 2015).

The rheology results above show that when the yield stress for both the mortar and the paste decreases, the viscosity increases. These findings are in agreement with the conclusion reached by Anagnostopoulos (2014) on the effect that superplasticisers have on cement grouts. This can be credited to the effectiveness of polycarboxylate based superplasticisers in keeping the cement particles apart, while the viscosity increase was due to side chain ability to adsorb the cement particles and create cohesiveness in the cement solutions. A recent study by Zhang et al. (2020) also discovered that viscosity increases when the superplasticiser molecular weight is decreased in cement pastes. SP2 had a lower molecular weight than SP1, resulting in an increase in viscosity with an increase in SP2 dosage which was evident in the results shown in Figure 5.10.

The viscosity results show that a lower viscosity in SCCP led to less segregation in the SCCM. These results are in contrast with the findings by Margarita et al. 2019 and other studies in the literature. This could have been attributed to the complex chemistry of blending the two superplasticisers and the fact that the superplasticisers were mixed at different fractions and the dosage was not necessarily increased.

The stability of mortar was assessed based on the reaction of superplasticiser SP3 and the different cements. A similar trend was evident in all cements both at paste or mortar scale as shown in Figures 5.5 and 5.9. In all the cements, an increase in SP2 fraction in the cementitious materials led to a decrease in the yield stress. This is linked to the adsorption of the superplasticiser to the cement particles (Lange & Plank, 2016).

5.7 Adsorption of superplasticiser with Cements A, B and C

The complex nature of mixing two polymers to produce SP3 and using three different cements with different phase compositions made analysing the results more challenging. The superplasticiser assessed was a blend of SP1 and SP2 in different proportions. SP1 was the cheaper of the two superplasticisers; they were combined to see how they would perform when blended from the manufacturer’s perspective.

The total organic carbon (TOC) test was conducted to understand the adsorption behaviour of the admixtures with the cements and to assess if this corresponded to the rheology results in line with similar adsorption studies by Burgos-Montes et al. (2012) and later studies by Matsuzawa et al. (2019).

The TOC was conducted after the supernatant was obtained from the centrifuge at 5000 rpm. The diluted supernatant TOC reading was then subtracted from neat cement with water and the neat superplasticiser with water supernatant to obtain the adsorption from the total carbon analysis. The adsorption results affirmed the rheology results obtained; Figure 5.11 shows the adsorption results of the three cements in relation to their yield stress.

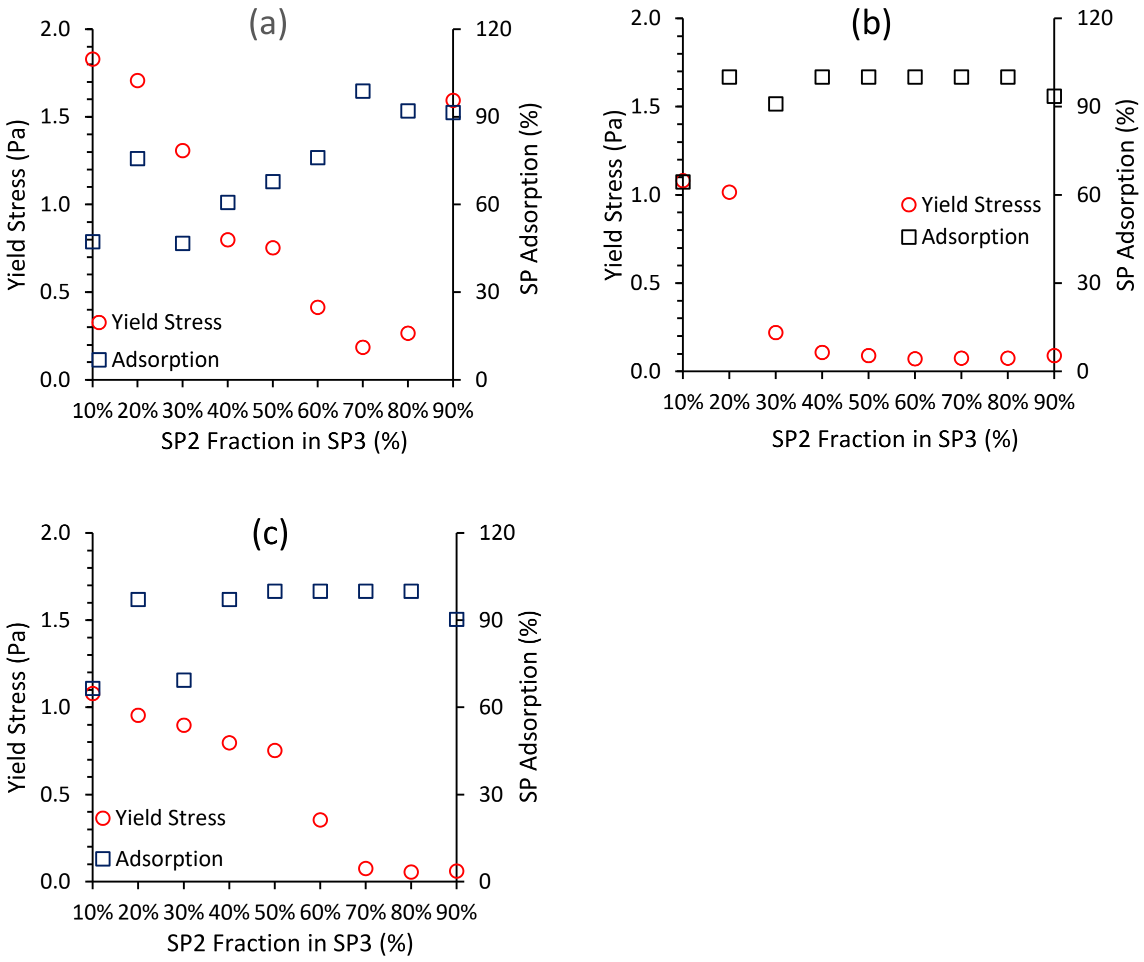


Figure 5.11: Yield stress and adsorption: (a) Cem A; (b) Cem B; (c) Cem C

The results for all cements indicated that a high fraction of SP2 in SP3 reduced the yield stress while SP1 increased the yield stress of the mortar mixes, a finding in agreement with Feng et al. (2018) who concluded that polycarboxylate-based superplasticisers with longer side chains and densities get adsorbed less effectively. This was the case with SP1 which had long side chains. This resulted in higher yield stress values in all cements compared to SP2, suggesting that greater adsorption took place between SP2 and the cement particles. However, because SP1 had a greater molecular weight than SP2, Cements A and C showed an increase in yield stress from 30% SP1 content and higher. This shows that the molecular weight of the superplasticisers plays a critical role through competitive adsorption and likewise affects the rheology (Winnefeld et al., 2007). Winnefeld et al.'s study concluded that polymers with higher molecular mass will result in increased adsorption to cement particles.

The fact that higher yield stresses occurred while SP1 fraction in SP3 was still minimal and increased with the dosage increase suggests that the SP1 started competing with

SP2 even though it had longer side chains, but due to the difference in molecular weight. The TOC test in this study could not provide measurements to indicate which of the two superplasticisers were adsorbed the most. Therefore, it was difficult to prove whether the molecular weight was the governing factor in the adsorption process as found in the study by Zhang et al. (2020) or whether this was due to other factors. However, it can be seen that the adsorption of superplasticiser particles has a direct relationship with the rheology of self-compacting concrete mortar and influences the yield stress of the mortar. This in turn affects the stability.

Adsorption greater than 90% seemed to produce good flowability results for all cements, indicating that the non-adsorbed superplasticiser molecules had no major impact on the flowability of the mortars, like in the study by Lange and Plank (2016). This was in contrast to the conclusion reached by Matsuzawa et al. (2019) in their study of non-adsorbed superplasticisers, as they suggested that non-adsorbed superplasticisers should increase the flow in cement paste, but warned that this depended on the molecular structure of the superplasticiser used. Therefore, their findings will vary depending on the chemical and physical structure of each superplasticiser.

The results showed that an increase in yield stress resulted in less adsorption of the admixture onto the cement particles, as described by Bessaies et al. (2014), and high adsorption led to lower yield stress values as expected due to the steric hindrance in the suspension caused by the repulsion of the particles from each other (Flatt & Houst, 2001). This resulted in less segregation and bleeding for all the SCCMs tested in this study. The major difference between the cements was the that the optimum adsorption is reached at approximately 70% SP2 for Cement A, 40% SP2 for Cement B and 50% SP2 for Cement C.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 Introduction

The effect of the rheological parameters on the stability of SCCM is not well understood. The aim of this study was to determine the effect of cement paste yield stress on the cement mortar stability. This was achieved by designing cement mortars and pastes with different yield stress values using three different cements and two distinct superplasticisers. The interaction between cements and superplasticisers was first optimised and cement mortar and paste yield stress were obtained by blending the two superplasticisers in different proportions at the obtained optimum dosage. This chapter outlines the conclusions from the discussions, observations and results obtained for this study, concluding with recommendations for future studies.

6.2 Individual superplasticiser performance (optimisation)

At the lowest dosage of SP1, the flowability results were similar for all three cements showing that the adsorption and dispersion at 0.5% was similar when SP1 was used. Improved flowability values were achieved when using Cement C in the presence of SP1 compared to other cements. In particular, Cement A exhibited some inconsistency in its flow behaviour when used at superplasticiser concentration in the range of 0.5 - 0.7%.

In general, SP2 seemed to be more effective than SP1 in the presence of all cements used. Expectedly, the flowability of cements greatly improved with the increase in superplasticiser dosage. The optimum dosage for both superplasticisers with all three cements was achieved at around 0.9%. Finally, it can be seen that SP2 was more compatible with all cements than SP1 since fewer fluctuations in their respective cement mortar rheological behaviour were observed.

6.3 Effectiveness of blended superplasticiser SP3

It was noticed that when blending SP1 and SP2 in different proportions and used at 0.9% in all cements, the flowability of all cement mortars was improved compared to the individual performance of these superplasticisers.

SP3 reduced the yield stress values for all cements compared to SP1, but not much change occurred when SP2 and Cement C were tested. The yield stress value of Cement A was reduced by 15% and Cement B by 40% when SP3 was used. There was no reduction in yield stress from the results obtained with Cement C and SP2, but the yield stress results were reduced by approximately 20% compared to Cement C and SP1. In general, mortar mixes with a high fraction of SP1 in SP3 decreased the flowability of all cement mortars. This led to an increase in yield stress of all cement pastes while their viscosity values decreased.

Therefore, it can be concluded that SP3 further reduced the yield stress results compared to when SP1 and SP2 were used separately in the mortar mixes, except for Cement C and SP2 mixes.

6.4 Cement paste rheology and mortar stability

It was found that cement mortar with a cement paste that has a low yield stress value resulted in an unstable suspension. In other words, cement paste with low yield stress caused higher segregation indexes in corresponding cement mortar mix. At lower viscosity values, the cement pastes had mortars which showed some stability. It could be seen that stable mortars can only be achieved when the composite cement paste exhibits enough strength or higher yield stress that prevent aggregates from sinking.

Surprisingly, there was no relationship observed between the rheological parameters of cement paste and the bleeding of corresponding cement mortars for all cements, with the exception of two mixes for Cement C only. This phenomenon is probably more a result of the packing of the solid phase within the system than the rheology of the liquid phase.

The stability of all cements was mostly achieved at 40% SP1 fraction and above for SP3, while only at 60% and below SP2 fraction a similar behaviour was observed. At low fractions of SP2 up to 60%, cement pastes resulted in mortars with a better stability. Between 10% and 60% SP2 fraction in SP3, the segregation index remained constant irrespective of the different yield stress values in this range. This suggested that in certain ranges of yield stress values, the effect of cement paste rheology on corresponding mortar stability can be misleading, causing an underestimation of the impact of cement paste rheological performance on the stability of the corresponding mortar. Judging from the only two cement mortar mixes for Cement C which had bleeding, low viscosity values reduce the bleeding of SCCM similarly to segregation in this study. Also, the higher the yield stress, the lower the segregation in the mortar.

The three cements did not have the same behaviour and outcome and therefore the effects that the rheological parameters have on segregation properties also differed for each cement depending on the chemical and physical structure of both the superplasticiser and cement. Cement A had the lowest specific surface area and a lower C_3A content than Cement B, leading to a slower rate of adsorption which reduced the rate of hydration. Cement B is more compatible with SP3 compared to the other two cements considering that no segregation or bleeding existed for the same slump flow diameters as Cement C: this can be attributed to the balance between high C_3S and high C_3A content present in the cement phases. Cement B achieved greater adsorption of the superplasticiser due to the high C_3A content present, leading to low yield stress values caused by the effective dispersion of cement particles. But Cement C, when used with SP2, achieved higher flowability results than 80% of the results of SP3. The stability assessment, however, was not done for the individual superplasticisers so there is no evidence that there was no bleeding or segregation.

Based on the results, it is not recommended to increase SP2 fraction in SP3 above 60% as the SCCM will be prone to segregation. However, it can be concluded that for all cements with high yield stress in SCCM and SCCP with corresponding lower viscosity, results will be lower segregation and no bleeding, thus providing stability in cement systems. With increased dosage of SP2 within the combined SP3 blended, superplasticiser yield stress decreases continuously and viscosity steadily increases. There is no evidence of a relationship between bleeding and viscosity nor with bleeding and yield stress for Cements A, B and C (only two of the tested mortars exhibited bleeding for Cement C only).

6.5 Recommendations

This study provided some insight into the relationship between the rheology of cement paste and the stability of its corresponding cement mortar. However, further investigation should assess the cause of the bleeding and to what extent it can influence the rheology of cement paste within a cement mortar system.

Furthermore, a comprehensive chemical investigation into the competitiveness of superplasticiser polymers when blending different superplasticisers with distinct chemical compositions would be interesting as this would help understand which property of the superplasticiser determines its efficiency and performance.

Regulated standards for the conformity criteria for the mini-slump and column segregation tests results need to be explored. Currently, only recommendations by other researchers are used as a guideline.

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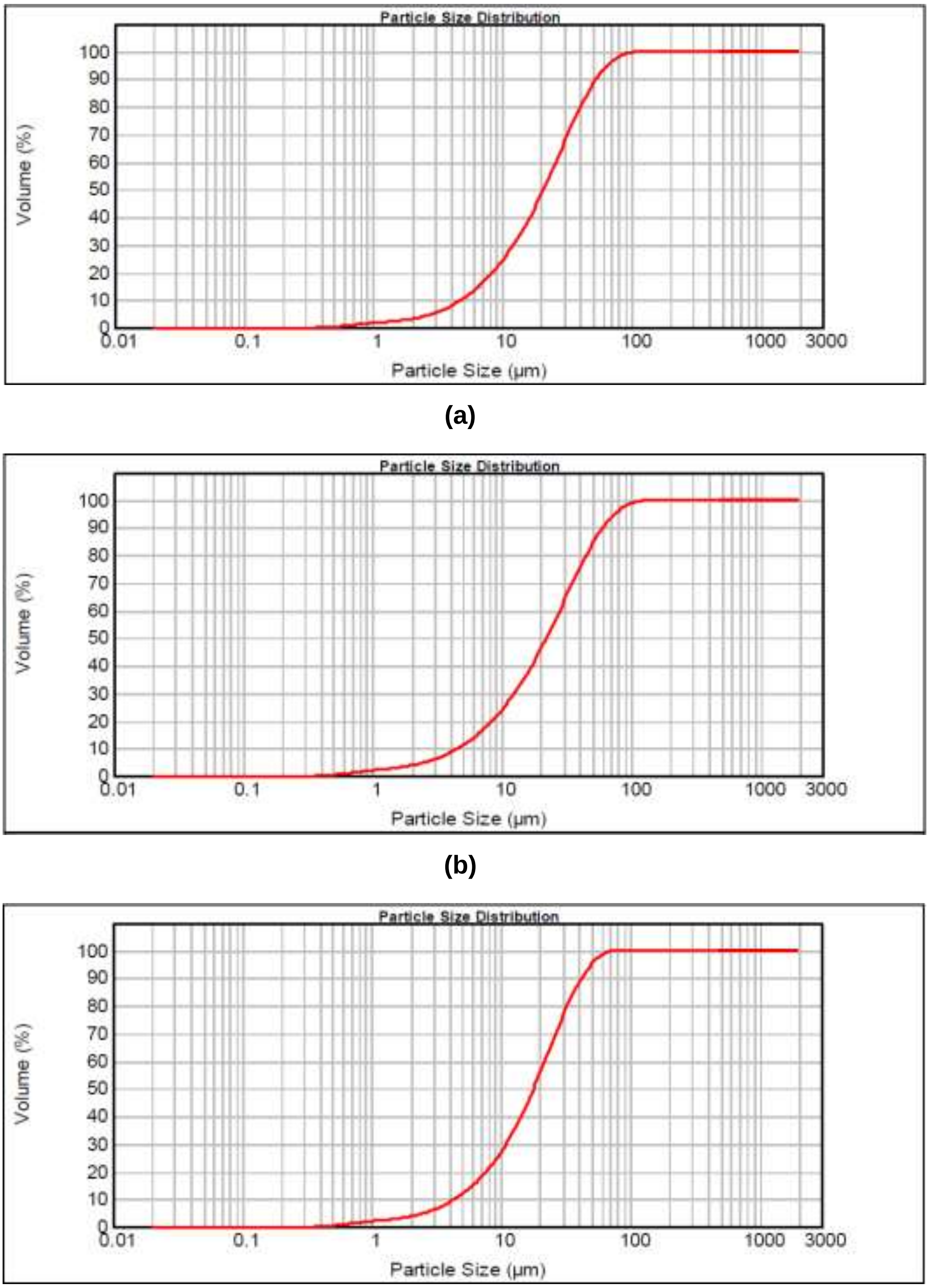
APPENDIX/APPENDICES

APPENDIX A : Cement particle size distribution analysis



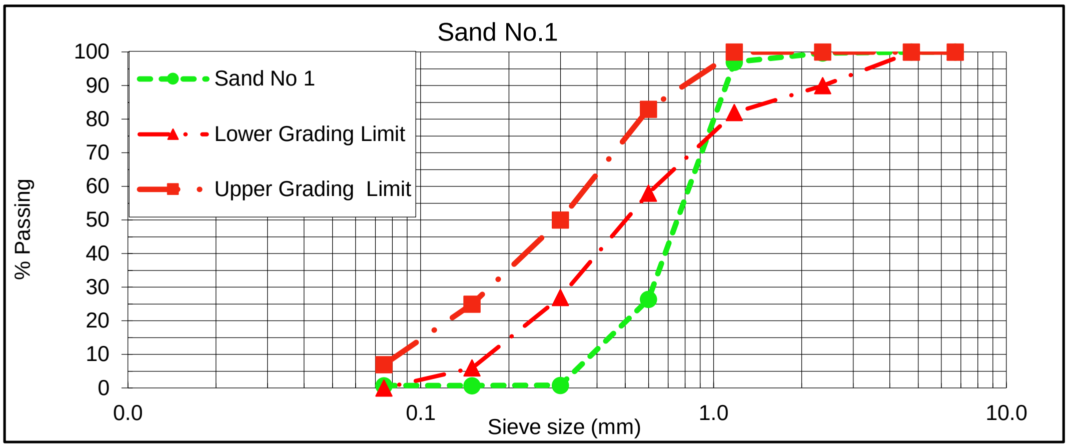
(a)

(b)

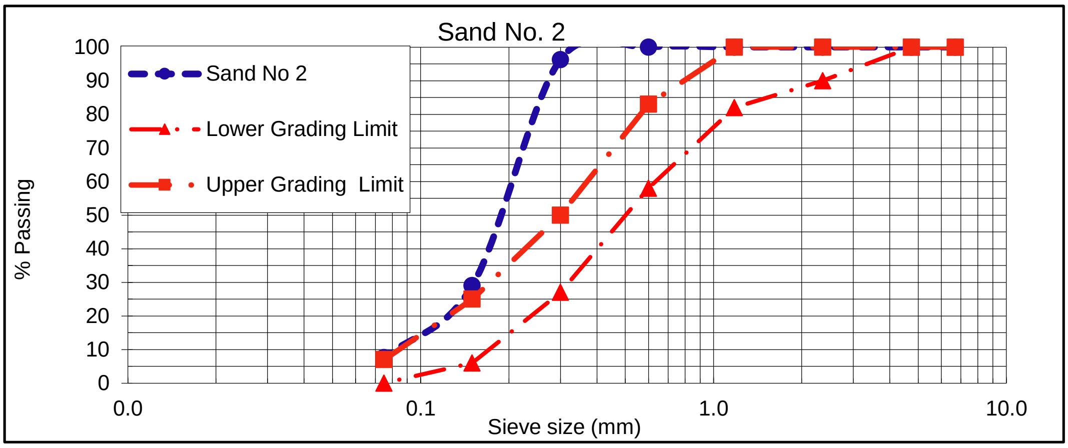
(c)

Figure A1: Particle size distribution of cement (a): CEM A; (b): CEMB and (c): CEMC as obtained from Malvern Mastersizer 2000 instrument

APPENDIX B: Sand particle size distribution analysis



(a)



(b)

Figure B1: Sand particle size distributions assessment (a): Sand No.1 and (b): Sand No.2

APPENDIX C: Slump flow test for cement mortar in the presence of SP3 with different fraction of SP1 and SP2

Table C1: Slump flow measurement and derivative yield stress values of cement mortars with CEM A in the presence of SP3 at 0.9%

SP3 composition	Slump Flow 1	Slump Flow 2	Slump Flow 3	Calculated Yield stress
10%SP2-90%SP1	64	73	71	1.83
20%SP2-80%SP1	56	70	76	1.71
30%SP2-70%SP1	67	74	80	1.31
40%SP2-60%SP1	77	88	82	0.80
50%SP2-50%SP1	88	84		0.75
60%SP2-40%SP1	50	95	100	0.41
70%SP2-30%SP1	111	116		0.18
80%SP2-20%SP1	68	103	109	0.26
90%SP2-10%SP1	76	73		1.59

Table C2: Slump flow measurement and derivative yield stress values of cement mortars with CEM B in the presence of SP3 at 0.9%

SP3 composition	Slump Flow 1	Slump Flow 2	Slump Flow 3	Calculated Yield stress
10%SP2-90%SP1	79	81	80	1.08
20%SP2-80%SP1	47	79	84	1.01
30%SP2-70%SP1	70	110	110	0.22
40%SP2-60%SP1	64	125	129	0.11
50%SP2-50%SP1	89	134	130	0.09
60%SP2-40%SP1	88	138	138	0.07
70%SP2-30%SP1	72	137	137	0.07
80%SP2-20%SP1	97	140	134	0.07
90%SP2-10%SP1	85	134	130	0.09

Table C3: Slump flow measurement and derivative yield stress values of cement mortars with CEM C in the presence of SP3 at 0.9%

SP3 composition	Slump Flow 1	Slump Flow 2	Slump Flow 3	Calculated Yield stress
10%SP2-90%SP1	76	82	82	1.08
20%SP2-80%SP1	77	88	82	0.95
30%SP2-70%SP1	77	87	84	0.90
40%SP2-60%SP1	99	83	86	0.80
50%SP2-50%SP1	82	90	87	0.75
60%SP2-40%SP1	103	97	99	0.35
70%SP2-30%SP1	132	141	136	0.08
80%SP2-20%SP1	142	149	145	0.06
90%SP2-10%SP1	143	142		0.06

APPENDIX D: Yield stress and viscosity value of cement paste with different cements in the presence of SP3

Table D1: Yield stress values of cement paste with CEM A in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	Yield stress 1	Yield stress 2	Yield stress 3	Average Yield stress
10%SP2-90%SP1	0.63	0.62	0.61	0.62
20%SP2-80%SP1	0.61	0.61	0.61	0.61
30%SP2-70%SP1	0.60	0.60		0.60
40%SP2-60%SP1	0.51	0.54	0.52	0.52
50%SP2-50%SP1	0.50	0.49	0.51	0.50
60%SP2-40%SP1	0.47	0.51		0.49
70%SP2-30%SP1	0.40	0.51		0.46
80%SP2-20%SP1	0.46	0.44		0.45
90%SP2-10%SP1	0.75	0.97	0.88	0.87

Table D2: Yield stress values of cement paste with CEM B in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	Yield stress 1	Yield stress 2	Yield stress 3	Average Yield stress
10%SP2-90%SP1	0.45	0.45	0.45	0.45
20%SP2-80%SP1	0.41	0.41	0.41	0.41
30%SP2-70%SP1	0.40	0.40	0.40	0.40
40%SP2-60%SP1	0.37	0.37	0.37	0.37
50%SP2-50%SP1	0.36	0.36	0.36	0.36
60%SP2-40%SP1	0.32	0.32	0.32	0.32
70%SP2-30%SP1	0.30	0.30	0.30	0.30
80%SP2-20%SP1	0.27	0.27	0.27	0.27
90%SP2-10%SP1	0.2	0.25	0.25	0.23

Table D3: Yield stress values of cement paste with CEM C in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	Yield stress 1	Yield stress 2	Yield stress 3	Average Yield stress
10%SP2-90%SP1	0.92	0.92	0.92	0.92
20%SP2-80%SP1	0.75	0.75	0.75	0.75
30%SP2-70%SP1	0.59	0.59	0.59	0.59
40%SP2-60%SP1	0.52	0.52	0.52	0.52
50%SP2-50%SP1	0.44	0.44	0.44	0.44
60%SP2-40%SP1	0.32	0.32	0.32	0.32
70%SP2-30%SP1	0.31	0.31	0.31	0.31
80%SP2-20%SP1	0.29	0.29	0.29	0.29
90%SP2-10%SP1	0.27	0.27	0.27	0.27

Table D4: Viscosity values of cement paste with CEM A in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	Viscosity 1	Viscosity 2	Viscosity 3	Average Viscosity
10%SP2-90%SP1	0.09	0.08	0.08	0.08
20%SP2-80%SP1	0.07	0.09	0.07	0.08
30%SP2-70%SP1	0.09	0.09		0.09
40%SP2-60%SP1	0.12	0.09	0.13	0.11
50%SP2-50%SP1	0.10	0.10	0.13	0.11
60%SP2-40%SP1	0.12	0.10		0.11
70%SP2-30%SP1	0.12	0.10		0.11
80%SP2-20%SP1	0.29	0.33		0.31
90%SP2-10%SP1	0.17	0.06	0.09	0.11

Table D5: Viscosity values of cement paste with CEM B in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	Viscosity 1	Viscosity 2	Viscosity 3	Average Viscosity
10%SP2-90%SP1	0.09	0.08	0.19	0.12
20%SP2-80%SP1	0.14	0.20	0.27	0.20
30%SP2-70%SP1	0.17	0.23	0.25	0.21
40%SP2-60%SP1	0.30	0.22	0.28	0.27
50%SP2-50%SP1	0.24	0.27	0.30	0.27
60%SP2-40%SP1	0.28	0.30	0.27	0.28
70%SP2-30%SP1	0.29	0.28	0.29	0.29
80%SP2-20%SP1	0.32	0.30	0.30	0.31
90%SP2-10%SP1	0.30	0.29	0.33	0.31

Table D6: Viscosity values of cement paste with CEM C in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	Viscosity 1	Viscosity 2	Viscosity 3	Average Viscosity
10%SP2-90%SP1	0.27	0.25	0.28	0.27
20%SP2-80%SP1	0.28	0.27	0.30	0.28
30%SP2-70%SP1	0.29	0.27	0.32	0.30
40%SP2-60%SP1	0.33	0.29	0.27	0.30
50%SP2-50%SP1	0.30	0.31	0.28	0.30
60%SP2-40%SP1	0.34	0.34	0.33	0.34
70%SP2-30%SP1	0.35	0.38	0.30	0.34
80%SP2-20%SP1	0.37	0.33	0.38	0.36
90%SP2-10%SP1	0.38	0.39	0.38	0.38

APPENDIX E: Bleeding of cement mortars with SP3 at 0.9%

Table E1: Bleeding values of cement mortar with CEM A in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	$Vw\ 1$	$Vw\ 2$	Bleeding 1 (%)	Bleeding 2 (%)	Bleeding Value
10%SP2-90%SP1	0	0	0.00%	0.00%	0.00%
20%SP2-80%SP1	0	0	0.00%	0.00%	0.00%
30%SP2-70%SP1	0	0	0.00%	0.00%	0.00%
40%SP2-60%SP1	0	0	0.00%	0.00%	0.00%
50%SP2-50%SP1	0	0	0.00%	0.00%	0.00%
60%SP2-40%SP1	0	0	0.00%	0.00%	0.00%
70%SP2-30%SP1	0	0	0.00%	0.00%	0.00%
80%SP2-20%SP1	0	0	0.00%	0.00%	0.00%
90%SP2-10%SP1	0	0	0.00%	0.00%	0.00%

Table E2: Bleeding values of cement mortar with CEM B in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	$Vw\ 1$	$Vw\ 2$	Bleeding 1 (%)	Bleeding 2 (%)	Bleeding Value
10%SP2-90%SP1	0	0	0.00%	0.00%	0.00%
20%SP2-80%SP1	0	0	0.00%	0.00%	0.00%
30%SP2-70%SP1	0	0	0.00%	0.00%	0.00%
40%SP2-60%SP1	0	0	0.00%	0.00%	0.00%
50%SP2-50%SP1	0	0	0.00%	0.00%	0.00%
60%SP2-40%SP1	0	0	0.00%	0.00%	0.00%
70%SP2-30%SP1	0	0	0.00%	0.00%	0.00%
80%SP2-20%SP1	0	0	0.00%	0.00%	0.00%
90%SP2-10%SP1	0	0	0.00%	0.00%	0.00%

Table E3: Bleeding values of cement mortar with CEM C in the presence of SP3 at 0.9% with different fraction of superplasticisers

SP3 composition	$Vw\ 1$	$Vw\ 2$	Bleeding 1 (%)	Bleeding 2 (%)	Bleeding Value
10%SP2-90%SP1	0	0	0.00%	0.00%	0.00%
20%SP2-80%SP1	0	0	0.00%	0.00%	0.00%
30%SP2-70%SP1	0	0	0.00%	0.00%	0.00%
40%SP2-60%SP1	0	0	0.00%	0.00%	0.00%
50%SP2-50%SP1	0	0	0.00%	0.00%	0.00%
60%SP2-40%SP1	0	0	0.00%	0.00%	0.00%
70%SP2-30%SP1	0	0	0.00%	0.00%	0.00%
80%SP2-20%SP1	10	9	1.25%	1.13%	1.20%
90%SP2-10%SP1	5	5	0.62%	0.63%	0.60%

APPENDIX F: Segregation of cement mortars with SP3 at 0.9%

Table F1: Segregation of cement mortars for Cement A with SP3 at 0.9%

SP3 composition	SI 1 (%)	SI 2 (%)	Average
10%SP2-90%SP1	7%	7%	7%
20%SP2-80%SP1	8%	6%	7%
30%SP2-70%SP1	6%	7%	6%
40%SP2-60%SP1	7%	7%	7%
50%SP2-50%SP1	8%	7%	8%
60%SP2-40%SP1	7%	6%	6%
70%SP2-30%SP1	13%	12%	12%
80%SP2-20%SP1	10%	10%	10%
90%SP2-10%SP1	4%	5%	4%

Table F2: Segregation of cement mortars for Cement B with SP3 at 0.9%

SP3 composition	SI 1 (%)	SI 2 (%)	Average
10%SP2-90%SP1	5%	5%	6%
20%SP2-80%SP1	5%	5%	5%
30%SP2-70%SP1	13%	16%	14%
40%SP2-60%SP1	9%	11%	10%
50%SP2-50%SP1	4%	4%	4%
60%SP2-40%SP1	5%	4%	4%
70%SP2-30%SP1	9%	8%	9%
80%SP2-20%SP1	13%	15%	14%
90%SP2-10%SP1	23%	27%	25%

Table F3: Segregation of cement mortars for Cement C with SP3 at 0.9%

SP3 composition	SI 1 (%)	SI 2 (%)	Average
10%SP2-90%SP1	5%	5%	5%
20%SP2-80%SP1	6%	5%	5%
30%SP2-70%SP1	5%	5%	5%
40%SP2-60%SP1	5%	5%	5%
50%SP2-50%SP1	8%	7%	8%
60%SP2-40%SP1	10%	12%	11%
70%SP2-30%SP1	13%	14%	14%
80%SP2-20%SP1	65%	75%	70%
90%SP2-10%SP1	63%	65%	64%