MARYLINDA SANTOS DE FRANÇA

**RHEOLOGICAL BEHAVIOR OF ENGINEERED CEMENTITIOUS
COMPOSITES REINFORCED WITH PVA FIBERS**

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Prof. Dr. Holmer Savastano Jr.

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To my love

Eduardo

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ABSTRACT

The rheological behavior analysis of Engineered Cementitious Composites (ECC) is key to understand how the different preparation techniques affect the composite mechanical performance. However, the rheological assessment of reinforced materials becomes more complex since fibers usually cause flow disturbances not found in non-reinforced cementitious materials. Besides that, simple workability measurement techniques are not able to fully understand the composite behavior in the fresh state creating the need for more precise techniques to be employed. The main objectives of this study were to evaluate the ECC rheological behavior using different rheometer devices (Vane system and Ball measuring system) and investigate the influence of mixing processes on the fiber homogenization and rheological behavior. Additionally to this, a link between rheological behavior and mechanical performance was investigated. In the end, the ball measuring system revealed to be more efficient than the vane system when evaluating the composite rheological behavior. In addition, the mixing process influenced the rheological behavior of PVA-ECC especially regarding the moment which fibers are added. Fiber addition after mortar mixture improved fibers homogenization and reduced mixing energy by around 8%. Moreover, a correlation between rheological and mechanical properties showed that a 2-times variation in either yield stress or viscosity can lead to a variation of more than 50% in flexural strength without significantly affecting the composite compressive strength. It was also found that the lower the composite yield stress and viscosity the higher was its ultimate strain. To conclude, all those parameters contributed to understand the composite rheological behavior and globally optimize its performance.

Keywords: Rheology. Rheometry. Mixing. Engineered cementitious composites (ECC). Polyvinyl alcohol (PVA) fibers.

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1

Introduction

The current research work focuses on studying the rheology of Engineered Cementitious Composites (ECC) reinforced with polyvinyl alcohol (PVA) fibers. It is well known that the rheological behavior is important to understand the composites in the fresh state since it affects the composite mechanical performance. In addition, if materials are not efficiently mixed and fibers well distributed, a series of problems can occur as a result like variability of the tensile strain capacity for instance.

Many researchers have previously used different techniques and equipment to study the rheology of cementitious materials. Especially for reinforced materials, the subject of most works is steel fiber reinforcement and, in particular for ECC mortars, the rheological behavior analysis usually refers to the material before fibers addition or it makes use of simple and empirical test methods. In order to fill in this gap to further understand and evaluate the rheological behavior of fiber reinforced systems, the research uses advanced techniques of rheometry.

To accomplish that, three relevant themes are discussed in the experimental program. First the techniques to measure the rheological behavior of ECC mortars. Then the evaluation of different mixing methods and the mixing sequence impact on fibers homogenization. Last but not least, possible correlations between rheological behavior and mechanical performance are investigated.

Engineered Cementitious Composites (ECC), also called Strain-hardening Cement-based Composite (SHCC), exhibit strain-hardening behavior when subjected to uniaxial tensile loading. This happens through the bridging of fine multiple cracks due to the use of short and well-distributed fibers. This distinct mechanical property of SHCC offers many possible applications as mentioned by (LI et al., 2000; FISCHER; LI, 2007; LI, 2008; MECHTCHERINE et al., 2011; MECHTCHERINE,

2013; SECRIERU et al., 2016). Nonetheless, strain-hardening behavior and its respective application is only enhanced if fibers are uniformly dispersed through the material. As such previous studies demonstrated that variations in composite tensile ductility are related to the influence of rheological behavior during processing (LI; LI, 2013) as well as the matrix flowability and mixing efficiency (FELEKOĞLU; TOSUN; BARADAN, 2009; FELEKOGLU et al., 2014).

The rheological behavior of reinforced cementitious materials plays indeed a very important role on the characterization in their fresh state. Unfortunately the literature about fiber reinforced systems is not abundant. Most research works in this area are focus on steel fiber reinforcement and reach the conclusion that fibers addition decreases the composite workability (MARTINIE; ROSSI; ROUSSEL, 2010). The same happens regarding the rheological properties of ECC mortars. There are only a few research works about the rheological properties of fresh SHCC (KONG; BIKE; LI, 2003a, 2003b; LI; LI, 2013; SECRIERU et al., 2016). Furthermore, their conclusions are based on the rheological behavior of matrices without fibers (ŞAHMARAN et al., 2013; LI; LI, 2013) or on simple measurement analysis such as mini-slump test and Marsh cone flow rate test. As reported by (LINDSTRÖM; UESAKA, 2009), in the presence of fibers no linear flow gradient can exist because they cause flow disturbances. Consequently, rheological measurements taking only into account the mortar or paste, can hardly match the rheological behavior of the composite. For this reason, the composite rheological behavior analysis is important to fully understand the composite properties in its fresh state.

Furthermore, fibers addition leads to loss of workability depending on fiber content, features, distribution and orientation in the matrix as well as fiber-fiber and fiber-matrix interactions (GRÜNEWALD; WALRAVEN, 2001; KESHTKAR; HEUZEY; CARREAU, 2009). During mixing, these fiber-related interactions make dispersion and homogenization processes more difficult due to the formation of fibers-particles and even fiber-fiber agglomerates. When trying to reduce or avoid the perturbations caused by fiber interactions during mixing, a closer look into the sequence of materials addition is necessary in order to optimize the processing of cement-based composites.

In this context the current work relies on knowledge of three main areas which are rheology, mixing and mechanical performance to correctly asses and optimize

the composite fresh properties and thus evaluate the impact of such properties on the composite mechanical behavior.

1.1. RESEARCH OBJECTIV

This research work focuses on rheometric systems and methods to determine the rheological behavior of Engineered Cementitious Composites (ECC) reinforced with PVA (polyvinyl alcohol) fibers in order to achieve strain hardening mechanical performance (multi-cracking and improvement of deformation/strain without losing load/stress capacity) of ECC mortars. The impact of PVA fibers addition on the mixing process is also evaluated by analyzing mixing time, mixing intensity, and amount of air trapped into the matrix. Furthermore, correlations between mortar fluidity before fiber addition and mixing time with fibers using admixtures for fiber dispersion are investigated besides proposing a simple method to quantify fibers homogenization in the fresh state. In the end it is possible to evaluate a link between the composite rheological behavior and its mechanical properties. Last but not least, after an in depth analysis of the results it should be possible to define a processing routine that globally improves the composite performance.

1.2. RESEARCH OVERVIEW

This research work starts with an introduction to the rheology of ECC mortars reinforced by PVA fibers in which the current knowledge about strain-hardening behavior and the rheometry of reinforced systems is summarized (*Chapter 2*).

A preliminary study about mixing sequences evaluated via rotational rheometry is discussed in *Chapter 3*. This chapter also explains the main concepts behind mixing processes with fibers besides establishing a relation between mixing behavior and rheology.

Chapter 4 aims to develop suitable techniques to evaluate the rheological behavior of PVA-ECC mortars. For this purpose, two different techniques are applied: vane test and ball measuring system. The follow up analysis then compares the rheological parameters from those two rheometer configurations regarding shear stress, viscosity, static yield stress and thixotropy. Possible limitations and

advantages in using each test configuration to measure PVA-ECC mortars are also discussed.

In *Chapter 5*, investigations are made regarding the influence of mixing processes when producing ECC mortars regarding its effect on fibers homogeneity. A correlation between mixing process, rheological properties and fibers homogenization is as well subject of this chapter.

A link between the rheological behavior and the mechanical performance of fiber reinforced mortars is the target of *Chapter 6*. The effect of mixing procedure, mixer type and mortar fluidity on the composite mechanical performance and rheological behavior is evaluated. Through the combined analysis of those different parameters, correlations between mechanical performance and rheological behavior can be found.

As for the reference matrix composition used, the composite did not show clear strain-hardening behavior. For this reason, investigations regarding new mix designs are proposed. As such, *Chapter 7 evaluates* the effects of mix design and fiber content on the composite properties. The strain-hardening properties are then evaluated using uniaxial tension test setup and the results are compared to the composite rheological behavior.

Chapter 8 summarizes the most important contributions by this study. Any remaining gaps in theoretical and experimental work that may lead to further research are also identified.

2

Theoretical considerations

2.1 INTRODUCTION

This chapter discusses the main theoretical considerations in terms of Strain-hardening Cement-based Composites (SHCC), also named Engineered Cementitious Composites (ECC) reinforced by polyvinyl alcohol (PVA) fibers. Relevant aspects of this type of composite like processing, rheological properties, fiber characteristics and mechanical performance are the target of this chapter.

At the beginning a brief explanation about Engineered Cementitious Composites (ECC), their main characteristics and applicability, is presented. Furthermore the composite mixing behavior analysis is reported based on the current knowledge. Also, techniques used to verify the mixing behavior of cementitious materials are explained and their viability when applied to fiber reinforced systems is discussed. Meanwhile, important topics for cementitious materials rheology analysis, rheometric techniques as well as the most suitable equipment for those measurements are introduced.

To conclude, this chapter shows how the main fiber characteristics reported in the literature affect the composite properties. Finally, the composite mechanical performance analysis is done.

2.2 STRAIN-HARDENING CEMENT-BASED COMPOSITES

Strain-hardening Cement-based Composites (SHCC) or Engineered Cementitious Composites (ECC) is a class of high-performance fiber-reinforced cementitious composites (HPFRCC) featuring high intrinsic tensile ductility and moderate fiber content (generally 2% or less by volume) (YANG; YANG; LI, 2007; LI, 2008; LI; LI, 2013). There are many works in the scientific literature discussing ECC based on PVA fiber but other fibers have also been used such as polyethylene (PE) and polypropylene (PP) (LI, 2008; MECHTCHERINE et al., 2011; YUN, 2013; CUROSU; MECHTCHERINE; MILLON, 2016). SHCC exhibits tensile strain-hardening behavior through multiple micro-cracking with self-controlled crack width, leading to fracture toughness (MAALEJ; HASHIDA; LI, 1995). The tensile performance of SHCC depends on the fiber and flaw size distributions. The matrix flowability and mix processing has also great effect on these parameters (TOSUN-FELEKOĞLU et al., 2014).

Due to the distinct SHCC mechanical properties, there are many possible applications for this composite such as link slabs in bridge decks to improve fatigue resistance, building dampers, retaining walls, surface repair of dams and irrigation channels and surface repair of viaducts for carbonation retardation. This class of composites is characterized by strain hardening and multiple cracking properties in uniaxial tension and by ultimate tensile strain capacity (FISCHER; LI, 2007). The tensile strain capacity can vary according to the processing materials (LI; WANG, 2006). The variability of the measured tensile strain capacity, ultimate tensile strength, width and density of micro-cracks depends on the type of mixer, mixing procedure and local ingredients characteristics (LI; LI, 2013). Consequently, robust composite properties can be obtained through a more uniform fiber dispersion and the rheology of ECC mortars is controlled to fall within this optimal range (LI; LI, 2013).

2.3 MIXING PROCESS

Mixing processes highly influence the fresh and hardened properties of cement composites reinforced with fibers. Mixing sequence, duration, intensity as well as mixer type have direct effect on the particles homogenization of the system.

During mixing, the formation of agglomerates occurs when the liquid (water) gets in contact with the solid particles, increasing the necessary effort for mixing until a point of maximum cohesion between particles is achieved. At this point, the mixing effort is high and there is a competition between forces of agglomerate formation and their breakage. The agglomerates are destroyed by impact and shear energy provided by the mixing equipment, thus promoting the dispersion of fines and aggregates as well as the homogenization of the solid particles among them and within water (PILEGGI et al., 2001; LEUENBERGER et al., 2009; CAZACLIU; ROQUET, 2009; CAVINATO et al., 2010).

The effort, required during the mixing process can be assessed by special rheometers capable of withstand and measure the torque levels generated by mixing highly concentrated suspensions (like mortars and concretes) (PILEGGI et al., 2001; DOMICIANO et al., 2003; CAVINATO et al., 2010). The effort can also be measured by mixers equipped to adequately quantify the energy consumption (LOWKE D; SCHIESSL P, 2005; LEUENBERGER et al., 2009; CAZACLIU; ROQUET, 2009; MAZANEC; LOWKE; SCHIEßL, 2010; RONDET et al., 2012) which also allows identifying the moment when the material starts to behave like a fluid (CAZACLIU; LEGRAND, 2008), i.e. the fluidity point. This point is stated in the mixing curve as the moment at which the torque levels start to stabilize and the first derivative of the curve tends asymptotically to zero (FRANÇA, 2012).

Mixing stages can be assessed by torque vs. time curves while shear cycles performed immediately after mixing provide information regarding the effects of mixing on the rheological behavior of the material, Figure 2.1.

Figure 2.1 – Torque vs. time curve at different mixing times (on the left) and rheological behavior (on the right). The rheological test is performed during 105 s immediately after mixing in the same equipment (FRANÇA; CARDOSO; PILEGGI, 2016).

Figure 2.1, each red point on the curve indicates a mixing time when the rheological profile of a mortar was evaluated by a shear cycle (accelerating and decelerating with steps at different rotation speeds). It is noted that the shorter the mixing time the larger the difference between acceleration and deceleration curves (hysteresis) indicating that the system was still rheologically unstable because the mixing was incomplete. Therefore the mortar needed additional energy – provided by the shear cycle – to proceed with the dispersion and homogenization and thus achieve a stable state. For longer mixing times the hysteresis area gets progressively smaller until it reaches zero or almost zero, which indicates that the procedure provided enough energy for an adequate mixing that produced a rheologically stable mortar for the experimental conditions applied.

Processing of reinforced materials should target an ideal rheology in order to optimize fiber dispersion thus minimizing the probability of creating “weak” sections with lower fiber content (LI; LI, 2013).

2.3.1 Mixing Sequence

During mixing, the fiber-related interactions make the dispersion and homogenization processes more difficult due to the formation of fiber-particle and

even fiber-fiber agglomerates. In order to reduce or avoid the perturbations caused by fiber interactions during mixing, some authors have suggested different mixing methods and sequences of raw materials addition to improve the processing of cementitious composites.

For mortars reinforced with polypropylene (PP) fibers, it was found that the sequence of materials addition and mixing time have influence on the homogeneity of the obtained mortar (SILVA, 2006). Others (AKKAYA; SHAH; ANKENMAN, 2001) showed that mixing PVA fibers with liquid before adding them to the particulate materials affected fiber dispersion and consequently the toughness of the composite.

A better dispersion of sand and fibers in fiber reinforced concretes can also be achieved by mixing the steel fibers with the dry particulate materials beforehand (KUDER et al., 2007; LASKAR; TALUKDAR, 2008). The addition of PVA fibers at the beginning of the mixing process was also investigated by (SHEN et al., 2008; ZHOU et al., 2013) in highly concentrated suspensions. The same mix procedure was performed by (SAID; RAZAK; OTHMAN, 2015) to produce Engineered Cementitious Composites (ECC) slabs and by (CUROSU; MECHTCHERINE; MILLON, 2016) while investigating strain-hardening cement-based composites (SHCCs) using high-density polyethylene fibers.

In relation to refractory concrete technology, some authors have studied alumina-based concretes with PP fiber addition by considering two types of processing: (i) mixing fibers with dry materials and (ii) addition of fibers into the, already mixed, fluid concrete (DOMICIANO et al., 2003). The study (DOMICIANO et al., 2003) has achieved to the addition method which resulted in different shear levels and damage to the fibers. In particular, damage was lower for shorter fibers added after the concrete was already mixed (DOMICIANO et al., 2003).

Another study also showed better results when fibers were mixed in the final processing stage, after all particulate materials have already been mixed with water (KIM; BOYD; LEE, 2011; FARES et al., 2015). The cementitious composites reinforced with PVA fibers obtained through this mixing method presented a more homogeneous fiber distribution, improved mechanical properties, and reduced shrinkage (KIM; BOYD; LEE, 2011). Furthermore, strain-hardening cementitious composites (SHCC) were produced by adding fibers after achieving the desired matrix flowability determined previously by Marsh cone flow test (KONG; BIKE; LI,

2003a; LI; LI, 2013; FELEKOGLU et al., 2014). Similar mix design was adopted by (MECHTCHERINE et al., 2012; YUN, 2013; PAN et al., 2015; CHOI et al., 2015) where PVA fibers were added into the mortar mixing once a consistent mixture was reached.

Another approach to improve the fiber distribution and mechanical properties of ECC mortars is adjusting the mixing sequence (ZHOU et al., 2012). The mix of solid materials with liquid is divided into two steps with the fibers addition taking place between them. The results show that adjustments in the mixing sequence increase the tensile strain capacity and ultimate tensile strength of ECC. It also improves the fiber distribution (ZHOU et al., 2012).

In another study, different mixing methods were evaluated to determine their effectiveness on the distribution of nanomaterials within the cementitious matrices (AL-DAHAWI et al., 2016). The authors concluded that mechanical mixing at very high speeds resulted in a more uniform distribution of nanomaterials in the presence of HRWA admixture (AL-DAHAWI et al., 2016).

In summary, different fibers addition sequences (either at the beginning of mixing process, or in between the steps or at the end of mixing process) must take into account the fiber feature, matrix constituents besides the equipment to prepare the mixture. More specifically, for cementitious composites reinforced with PVA fibers, different mixing sequences have been adopted to enhance their performance.

2.3.2 Mixing time

Usually the mixing time is a parameter closely associated to other parameters of the mixing process such as mixing sequence and mixer efficiency. As a general rule there is not a perfect way to process mixing with fibers but some important aspects must be taken into account. One of them, is the fiber damage during mixture which is associated to longer mixing times. Besides that, mixer efficiency is another parameter that can directly influence mixing time.

Mixing time is intrinsically related to the stabilization time during mixture. The concept of stabilization time is well understood due to previous research using power consumption curves recorded during the mixing process (CHOPIN; DE LARRARD;

CAZACLIU, 2004; CHOPIN et al., 2007; CAZACLIU; ROQUET, 2009). This way, the stabilization time is defined as the time at which the power vs. time curve reaches an asymptote (CHOPIN; DE LARRARD; CAZACLIU, 2004).

For cementitious materials without fibers, many authors have investigated the influence of mixing time and its effect on the material performance. For example, to prepare self-compacting concrete (SCC) it is shown that mixing time is an important parameter in order to make the mixture cohesive and sufficiently homogeneous to fill in the spaces between aggregates without segregation or bleeding (HEMALATHA et al., 2015). It has also been found that the maximum packing density, type of cement and superplasticizer content affect the mixing time of high performance concrete (MAZANEC; LOWKE; SCHIEßL, 2010). As seen in Figure 2.2 the stabilization time increases with the relative solid concentration $\phi/\phi_{\max}$, which is the actual concentration ratio of solids in a concrete mix ϕ to the maximum possible concentration of solids $\phi_{\max}$ obtained at the maximum packing density of the particles. This means that the amount of solid materials in a concrete mix and the maximum possible packing density of the particles are the main factors determining the necessary mixing time (MAZANEC; LOWKE; SCHIEßL, 2010). In fact, Chopin et al. (2004) also found that the fine content, admixture dosage and the use of silica fume have a significant effect on mixing time (CHOPIN; DE LARRARD; CAZACLIU, 2004).

Figure 2.2 – Effect of the relative solid fraction on the stabilization time t_s (MAZANEC; LOWKE; SCHIEßL, 2010).

In terms of reinforced materials, improvements to the matrix flow properties of ultra-high performance fiber reinforced concrete with the increase in mixing time has been observed (CAZACLIU et al., 2006). The results show that at least 5 min of wet mixing is necessary to obtain the desirable flow properties and longer mixing times do not lead to an improvement in mechanical properties (CAZACLIU et al., 2006). Furthermore, the mixing time and intensity effects on the distribution of steel fibers in ultra-high performance concrete (UHPC) have been investigated by (MAZANEC; SCHIEßL; GEHLEN, 2011). As seen in Figure 2.3, fiber distribution is significantly affected by mixing intensity and the time at which fibers are added.

Figure 2.3 – Effect of mixing time and mixing speed on the fiber dispersion (MAZANEC; SCHIEßL; GEHLEN, 2011).

With a tool speed of 2.9 m/s, the coefficients of variation decrease after about 120 s assuming it is a homogeneous mix, Figure 2.3a. In the other hand for a slower tool speed of 1.4 m/s, the mixing time necessary to reach the minimum coefficient of variation is 240 s, Figure 2.3a. Besides that, the coefficient of variation is lower with a slower mixing tool speed, Figure 2.3b. Additionally, longer mixing times do not improve fiber dispersion within this speed range. These results found by (MAZANEC; SCHIEßL; GEHLEN, 2011) show that fibers are better distributed at slow mixing intensity. The explanation for that is that steel fibers experience a larger centrifugal force than the other particles, thus fibers move through the mix towards the vessel perimeter and segregation may occur (MAZANEC; SCHIEßL; GEHLEN, 2011).

Notice that mixing time and mixing intensity are parameters intrinsically related. Hence, mixing intensity is subject of the next section.

2.3.3 Mixing intensity and mixer type

Different research works have focused on mixing intensity. For example, to prepare reinforced concretes with polyester/glass fibers two types of mixers are compared: an ordinary one with maximum speed of 180 rpm/min and another high speed mixer (Eirich with 1600 rpm/min). By doing so it is observed that the flexural

strength is slightly influenced by the mixing energy (SEBAIBI; BENZERZOUR; ABRIAK, 2014). For this setup matrix, mixer and fiber type, the higher mixer speed reduces fibers clusters and promotes better distribution regarding the loading direction. On the other hand, it increases the composite porosity content without affecting its mechanical behavior (SEBAIBI; BENZERZOUR; ABRIAK, 2014).

By evaluating the effect of fiber properties and matrix composition on the tensile behavior of strain-hardening cement-based composites (SHCC), it is found that a higher mixing energy is necessary to achieve proper fiber dispersion and to break the clumps of fibers and fines, which comes as a consequence of the low water-to-binder ratio and the high aspect ratio of the fibers (CUROSU; MECHTCHERINE; MILLON, 2016). Furthermore, mixing procedures using mixers with high shear rates tend to decrease plastic viscosity with an increase in mixing intensity (VANCE; SANT; NEITHALATH, 2015). In other words, as the mixing speed increases, the particle dispersion increases and agglomerates are reduced or destroyed. Moreover, such mixing procedures decrease inter-particle surface contacts and friction forces thus increasing the fluidity of the suspension (VANCE; SANT; NEITHALATH, 2015).

An investigation of the matrix flowability influence on the mechanical performance of Engineered Cementitious Composites (ECC) made with High Tenacity Polypropylene fibers shows that fiber clumping is reduced with a combination of three factors: longer mixing time, higher mixing speed and higher mixer torque (FELEKOGLU et al., 2014). The fast mixing speed seems to result in an optimal fiber dispersion which improves ultimate tensile strength and ductility (FELEKOGLU et al., 2014). This result depends on the mixer type used as previously discussed by (MAZANEC; SCHIEßL; GEHLEN, 2011).

Another study reported that intensive mixing is more effective in the breakdown of the agglomerates, resulting in lower yield stress, plastic viscosity and hysteresis (ROY; ASAGA, 1979). However it also showed that subjecting cement paste to high mixing intensities can increase its apparent viscosity once a certain mixing intensity threshold is achieved (HAN; FERRON, 2016). As such, this kind of cement paste shows weaker agglomeration strength and larger agglomerates than their counterpart pastes prepared at lower mixing speeds (HAN; FERRON, 2016).

In terms of mixing equipment, Chang and Peng (2001) identified that both mixer type and efficiency influence the required mixing time and superplasticizer dosage as well as the blending quality of the high performance concrete produced (CHANG; PENG, 2001). Also, different arrangements of mixer blades directly affect the homogeneity of the mix (HEMALATHA et al., 2015). For this reason, the fresh and hardened properties of SCC differ when different mixers are used although the same composition and mixing sequence are employed (HEMALATHA et al., 2015).

Another study using mixers equipped with vacuum pump able to lower the mixing pressure reported a performance improvement by lowering the air content of ultra-high performance concrete (DILS; BOEL; DE SCHUTTER, 2014). Vacuum mixing helps to reduce the total air content since the presence of air bubbles around fibers can have a negative effect on the bond strength between the reinforcement and the surrounding concrete (DILS; BOEL; DE SCHUTTER, 2014).

In summary, because of their impact in the rheological and mechanical properties, all these aspects of the mixing process should be taken into account when preparing strain-hardening cement-based Composites (SHCC). And since the mixing process has direct influence on the fiber homogenization, the rheological behavior of the composite may change impairing or improving its mechanical performance. This will be discussed in the following sections.

2.4 RHEOLOGICAL BEHAVIOR

Rheological studies of cementitious materials provide information how these materials behave in the fresh state and help monitor structural formation (i.e. setting) that has direct influence on their mechanical properties (SANT; FERRARIS; WEISS, 2008). Usually, rheological experiments of cementitious materials are carried out using a rotational rheometer. Yield stress and plastic viscosity are the two main rheological parameters that describe flow. While plastic viscosity is a measure of the flow-ability of a fluid and is influenced by inter-particle friction and surface contacts, yield stress is reported as the stress required to initiate the fluid's flow (VANCE; SANT; NEITHALATH, 2015). Therefore, yield stress τ_0 divides the material behavior into two states: with stress below τ_0 the material does not flow (i.e. behaves like a

solid), but when stress exceeds τ_0 the material flows due to inter-particle bonds breakup (MBASHA et al., 2015). The authors (MBASHA et al., 2015) use different rheological approaches to measure the yield stress of cement pastes. They showed that the yield stress obtained via flow curves is the method which gives more information about the rheological properties and structural evolution of cement pastes.

Most of the research work about rheological behavior measurements of reinforced materials is focused on steel fiber reinforced concretes. For instance, the authors (LASKAR; TALUKDAR, 2008) used a modified setup of parallel plate rheometer with a vane geometry to perform rheological measurements of steel fiber reinforced concrete. They calculated the yield stress and plastic viscosity using torque-speed relationship curves and concluded that both plastic viscosity and yield stress increase linearly with the fibers volume.

In another study, a parallel plate rheometer was also used to evaluate the rheology of stiff fiber reinforced cement paste (KUDER et al., 2007). They observed that stiff steel fibers might increase the amount of structural breakdown that occurs during mixing, reducing the yield stress and viscosity of the material. The mechanical interlocking or entangling of the fibers can dominate the flow behavior once a certain volume of fibers is reached (KUDER et al., 2007).

Furthermore, Grünewald (2004) found that fibers affect the characteristics of self-consolidating concrete (SCC) in its fresh state, i.e. slump flow decreases while the plastic viscosity (resistance to flow) increases when compared to a reference SCC without fibers (GRÜNEWALD, 2004).

Regarding fresh engineered cementitious composites (ECC) mortars containing 2% vol. of PVA fibers, an optimal Marsh cone flow time between 24 and 33 s is to be used as a guide for ECC rheology control. For this reason, viscosity and flowability are measured using the Marsh cone flow test, mini-slump test and a rotational viscometer before fibers addition (LI; LI, 2013).

In the study of carbon fiber reinforced mortar, the authors (BANFILL et al., 2006) used the Viskomat NT to investigate the effect of mixture composition, admixtures and fine particles on the mortar rheology. The results show that by

increasing the fiber volume concentration as well as the fiber length, both yield stress and plastic viscosity increase (BANFILL et al., 2006).

Another method widely used in the cement industry to measure the fresh mortar properties is the spread measured by slump flow. It is a quick method and its value is related to yield stress (τ_0) by different means found in the literature for a range of spreads. For instance, Flatt et al. (2006) show good agreement between yield stress obtained from Viskomat and the yield stress measured from cement paste spreads (FLATT; LAROSA; ROUSSEL, 2006).

The importance of the rheological characteristics of reinforced cementitious materials has been discussed over time. Nonetheless the literature on fiber reinforced systems is not abundant. Most works in this area focus on steel fiber reinforcement. Something similar happens regarding the rheological properties of fresh SHCC. There are few research works on this subject (KONG; BIKE; LI, 2003a, 2003b; LI; LI, 2013; SECRIERU et al., 2016). Furthermore, the conclusions are either based on the rheological behavior of a matrix without fibers (ŞAHMARAN et al., 2013; LI; LI, 2013) or simple workability tests such as mini-slump and Marsh cone flow rate tests. As reported by (LINDSTRÖM; UESAKA, 2009), in the presence of fibers no linear flow gradient can exist because the fibers promote flow disturbances. Consequently the rheological measurements that only take into account the mortar or paste may significantly differ from those of the composite. For this reason, the composite rheological behavior analysis has an important role when studying its properties in the fresh state.

2.4.1 Rheometers

There are many different types of rheometers used to measure the rheological behavior of cementitious materials. For example, a systematic study comparing several rheometers has been done and the results show that rheometers usually give different values for yield stress and plastic viscosity mainly because the instruments operate on different principles and within a range of different geometries (FERRARIS et al., 2001).

Despite most research works only focusing on the rheology of matrix without fibers there are special equipments built to measure the rheological properties of

fiber reinforced systems. For instance, a cone consistency test was developed to measure the rheological behavior of fiber reinforced composites (KAUFMANN et al., 2007). The results show that this method is suitable to characterize the rheological properties of such composites in a spin casting application (KAUFMANN et al., 2007). On the other hand the ball measuring system (BMS) has been employed as a viable rheometric tool for measuring the rheological behavior of large-particle fluids in the debris flow rheology (SCHATZMANN et al., 2003) as well as for the rheological analysis of self-compacting concrete (SCC) (FLEISCHMANN; GREIM, 2014; LOWKE, 2018). Furthermore, a similar ball-shaped probe configuration called Viscoprobe was used as an in-mixer measurement during concrete mixing (CAZACLIU, 2008; MOKÉDDEM, 2012).

In this work, two different rheometer devices are used to measure the rheological behavior of SHCCs: the ball measuring system and the vane geometry. As a matter of fact, in order to simplify the analysis and focus on the main issues only these two rheometer configurations will be discussed here. Vane geometry was chosen because there are many studies in cementitious materials using this geometry but not for fiber reinforced systems measurements. On the other hand the ball measuring system benefits from a different configuration that can measure fiber reinforced systems by eliminating wall effects and the sheared gap.

2.4.1.1 Ball measuring system

The ball measuring system (BMS) comprises of a sphere that is dragged across a fluid at different specified velocities while the required drag forces are measured, Figure 2.4 and Figure 2.5.

Figure 2.4 – Scheme of the ball measuring system (SCHATZMANN, 2005).

To obtain the physical properties using a ball measuring system (BMS), the measured raw data (torque and angular velocity) must be converted to rheological variables, such as shear stress and shear rate (SCHATZMANN et al., 2003). This happens because the ball measuring system does not rely on a laminar flow usually recognized by rotational rheometers (FLEISCHMANN; GREIM, 2014).

Figure 2.5 – Ball measuring system. Viskomat NT probe V0007 details on the right side. Photo: Schleibinger testing systems.

Many studies discuss the motion of rigid spheres in rheologically complex fluids, being it yield stress fluids (TABUTEAU; COUSSOT; DE BRUYN, 2007; AHONGUIO; JOSSIC; MAGNIN, 2014), viscoplastic materials described by Bingham equations (BERIS et al., 1985) or Herschel-Bulkley fluids (ATAPATTU; CHHABRA; UHLHERR, 1995; BEAULNE; MITSOULIS, 1997). Regarding cementitious materials, particularly the material composites, the fluid mechanics analysis is more complex since due to the yield stress the hydration process is remarkable important. In spite of that, it is possible to transform torque and rotational speed into shear stress and shear rate using the method developed by (BERIS et al., 1985; SCHATZMANN, 2005).

The ball measurement system has several advantages compared to conventional rheometric systems since wall slip and shear induced sedimentation are minimized using this geometry (LEEMANN; WINNEFELD, 2007).

2.4.1.2 Vane test configuration

The vane method has become a standard for measuring yield stress (τ_0) because of its simplicity and the possibility of preventing slip during shearing. Vane rheometers provide a convenient and relatively accurate way of measuring the yield properties of concentrated suspensions directly (YAN; JAMES, 1997).

The vane device (Figure 2.6), connected to a stress-controlled rheometer is fully immersed in the material and rotated at a sufficiently low shear rate to determine the maximum torque required to initiate flow. The torque is characteristic to the static yield stress of the material and has its origin in the structural breaking of bonds in flocculated systems (ASSAAD; HARB; MAALOUF, 2016).

Figure 2.6 – Vane configuration for Viskomat NT rheometer on the left side. Photo: Schleibinger testing systems. Top view of a coaxial cylinder on the right side. R_1 – vane blade radius; R_2 – container radius, r – radius of the sheared gap (KOEHLER; FOWLER, 2004).

When a vane made of either square or rectangular-shaped blades rotates inside the material it cuts a cylindrical volume. The yielding is considered to occur along the periphery of a cylindrical surface defined by the blades tips. The use of a coaxial cylindrical vane results in abrupt breakdown of material structure along with the lowest yield stress (τ_0) responses (ASSAAD; HARB; MAALOUF, 2016). The top view of a coaxial cylinder rheometer can be seen in Figure 2.6b. For calculating the yield stress values only the material within the inner and outer cylinders, called annulus is taken into consideration (KOEHLER; FOWLER, 2004). The rheometer records the torque T acting on the inner or outer cylinder and the angular velocity Ω of one cylinder relative to the other. The flow parameters can thus be derived from a constitutive equation. Koehler and Fowler (2004) show the equations derivations for Newtonian and Bingham fluids in function of viscosity, cylinder height, and cylinder radii resulting in the Reiner-Riwlin equation shown in Equation (2.1). The Reiner-Riwlin equation is an expression for a straight line where the slope is defined considering the plastic viscosity μ , cylinder height h , and cylinder radii (R_1 and R_2) while the intercept is defined in terms of yield stress τ_0 , viscosity μ , and cylinder radii (KOEHLER; FOWLER, 2004).

Equation (2.1):

$$\Omega = \frac{T}{4\pi h\mu} \left(\frac{1}{R_1^2} - \frac{1}{R_2^2} \right) - \frac{\tau_0}{\mu} \ln \left(\frac{R_2}{R_1} \right)$$

From the Equation (2.1), where T is the torque and Ω the angular velocity, Reiner-Riwlin defines a linear relationship between T and Ω resulting in the Bingham parameters to calculate plastic viscosity and yield stress.

Vane shear flow interpretation is usually based on two aspects: (1) the material is sheared along a cylindrical surface defined by the vane height and diameter and; (2) the stress distribution is uniform over the cylindrical sheared surface (ESTELLÉ et al., 2008). Consequently, vane geometry is similar to a Couette system, i.e. varying thickness of sheared material. Based on this assumption Estellé et al (2008) propose a procedure to convert torque and rotational velocity data into shear stress and shear rate relationships. The approach approximates the sheared material to a Bingham fluid in order to calculate a shear rate average in vane geometry (ESTELLÉ et al., 2008).

According to Liddell and Boger (1996), a typical vane consists of four thin blades arranged at equal angles around a small cylindrical shaft (LIDDEL; BOGER, 1996). A typical vane has also a rate controlled mode in which a constant rotational speed is applied to the blades immersed in the material and the resulting stress is measured as a function of time. For this reason, vane geometry offers an important advantage over conventional rheometer geometry (i.e. cone and plate, parallel plate, concentric cylinder) in the yield stress measurement since yielding occurs along a localized cylindrical surface circumscribed by the vane which reduces problems with slip flow. Another advantage of the vane geometry refers to its insertion into the suspension. The authors (LIDDEL; BOGER, 1996) suggest that it does not cause any significant disturbance to the sample prior to measurement. However, this affirmation does not completely apply to stiff suspensions with high cohesiveness such as some fiber reinforced systems. Moreover, fiber orientation can occur along the flow promoting segregation and wall effect.

As another example why vane geometry is commonly used to measure the rheological behavior of cementitious materials, SCC flow curves were evaluated from vane geometry rheometer and a comparison among rheological parameters and spreading and V-Funnel tests found very satisfactory correlations (LANOS; ESTELLÉ, 2016). Furthermore, vane configuration was also used to evaluate the fresh properties of SHCC (SECRIERU et al., 2016). A Couette rotational viscometer was used to perform those rheological tests. To do the measurements they also took

into consideration the fact that the length of the thin, very flexible PVA fibers equal to 12 mm, is considerably smaller than the minimum required lateral gap size of $4.L_{\max}$ ($L_{\max}$ refers to the length of the fiber) (SECRIERU et al., 2016).

2.4.2 Thixotropy

Thixotropy is defined as the continuous decrease of viscosity with time when flow is applied to a sample that has been previously at rest and the subsequent recovery of viscosity with time when the flow is discontinued (MEWIS; WAGNER, 2009). In other words, thixotropy reflects the time taken to move away from any microstructure state and back again. The driving force behind this microstructural change in flow results from the competition between flow stress break-down and in-flow build-up due to collisions and Brownian motion (BARNES, 1997).

In practice, it means that a concrete is called thixotropic if it seems to quickly flocculate at rest and apparently becomes more fluid while flowing. According to Roussel (2006), a thixotropic concrete displays flocculation and de-flocculation characteristics in the shear rate range 1 to 10 s^{-1} from (ROUSSEL, 2006). Furthermore, a classification for SCC is proposed in terms of thixotropic behavior according to their flocculation rate, i.e. A_{thix} (Pa/s). Non-thixotropic SCC shows A_{thix} less than 0.1 Pa/s while for thixotropic SCC A_{thix} it stays between 0.1 and 0.5 Pa/s. A highly thixotropic SCC is found when A_{thix} is higher than 0.5 Pa/s (ROUSSEL, 2006). The thixotropy has also been linked to the kind of element casting, wall or slab for instance (ROUSSEL, 2007). For wall casting, a highly thixotropic SCC which is also highly viscous and stable, is required in order to decrease formwork pressure and increase the mixture stability. Conversely, a low thixotropy SCC with a low plastic viscosity, is more appropriate for slab casting (ROUSSEL, 2006, 2007; ROUSSEL; CUSSIGH, 2008).

In order to investigate the mechanism responsible for the thixotropy of self-compacting concretes, a model describing the thixotropic structural build-up based on inter-particle interactions and hydration kinetics was developed (LOWKE, 2018). According to this model, the contribution of both colloidal surface interactions and hydration reactions to thixotropy increase with surface coverage decrease thus promoting particle aggregation. Moreover, thixotropy increases with contact

interactions resulting in either an increase of solid volume fraction or lower maximum packing density (LOWKE, 2018).

Usually thixotropy has been quantified experimentally by measuring the thixotropic loop in stress/ shear rate curves Figure 2.7a or by measuring the apparent yield stress evolution at rest Figure 2.7b.

Figure 2.7 – Examples of experimental quantification of thixotropy. a) Thixotropic loop obtained by successively submitting the material to increasing and decreasing shear rate ramps (ROUSSEL, 2006). b) Thixotropy measured from the apparent yield stress evolution at rest (LOWKE, 2013).

Thixotropic loop (Figure 2.7a) is a method very dependent on test apparatus and procedure as found in (BANFILL; SAUNDERS, 1981). According to Roussel

(2006), during the thixotropic loop test de-flocculation and flocculation occur but not quickly enough to reach the steady state shear stress leading to super-estimation or sub-estimation of the measured stress (ROUSSEL, 2006).

Thixotropy can also be measured as shown in Figure 2.7b by increasing the apparent yield stress at rest via a slowly rotating rheometer (BILLBERG, 2006; LOWKE, 2013). In this setup, the thixotropy A_{thix} (defined as the rate of change of yield stress) is given by the first derivative of apparent yield stress as a function of time at rest (LOWKE, 2018).

2.5 FIBER CHARACTERISTICS

2.5.1 Fiber type

There are various types of fibers mainly used in cementitious matrices: natural, metallic, ceramic, glassy, polymeric (CAMPELLO; PEREIRA; DARWISH, 2014). They are classified according to the raw material from which they are produced, inorganic or organic types; and they can also be characterized by length, shape and thickness. Based on their size, they are either called macrofibers (diameter bigger than 0.3mm and length from 30 to 60mm) or microfibers (diameter between 10 and 30 μ m and length between 3 and 18mm) (SALVADOR, 2012). In general, microfibers are used to reduce shrinkage cracking while macrofibers are used for structural reinforcement (DING et al., 2008).

Regarding rigid or non-rigid fibers, a suitable criteria for categorizing then is to take into account the Young modulus of the fiber material and its shape (MARTINIE; ROSSI; ROUSSEL, 2010). Rigid fibers push apart particles that are relatively large compared to the fiber length thus increasing the granular skeleton porosity while flexible fibers fill in the space between aggregates (GRÜNEWALD, 2004).

The viscous and elastic properties of the fiber suspensions are shown to be strongly enhanced by fiber flexibility. By increasing fiber flexibility the suspension exhibits enhanced shear-thinning and decreased shear rate for the shear-thinning onset (KESHTKAR; HEUZEY; CARREAU, 2009).

PVA fibers are considered non-rigid fibers. Figure 2.8 shows that PVA fibers contain hydroxyl groups which have the potential to form hydrogen bonds between molecules resulting in remarkable changes in the surface bond strength between fibers and matrix (XU; TOUTANJI; GILBERT, 2010). Due to the presence of the hydroxyl (OH) group in PVA fibers the chemical bond is high (LI; WANG; WU, 2001; LI et al., 2002), which increases the fibers wettability in the polar matrix mix enhancing their dispersion when coupled with mechanical agitation during mixing (KONG; BIKE; LI, 2003a).

Figure 2.8 – PVA fiber structure (NOUSHINI; SAMALI; VESSALAS, 2013)

Surface image analysis via Scanning Electron Microscope (SEM) was used to compare three kinds of fibers: polyvinyl alcohol (PVA), polypropylene (PP) and steel fibers Figure 2.9. Steel fibers are rough providing a greater surface area which may improve the cement paste bonding, Figure 2.9a. The same steel fiber surface features were identified by (LOWKE et al., 2012). They also concluded that very fine particles accumulate on the fiber surface in the interfacial transition zone (ITZ) increasing the fiber surface roughness. Consequently promoting micro interlocking in the bonding zone (LOWKE et al., 2012). On the other hand, the surface of PVA fibers Figure 2.9b indicated the presence of many small split gaps, providing a larger surface area potentially improving the cement paste bonding too. The PP fiber surface was smooth with long striations through the fibers showing fracture planes in the fibers, resulting in a poor bond in the concrete matrix Figure 2.9c (KIM; BOYD; LEE, 2011).

Figure 2.9 – Fiber surface images obtained via SEM. a) steel fiber (1500x); b) PVA fiber (1000x); c) PP fiber (1000x), adapted (KIM; BOYD; LEE, 2011).

Furthermore, PVA fibers in a cementitious matrix tend to rupture instead of pulling out due to their strong chemical bonding to cement hydrates. For this reason fibers are usually coated with an oiling agent to reduce their surface hydrophilicity (LI; WANG; WU, 2001; LI et al., 2002; WANG; LI, 2007). In order to evaluate the PVA-ECC interface properties five levels of coating (from 0% to 1.2% by weight) were applied. The results showed that an increase in coating level leads to an increase in tensile strain capacity accompanied by a larger crack width and reduced crack spacing. Particularly, for this experimental study an oiling content of 1.2% by weight gave the best results (LI et al., 2002). Another research used oil coating and fly ash together. They found that in addition to 1.2% by weight oil coating some fly ash amount provides extra lubricant for fiber pullout (WANG; LI, 2007).

2.5.2 Fiber content and aspect ratio

Fiber content significantly affects mixing, placing and hardened performance. Likewise, fiber content affects the choice of production technology. A low amount of fibers is well suited for batch mix preparation using conventional mixing equipment and drop placement. Higher fiber contents often require special mixing tools or placement techniques (ZOLLO, 1997). Otherwise, high fiber content can be incorporated into the system if mixing technology is controlled via advanced formulations based on sophisticated rheological control (BENTUR; MINDESS, 2007).

Results show that fiber content has great influence in the concrete mobility since fibers act as a barrier by making aggregates movement coarse, reducing slump and increasing exponentially the Ve-Be time (FIGUEIREDO; CECCATO, 2015). However the concrete becomes more difficult to mix if the aggregate size or the fiber aspect ratio increases (GRÜNEWALD; WALRAVEN, 2001; MARTINIE; ROSSI; ROUSSEL, 2010; FIGUEIREDO, 2011; ILLGUTH; LOWKE; GEHLEN, 2013).

The rheological behavior of low carbon fiber content reinforced cement mortar conforms to the Bingham model, i.e. by increasing fiber volume and fiber length both yield stress and plastic viscosity increases (BANFILL et al., 2006). In addition, fiber volume should be optimized to achieve optimal rheological properties while maintaining high ductility (CHOI et al., 2015).

SHCC, for instance, are produced using high performance polyvinyl alcohol (PVA) fibers, usually up to 2% by volume (LI, 2003; MECHTCHERINE et al., 2011). Most research works use this kind of PVA fiber which is produced by the Kuraray company and has a Young's modulus of 40 GPa and tensile strength of 1.6 GPa (CUROSU et al., 2017).

Fiber content is an important factor since it directly affects the mechanical performance. Figure 2.10 shows the comparison between two stress-deformation curves recorded via uniaxial tension tests for strain-hardening polyethylene fiber-cement composites. A pseudo-strain-hardening behavior is seen for 2% fiber volume while for 0.4% fiber volume a behavior similar to that of a quasi-brittle material is observed (MAALEJ; HASHIDA; LI, 1995).

Figure 2.10 – Influence of fiber content on uniaxial tensile stress-deformation response (MAALEJ; HASHIDA; LI, 1995).

Furthermore, studies of the tensile properties of mortars reinforced with PVA fibers have found that the first peak stress increases per fiber volume fraction and decreases per fiber diameter for a constant fiber aspect ratio (BETTERMAN; OUYANG; SHAH, 1995).

The fiber aspect ratio is given by the fiber length divided by the equivalent fiber diameter for any individual fiber. This parameter is also a measure of fiber stiffness and will affect mixing and placing (ZOLLO, 1997). It was observed that given an increment of 10% in the aspect ratio the strength and tenacity increases respectively by 26% and 23% (PAKRAVAN et al., 2012). Additionally, was reported that PVA fibers with smaller diameters (14 μm diameter and 6 mm length) result in more severe fiber rupture. As consequence, the composite exhibits a minimal strain hardening behavior under uniaxial tensile loading compared to fibers with 39 μm in diameter and 12 mm in length for instance (LI; WANG; WU, 2001).

2.6 FIBER DISTRIBUTION

Fiber dispersion is another important factor to enhance the mechanical properties of SHCC, ductility mainly (TORIGOE et al., 2003; ZHOU et al., 2012), and reduce the variability. In order to obtain a good fiber distribution, Zhou et al. (2012) suggest controlling the plastic viscosity of ECC mortars before adding fibers (ZHOU et al., 2012). Another study proposed a plastic viscosity range of 5.0 to 14 Pa.s to ensure uniform fiber dispersion of high performance fiber-reinforced cement composite (HPFRCC) (CHOI et al., 2014, 2015). Also, due to the shear force triggered by the mixer they employed a high-speed mixer and a longer mixing time to achieve good fiber dispersion (CHOI et al., 2015). In addition, (TORIGOE et al., 2003) evaluated the relationship between the degree of distribution and the ultimate tensile strain of the composite using a fluorescence technique.

A uniform fiber dispersion is vital to the development of cementitious composites which effectively take advantage of the fibers reinforcement properties (AHMED; MIHASHI, 2011). For instance, some authors used cellulose powder to facilitate uniform PVA fiber dispersion because it acts as a colloidal stabilizer and controls the fresh mix rheology (AHMED; MIHASHI, 2011). The use of slag particles was also reported to improve fiber dispersion. An electrical double layer formed by the oxidized slag surface provides a driving force to the mortar matrix flow better dispersing the fibers, hence leading to a more ductile composite (KIM et al., 2007).

Furthermore, fiber dispersion uniformity influences the tensile strain-hardening behavior by increasing the tensile strain capacity of Engineered Cementitious Composites (ECC) responsible for fiber bridging strength, critical flaw size and degree of multiple crack saturation (LI; LI, 2013; TOSUN-FELEKOĞLU et al., 2014). Conversely, inadequate fiber dispersion causes free water retention within agglomerates and inter-particle bond formation, all of which act to restrict the suspension flow (VANCE; SANT; NEITHALATH, 2015). Additionally, fibers entanglement act as an obstacle to the flow resulting in segregation between fibers and the fresh matrix (KONG; BIKE; LI, 2003b) as seen in Figure 2.11. Consequently, insufficient dispersing of fibers in the cement matrix during mixing decreases the compressive strength of cement-based composites with fibers (YUN, 2013).

Figure 2.11 – Non-uniform fiber distribution. Photo by Mazanec (TUM: Centrum Baustoffe und Materialprüfung – CBM).

Fibers can entangle with themselves or with other particles affecting the system workability as seen in Figure 2.11. Therefore, some characteristics of the fresh material like mixing, rheological behavior and segregation need to be taken into consideration because they define the initial conditions for an appropriate microstructure.

2.7 MECHANICAL PERFORMANCE

The addition of fibers into mortars has the greatest influence on fracture toughness, bond strength and impact toughness (LEONHARDT et al., 2011; LEONHARDT; LOWKE; GEHLEN, 2012a, 2012b; LUO et al., 2013). Strength and toughness measurements are affected by the particular testing machinery and measurement devices employed as well as by the size and shape of testing specimens (ZOLLO, 1997). Additionally, the number of fibers in a unit volume of matrix material is directly related to the statistical probability of cracks encountering fibers. The fiber specific surface is related to the amount of energy that is absorbed in encounters between cracks and fibers. In this way weak brittle matrices can be improved gaining some measure of post-cracking toughness since fibers cross the paths of potential cracks transmitting stress between fibers and matrix through interfacial bonds (ZOLLO, 1997). Likewise, the fine fibers control the opening and

propagation of micro cracks as they are densely dispersed in the cement matrix. Longer fibers (up to 50 or 80 mm) control larger cracks contributing to increase the final strength of the reinforced material (BETTERMAN; OUYANG; SHAH, 1995; BRANDT, 2008). These findings are illustrated in Figure 2.12.

Figure 2.12 – Fiber size influence on crack bridging (BETTERMAN; OUYANG; SHAH, 1995).

Furthermore, in a quasi-brittle matrix composite like that of strain-hardening cementitious composites, flaws are typically the places where cracks appear. Also, most flaws that inherent from mixing processes have 4 mm in size, and their existence considerably reduces the intrinsic material cracking strength (LI; WANG, 2006).

2.7.1 Strain-hardening behavior

Strain hardening cement-based composites display high strain capacity when subjected to tensile loading. This quasi-ductile behavior results from progressive multiple cracking achieved by the optimized crack-bridging action of short, thin, well

distributed, polymeric fibers (MECHTCHERINE et al., 2012). During strain-hardening, deformation happens via the opening of multiple sub parallel fine cracks and the consequently elastic stretching of the material between these cracks (FISCHER; LI, 2007). PVA fibers are often used to promote high ductility and tight crack width control in cement based composites since they show a strain-hardening behavior under tensile load. This phenomena is analyzed by different authors showing that the largest possible tensile strain capacity is achieved by maximizing multiple cracking behavior (LEE et al., 2012; MECHTCHERINE et al., 2012; LI; LI, 2013; MAGALHÃES; TOLEDO FILHO; FAIRBAIRN, 2015; LIU et al., 2016).

Based on the concept of composite optimization by means of micromechanics-based strain-hardening model, composite tensile strain-hardening behavior is achieved by adopting a properly tailored minimum amount of fibers, matrix, and interface (YANG; LI, 2010).

Figure 2.13 shows that brittle matrices lose their tensile load-carrying capacity immediately after the formation of the first matrix crack. In fiber reinforced concrete (FRC) the addition of fibers can increase its toughness. However the strain capacity beyond the first cracking is not enhanced. High performance fiber reinforced cement composites (HPFRCC) are defined by an ultimate strength higher than their first cracking strength and the formation of multiple cracking (FISCHER; LI, 2007).

Figure 2.13 –Schematic stress-strain behavior of cementitious matrices in tension (FISCHER; LI, 2007).

Once a crack is formed in a uniaxial tensile specimen, the bridging fibers across the crack define the loading carrying capacity of the composite. A large crack spacing implies inefficient utilization of the reinforcing fibers (LI; WANG, 2006).

The fundamental requirement for multiple cracking is that steady-state flat crack extension prevails under tension, which requires the crack tip toughness J_{tip} to be less than the complementary energy J'_b calculated from the bridging stress σ versus the crack opening δ curve. In other words, the maximum energy available for steady state crack propagation should exceed the energy necessary to break down the matrix (KANDA; LI, 2006).

Figure 2.14 – Typical $\sigma(\delta)$ curve for tensile strain-hardening composites. Hatched area represents maximum complementary energy J'_b . Shaded area represents crack tip toughness J_{tip} (YANG; LI, 2010).

According to Equation (2.2), where $J_{tip} = K_m^2/E_m$, σ_0 is the maximum bridging stress corresponding to the opening δ_0 , K_m is the matrix fracture toughness and E_m is the matrix Young's modulus. Equation (2.2) relies on the concept of energy balance during flat crack extension between external work, crack tip energy absorption through matrix breakdown, and crack flank energy absorption through fiber/matrix interface debonding and sliding (YANG; LI, 2010).

Equation (2.2):

$$J_{tip} \leq \sigma_0 \delta_0 - \int_0^{\delta_0} \sigma(\delta) d\delta \equiv J'_b$$

Another condition for pseudo strain-hardening is that the matrix first cracking strength σ_{fc} must not exceed the maximum fiber bridging strength σ_0 .

Equation (2.3):

$$\sigma_{fc} < \sigma_0$$

Satisfaction of both Equations (2.2) and (2.3) is necessary to achieve ductile strain-hardening behavior (YANG; LI, 2010). Furthermore the pseudo strain-hardening (PSH) performance index is used to quantitatively evaluate the margin between J'_b and J_{tip} , as well σ_{fc} and σ_0 and is defined by:

Equation (2.4):

$$PSH \text{ energy} = \frac{J'_b}{J_{tip}}$$

Equation (2.5):

$$PSH \text{ strength} = \frac{\sigma_0}{\sigma_{fc}}$$

Materials with larger PSH indexes should have a better chance of saturated multiple cracking while unsaturated PSH behavior often leads to small tensile strain capacity and variability in tensile ductility (YANG; LI, 2010).

When describing the tensile stress-strain curve according to (ZHOU et al., 2012), the tensile stress increases at the beginning and then drops. The first stress drop corresponds to the first cracking in the specimen while the peak stress is called the first cracking strength which is related to the tensile strength of the matrix. Consequently, as the tensile stress slowly increases, the specimens exhibit strain-hardening and multiple-cracking behavior. The ultimate tensile strength is defined as the maximum stress in the tensile stress-strain curve corresponding to the tensile strain capacity (ZHOU et al., 2012). Therefore, from the tensile stress-strain curve are measured the first crack strength, ultimate tensile strength and ultimate tensile strain (KONG; BIKE; LI, 2003b).

The shape of the σ (δ) curve shown in Figure 2.14 is related to a different number of mechanisms for fiber-matrix interaction. When fibers and matrix are in

frictional contact, the stiffness of the bridges is governed by the fiber volume content, fiber diameter, fiber length and stiffness, and the interface frictional bond. In case a chemical bond is present the starting point of the σ (δ) curve is shifted upwards (LI et al., 2002).

It was found in the study of strain-hardening fiber reinforced alkali activated mortar using PVA fibers that the tensile strain capacity increases with a stress performance index increase (i.e. the ratio of peak bridging stress to the first cracking strength). It also increases with the energy performance index (i.e. the ratio of complementary energy to the matrix fracture toughness). Besides that, the tensile strain capacity ranged from 1.53% to 4.48% (LEE et al., 2012).

Three point bending tests are also carried out on notched specimens (40 x 40 x 160 mm in dimension) to obtain the load-crack mouth opening displacement response (AHMED; MIHASHI, 2011) although uniaxial tensile test is considered the most convincing method to evaluate material strain-hardening behavior (KIM et al., 2007; LI; LI, 2013). This happens when, a fiber reinforced concrete (FRC) for instance, shows apparent strain-hardening under flexural loading depending on its specimen geometry (KIM et al., 2007).

In summary, strain-hardening behavior is a very desirable property, generally in conjunction with multiple cracking and related large energy absorption capacity (NAAMAN; REINHARDT, 2003; SHI; MO, 2008).

3

Mixing sequences of PVA reinforced mortars

3.1 INTRODUCTION

This chapter discusses an initial approach about different mixing sequences of mortars reinforced by Polyvinyl Alcohol (PVA) fibers. The influence of fibers and water addition on mixing and rheological behavior in terms of torque and rotational speed are evaluated using the rotational rheometry.

The investigation of diverse mixing procedures for cementitious composites containing fibers seeks optimum dispersion of fibers within the matrix with reduced of the fibers damage (that may occur during processing), so that the fibers can perform their role as reinforcement as expected and, consequently, produce a composite that can achieve its designed properties.

In this context, five different mixing sequences are studied: S1, S2 and S3 are based on the addition of fibers at different stages of the mixing process; while in S4 and S5 not only the fibers are added at different stages, but also the water addition is performed in two steps (25% first and 75% later).

The torque measurement during the mixing process is correlated to the different mixture stages. In addition, the rheological behavior is measured immediately after the mixing process via shear cycle curves (torque vs. rotational speed). From these results is expected to be found a correlation between the final stage of mixture and its rheological behavior.

3.2 EXPERIMENTAL PROGRAM

3.2.1 Materials

The reference mortar – R – is composed of ordinary Portland cement, hydrated lime, calcium carbonate filler and crushed sand with maximum particle size of 2 mm. Two compositions are studied: the reference one – R and another one with sodium polycarboxylate admixture – D. The powder admixture, with proportion of 0.08% in relation to the cement powder mass, is added to the dry material. Mix design is demonstrated in Table 3.1 along with the physical characteristics (specific gravity and specific surface area) of raw materials. Water content in weight was 16% for all mixtures (i.e. water/solids ratio of 0.16).

Table 3.1 – Mix proportions and physical characteristics (specific gravity, BET specific surface area) of raw materials.

Raw material	BET surface area (m ² /g)	Specific gravity (g/cm ³)	Weight Proportion*
Filler	1.23	2.76	0.75
Cement	1.43	3.10	1.00
Hydrated Lime	6.15	2.49	0.25
Crushed sand	0.27	2.79	4.25

*With regard to the cement weight

Polyvinyl Alcohol (PVA) fibers with 6 mm in length, 16 μ m in thickness and specific gravity of 1.3 g/cm³ were used in these experimentals. A fiber content of 0.2 vol. % was added to the reference composition (0% of fibers). Fibers were dispersed manually and, then, with the aid of an air jet flow inside a plastic bag, before adding in the mortars.

3.2.2 Equipment

The rotational rheometer for mortars and concretes (Figure 3.1) developed at Poli-USP (Brazil) was used. The equipment performs speed-controlled tests while the electric power of the engine is measured and then, using a calibration constant, converted into torque values. The impeller used in the planetary setup for mixing and

rheological tests has six blades in spiral configuration, and the blades stand at a minimum distance of 1mm from the wall and bottom of the vessel. The device is capable to measure the mixing of mortars from dry powder to fluid state and to perform shear cycles to determine their rheological behavior.

Figure 3.1 – a) Rotational rheometer Poli-USP, (1) rotational device, (2) base reaction and console, (3) lift, (4) mortar vessel, (5) mortar impeller; b) Vessel tool details.

3.2.3 Mixing sequences

Five mortar mixing sequences were studied. Sequences S1, S2 and S3 were based on the addition of fibers at different stages of mixing process: S1 – fibers were added at the beginning of the process with the particulate materials; S2 – fibers were introduced at the fluidity point; S3 – fine powder and water were prepared apart in a high speed mixer and added into the mortar vessel where fibers were pre mixed with sand. While in S4 and S5 procedures not only the fibers were added at different stages, but also the water addition was performed in two steps (25% first and 75% later). Those previous sequences were based on preliminary studies (ANTUNES, 2005; CARDOSO, 2009; ZHOU et al., 2012; FRANÇA; CARDOSO; PILEGGI, 2013) which showed that the addition of water in two steps increased mixing energy and improved the dispersion of particles. All mixing sequences are described below and also in Figure 3.2.

Mixing sequence 1 – S1: Dry PVA fibers previously dispersed were incorporated into the 4 kg-batch of dry mixture of raw materials and the whole mixture was homogenized inside a plastic bag. Then this mixture was moved to the rheometer

vessel. The first 20 s of mixing in the rheometer consisted of a dry homogenization step, then all the water was added at a rate of 128 g/s. Mixing continues at 126.5 rpm until 317 s.

Sequence 2 – S2: This sequence is similar to S1, but the mortar is mixed without fibers, which are only added manually during 10 s at the fluidity point (100 s). This fluidity point was determined by analyzing the mixing curve of sequence S1 with the same methodology used in (FRANÇA, 2012).

Sequence 3 – S3: PVA fibers were initially mixed with sand within a plastic bag before being introduced into the mortar vessel. Powder content and water were prepared apart in a high speed (10000 rpm) mixer during 3 min. The paste was added into the system after 20 s of dry mixing (sand + PVA fibers). The paste addition takes 10 s and mixing continues until 317 s.

Sequence 4 – S4: Dry particulate materials were introduced into the mortar vessel. After 20 s of dry mixing, 25% of water was added at a rate of 128 g/s and mixed for 120 s. Then the remaining 75% of water was added at the same rate and mixing continues. After 120 s, the fibers were introduced manually into the system during 10 s and mixing continues until 317 s.

Sequence 5 – S5: Dry particulate materials were introduced into the vessel. After 20 s of dry mixing, 25% of water was added at a rate of 128 g/s and mixed for 120 s. Then fibers were introduced manually into the system during 10 s and mixing continues. At 200 s, the remaining 75% of water was added at the same flow rate and mixing continues until 317 s.

Figure 3.2 – Summary table of mixing sequences.

3.2.4 Assessment of mixing behavior

Batches of 4 kg of dry material were mixed in the rotational rheometer with the planetary setup (Figure 3.1). Mixing speed, total mixing time and flow rate of water addition were kept constant and equal to: 126.5 rpm, 317 s, and 128 g/s respectively. The variation of torque required to maintain the impeller at a constant rotation speed was monitored by the rheometer during the whole process, from dry material mixing to the end of each procedure (described in 3.2.3), thus resulting in the mixing curves (torque vs. time). The overall work during each mixing sequence was quantified by the area under the torque vs. time curve (integral).

3.2.5 Rheological evaluation

Immediately after mixing, the mortars were subjected to a rotational shear cycle with the same rheometer setup used for mixing (impeller geometry and planetary configuration). The shear cycle consists of accelerating and decelerating the mortar in 5-second steps at different rotations (6.3; 12.7; 25.3; 38; 63.3; 126.5; 190, 253; and 316 rpm) as illustrated in Figure 3.3. The rheological behavior was obtained from the shear cycle as well as the hysteresis area (the difference between acceleration and deceleration curves) which indicates the stability condition of the material, therefore, also indicating the mixing efficiency provided by the procedure applied (PILEGGI et al., 2001).

Figure 3.3 – a) Rotation test program of the shear cycle. b) Typical shear cycle result.

After the rheological test, fresh density and air content of the mortars were measured using gravimetric method as per the Brazilian standard ABNT NBR 13278 (2005) (ABNT NBR 13278, 2005).

3.3 RESULTS AND DISCUSSION

3.3.1 Air content

Table 3.2 shows the amount of air content entrained within the mortars after the shearing cycle. For the reference composition (0% fiber), no influence of the mixing sequence on the entrained air is observed. On the other hand, the fiber reinforced mortars presented slightly higher air content than the reference compositions, especially for sequences S4 and S5. Regarding the compositions with admixture – D,

fibers have more impact in the air content doubling up these values in comparison to R composition, for the same water/solids content. Since the admixture improved particle dispersion while increasing the free water in the system, this free water can be responsible for raising the air content.

Table 3.2 – Air content of the mortars prepared by different mixing sequences. Reference composition – R. Admixture in composition – D. Fiber content is given as a volume of the initial raw materials. The air content value refers to each mixture.

0% fiber	Air content (%)	0.2% fibers	Air content (%)
RS1_0%	5.1	RS1_0.2%	5.6
RS2_0%	5.1	RS2_0.2%	5.8
RS3_0%	3.8	RS3_0.2%	6.6
RS4_0%	4.5	RS4_0.2%	7.8
RS5_0%	5.2	RS5_0.2%	6.5
DS1_0%	10.1	DS1_0.2%	12.2
DS2_0%	10.1	DS2_0.2%	12.2
DS3_0%	13.0	DS3_0.2%	12.1
DS4_0%	12.1	DS4_0.2%	14.7
DS5_0%	12.1	DS5_0.2%	13.9

3.3.2 Mixing curves

3.3.2.1 S1, S2 and S3 mixing sequences

Mixing curves from mortars prepared by S1, S2, S3 sequences are shown in Figure 3.4 and Figure 3.5. After water was added, torque levels increased rapidly until peak, which corresponds to the point of maximum mixture cohesion. Then shear forces, due to intense mixing, started to overcome capillary and van der Waals forces broke the agglomerates distributing entrapped water throughout the system causing a gradual decrease of torque. Notice that when torque levels stabilize, it signals the point from which the material started to behave as a fluid, i.e. the fluidity point.

Figure 3.4 – Mixing curves S1, S2 and S3 for reference composition – R.

The torque levels of the mortars with PVA fibers mixed following S1 are considerably higher than the reference mortar prepared by the same sequence. Due to the presence of the fibers a strong agglomerate structure is formed since the non-linear shape of the flexible fibers create a greater interaction between the particles (KESHTKAR; HEUZEY; CARREAU, 2009), probably creating bigger agglomerates and mechanical bridging between agglomerates and between the fibers themselves as well. Moreover, van der Waals attractive forces added by the fibers collaborate for the formation of the agglomerates, making dispersion of the fine particles even more difficult and consequently increasing mixing efforts and reducing flowability (COLLINS; LAMBERT; DUAN, 2012).

The fibers were previously mixed with the dry mortar in S1 resulting in greater efforts when compared to S2, where the fibers were added only at the fluidity point. In S1, fibers participate in the formation of the agglomerates since the beginning of the process, having an adverse effect on dispersion and homogenization of particles and on their own dispersion as well. It is reasonable to expect that the mechanical damage of fibers is more intense in the mortar prepared by S1 than in the one prepared by S2 because mixing time and shear forces are considerably higher in the first than in the latter sequence. In S2, fibers were introduced when the material was already in a fluid condition, i.e. at a much lower torque when compared to S1. From this point on, fibers were mostly homogenized throughout the system and a moderate increase in torque

occurred immediately after their addition, but it tended to decrease slightly by the end of the procedure.

In S3, paste prepared separately shows a high torque level Figure 3.4, and thus increasing mixing efforts. This result is contrary to what is usually found in literature. It could be linked to routine processes during paste preparation not promoting sufficient particle dispersion. However for admixture in composition – D, Figure 3.5, S3 sequence shows low torque level indicating a good mixing homogenization even for system with fibers. This means that the admixture improves the cement particle dispersion reducing the mixing efforts.

Figure 3.5 – Mixing curves S1, S2 and S3 for admixture composition – D.

Figure 3.5 shows that for D - mortars, differences between the mixing curves are smaller. The admixture reduces the mixing efforts and torque values by half when compared to R – mortars and also decreases the mixing sensibility. S2 sequence also shows more adequate mixing process in D - mortars. It should be noted that superplasticizer improves workability and promote good fiber homogenization as showed by (PASSUELLO; MORICONI; SHAH, 2009).

The polycarboxylate admixtures adsorbed on cement particles provide steric hindrance resulting in highly negative electric charges which cause inter-particle repulsion while at the same time the lateral chains in the admixture may generate steric

repulsion. Once the interaction between particles are weak, they are dispersed and therefore slightly smaller forces are required for the system to flow (CRIADO et al., 2009).

3.3.2.2 S4 and S5 mixing sequences

In the preparation sequences S4 and S5, water was added in two steps (25% and 75%); therefore, the mixing curve profile was changed, as illustrated in Figure 3.6. After the initial 25% of water addition, torque rose abruptly (as for S1, S2 and S3) and reached a plateau (≈ 5 N.m) until the remaining 75% of water were introduced at 140 s for S4. The amount of water corresponding to 25% of the total volume was not enough for this composition to turn into a fluid-like material, therefore torque levels remained high until the remaining water was added, which caused an instantaneous drop of torque. For the reference mortar, torque levels continued to decrease until the end of the mixing time as dispersion and homogenization processes with all water content took place. In S4, fibers were added to the mortar only at 260 s and caused the torque to increase.

Figure 3.6 – Mixing curves R composition with and without fibers S4 and S5.

The test of adding only 25% of water in the beginning of the mixing process promoted the raw materials pre-homogenization with a low level of liquid saturation. Torque increased until the maximum cohesion state (or capillary forces) was reached

when water created capillary bridges surrounding the particles, thus forming small granules (Figure 3.7). During sequence S5, the fibers were introduced under this high-torque plateau situation therefore causing the cohesion to increase even more and pushing torque to higher values. As already mentioned, due to fibers-particle interaction, fibers are likely to create bridges between different granules thus promoting them to grow and intensify cohesion forces (through mechanical bridging and surface area increase). When the remaining 75% of water was added the liquid quickly reached saturation and most of the granules were destroyed. This increased the inter-particle separation distance and also the distance between fibers making the system much more disperse and, consequently, more fluid.

Figure 3.7 – Scheme of granules formation in the mixture with water and fibers addition according to the sequence S5.

In D mortars the superplasticizer acts after all water is added into the system reducing the torque level Figure 3.8. Before this no difference is noted in comparison to R composition. S4 and S5 sequences have shown less efficiency in terms of mixing efforts for D compositions. With respect to the final torque level a slight difference can be seen between R and D composition.

Figure 3.8 – Mixing curves of D composition prepared according to S4 and S5 sequences.

The overall work associated to each mixing sequence was quantified by the area under the torque vs. time curves (integral) and results are shown in Figure 3.9. Compared to the reference mortar without fibers, S4 and S5 sequences show much larger values than S1 and S2 since the mixing time with only 25% water content in S4 and S5 resulted in significantly high torque levels, thus increasing shear energy during the process.

Figure 3.9 – Equivalent work associated to each mixing sequence.

In case of mortars with 0.2% PVA fiber content, adding the fibers at the fluidity point as in S2 reduced the work by 25% when compared to S1 where the fibers were mixed with the particulate materials since the beginning of the process.

Adding the fibers at the fluidity point, as in S2, seems to be a more appropriate procedure because it demands less energy (which also means that equipment with lower power capacity is required). According to Figure 3.9, S2 resulted in the lowest mixing work when compared to the others sequences which were applied to the compositions with fibers. When comparing S2 to S5, their final torque results are similar, but the work associated to the former is considerably lower (42%) than the work of the latter. On the other hand S4 did not increase the work but the final torque was higher and not yet stabilized since fibers were added at the end of the mixing procedure and did not had time to disperse properly.

Results showed that the sequence of fiber and water addition does indeed have a significant influence on mixing behavior, which may contribute for fiber distribution and reducing the required mixing energy. Work of Zhou et al (ZHOU et al., 2012) demonstrated that the plastic viscosity of the mortar before fiber addition needs to be controlled in order to promote a good fiber distribution and that changes in the mixing sequence help to improve this process.

3.3.3 Rheological behavior

Figure 3.10 shows the results of the shear cycles for sequences S1, S2 and S3. The mortar without fibers RS1_0% displays a curve with much lower torque levels than the mortar containing fiber mixed as per sequence S1 (RS1_0.2%). Hysteresis area for RS1_0% is also lower than RS1_0.2%. For S2, in which the fibers were added at the fluidity point, torque levels are at an intermediate position among the reference composition, RS1_0.2% and RS3_0.2% all of which are in accordance to the final mixing torques showed in Figure 3.4.

Figure 3.10 – Results of mortar shear cycles of. a) RS1 and RS3 without fibers. b) RS1, RS2 and RS3 with 0.2% fibers. c) DS1 and DS3 without fibers, D composition. d) DS1, DS2 and DS3 with 0.2% fibers, D composition.

The D mortars shear cycles results Figure 3.10 c, d show lower torque levels than those of R composition. In this case a difference between 0% and 0.2% fiber content are minimal. Also, no difference between mixing sequences are seen in D mortars. The yield stress shows a significant decrease with presence of superplasticizer. These results are in agreement with previous works by (WALLEVIK; WALLEVIK, 2011; LOWKE, 2013; BEY et al., 2014; ASGHARI et al., 2016) which state that changing the amount of superplasticizer mainly reduces the yield stress.

For sequences RS4 and RS5 without fibers, Figure 3.11a, the shear cycle torque levels are lower than those of mortars with fibers. The mortars with fibers prepared by S4 and S5 displayed torque levels two times greater than the reference mortars prepared by the same procedures. They also yielded the largest hysteresis areas among all curves.

Figure 3.11 – Results of mortar shear cycles S4 and S5. a) R composition. b) D composition.

From the shear cycles of Figure 3.11a, it can be seen that the mortars without fibers presented lower yield torque than the ones containing fiber. While for D composition Figure 3.11b, the shear cycle behavior is very similar, independent of which parameter is evaluated. Among the mortars with fibers, S2_0.2% presented the lowest yield torque which indicates a lower initial stress demand to start flowing. On the other hand S4_0.2% displayed the highest yield torque.

Ultimately, for the experimental conditions employed in this study, the sequence S2 showed the best performance regarding the mixing process of fiber reinforced mortars, as well as, it yielded the mortar with the most flow able rheological behavior. Adding the fibers at the fluidity point not only reduced the overall energy required for mixing but it also decreased yield torque and the shear cycle torque levels.

In terms of composition type, D mortars promote less effort during mixing. Besides that, superplasticizer presence decreased the yield stress as well as the differences between the several mixing sequences. This behavior can be associated with the homogenization of fibers since the admixture improves fluidity and fibers have more possibilities to move without affecting the flowability of the system.

3.4 CHAPTER CONCLUSIONS

In this chapter five different addition sequences of water and PVA fibers were evaluated using two compositions: R, reference one and D, containing polycarboxylate admixture type. The mixing process had a great effect on the rheological behavior of fiber reinforced reference mortars (R), especially regarding the moment of fiber

addition during the procedure. When the fibers were added at the beginning of the procedure – dry composition – it demanded much higher mixing energy than when fibers were added after an initial mixing of the particulate materials with water. The two-step water addition procedures did not improve the rheological behavior of the mortars with fibers. On the contrary, it even increased the overall mixing work of these systems.

Polycarboxylate-based admixtures increased the fluidity in D mortars by decreasing the differences among the mixing procedures. However, the rheological behavior did not change in presence of this fiber content evaluated for D mortars.

The introduction of the fibers at the fluidity point – sequence S2 – significantly reduced the mixing work and improved the rheological behavior of the mortar when compared to the other mixing sequences. Therefore, this study showed that it is possible to optimize the mixing process of mortars containing PVA fibers by simply changing the sequence of water and fiber addition.

Finally, it is recommended to prepare the mortar before the addition of fibers in order to get a more robust system.

4

Rheological behavior of PVA-ECC mortars

4.1 INTRODUCTION

Rheological analysis of Engineered Cementitious Composites (ECC) materials is a difficult subject. Fibers completely change its flow properties as well as its consistency. Besides that, a uniform fiber distribution is a key factor to ensure ECC's tensile strain hardening behavior.

This chapter aims to develop suitable techniques to evaluate the rheological behavior of PVA-ECC mortars. For this purpose the rotational rheometer Viskomat NT was used to measure the rheological properties employing two different techniques: vane system and ball measuring system. The main parameters under investigation are mixing time with fibers and mortar consistency. The analysis compares the rheological response from those rheometer configurations regarding shear stress, viscosity, and thixotropy as a measure of static yield stress development with time at rest. Furthermore, limitations and advantages in using each test configuration to measure PVA-ECC mortars are discussed.

Based on these results, a method is proposed to measure the rheological properties of reinforced mortars.

4.2 EXPERIMENTAL PROGRAM

The experimental investigations were carried out on ECC mortars following a test sequence shown in Figure 4.1. Initially, mix proportions are chosen by varying superplasticizer (SP) content to obtain different fluidity levels (low, moderate and high fluidity). The same mixing process is applied to all tests. Mini slump flow is measured and the rheological tests begin 13 minutes after water addition in the mixture. The details of each step are discussed below.

Figure 4.1 – Overview of tests sequence.

4.2.1 Materials

4.2.1.1 Mixture composition

The mixture proportion consists of ordinary Portland cement, fly ash, fine silica sand (average size of 0.125 mm and maximum grain size of 0.250 mm) with density of 2.65 g/cm³, superplasticizer (SP) MasterGlenium 51 polycarboxylic ether (PCE) and polyvinyl alcohol (PVA) fibers as listed in Table 4.1. The mixture composition is based on results from (LI, 2008). Fiber content is given in percentage of volume.

Table 4.1 – Composition mix design by weight ratio, density and material consumption.

Mix	Weight proportion	Density (g/cm ³)	Consumption (kg/m ³)
Cement	1.00	3.10	573.61
Fly ash	1.20	2.29	688.33
Sand	0.80	2.65	458.89
Water	0.56	1.00	321.22
HRWR	0.017	1.09	9.75
PVA fiber (%Vol.)	2.00	1.30	26.00

4.2.1.2 *Fibers*

Polyvinyl alcohol (PVA) fibers are produced by Kuraray, Kuralon (RECS-15). The fiber dimensions are 8 mm in length and 40 μm in thickness with aspect ratio (l/d) of 200. The fiber volume content was 2% by volume. The fiber tensile strength is 1.6 GPa, Young's Modulus 41GPa and specific gravity is 1.3 g/cm³, Table 4.2.

Table 4.2 – Properties of RECS-15 PVA fiber.

Fiber type	Specific gravity (g/cm ³)	Diameter micron (μm)	Length (mm)	Tensile strength (GPa)	Elongation at break %	Young's Modulus (GPa)	Aspect ratio
PVA	1.3	40	8	1.6	6.5	41	200

4.2.1.3 *Superplasticizer*

To reach adequate mortar flowability high range water reducer (HRWR) a type of polycarboxylic ether admixture with density of 1.09 g/cm³, named MasterGlenium 51 is used. HRWRs have dispersing effects on the cement particles through steric and electrostatic repulsion. For each test, water and superplasticizer are mixed together and added to the raw material during mixing. All dosages of liquid admixtures are referred to the aqueous solutions and not to the solid fraction.

4.2.2 Method

ECC consistency was initially evaluated without fibers. Different admixture contents were studied to obtain their fluidity levels (low - LF, moderate - MF, high fluidity - HF) for a fixed water binder (cement + fly ash) ratio of 0.255. Then it was measured the spread in the mini slump flow until the admixture saturation point was reached, i.e. spread values did not increase any more. From these slump flow values three different fluidity levels were selected. They refer to the fluidity levels (low - LF, moderate - MF, high fluidity - HF). These results are shown in Appendix A. The relation superplasticizer binder (SP/binder) for each fluidity level is LF = 0.636%, MF = 0.773% and HF = 0.909% for low (LF), moderate (MF) and high fluidity (HF) respectively.

From the previous composite analysis, the fluidity levels now are evaluated with addition of fibers under different conditions as shown in Figure 4.2. After a fixed mixing time the composite fluidity is evaluated by mini slump flow test immediately after mixing process and its rheological properties measured in the rheometer. Following these tests, the fibers were added and the total mixing time with fibers increased, as shown in Figure 4.2.

Figure 4.2 – Test configuration.

4.2.2.1 *Mixing procedure*

Tests are conducted in an intensive laboratory pan Eirich mixer, with a capacity of 5 L. In this study, all tests are carried out with a vessel velocity of $v_1 = 45$ rpm and the impeller turned at velocity $v_2 = 200$ rpm. The mixer is set in counter current configuration; i. e. the vessel and device rotate in opposite direction. The batch volume is kept constant and equal to 2 L in all tests.

Batches are carried out according to Table 4.1 at a temperature of 20 ± 3 °C. The solid materials are loaded before starting the mixer. After mixing the solid constituents for 30 s to ensure materials dispersion and homogenization, water containing dissolved liquid admixture is progressively added during the next 10s. The solid material and liquids are mixed for additional 3 min. The total mixing time without

fibers is constant and equal to 5 min. Fibers are added during 20 s and the mixing times with fibers are either: 60 s, 120 s or 180 s.

4.2.2.2 Mini-slump flow

The flowability is assessed immediately after the mixing process using a mini-slump flow test. The mini-slump cone has a top diameter of 70 mm, a bottom diameter of 100 mm and height of 60 mm. The volume is 0.344 L. The cone is then lifted up to allow the mortar to flow without shocking. Once the flowing stopped, the spread of the mortar is recorded as the average of the diameters measured along two diagonals. This geometrical value is linked to the dynamic yield stress value of the material using the equation (4.1), (ROUSSEL; STEFANI; LEROY, 2005).

$$\tau_0 = 1.747\rho V^2 R^{-5} - \lambda \frac{R^2}{V} \quad (4.1)$$

The λ coefficient is a function of fluid surface tension and contact angle and it is assumed to be equal to 0.005. The tested volume V is 0.344 liters, ρ is the fluid density equal to 2250 kg/m³ and R is the spread radius.

The Murata approach is also used to describe slump as a function of yield stress τ_0 (equation 4.2) as well as von Misès yield criterion (equation 4.3). Both approaches are adequate for low spread (< 200 mm) according to Flatt et. al (FLATT; LAROSA; ROUSSEL, 2006).

$$\tau_0 = \frac{\rho V g}{2\pi R^2} \quad (4.2)$$

$$\tau_0 = \frac{\rho V g}{\sqrt{3}\pi R^2} \quad (4.3)$$

Where ρ and V are respectively the density and the volume of the sample, R is the spread radius and g is the gravity load.

4.2.3 Rheometry

Rheological flow curves are determined 13 minutes after water addition using a Schleibinger (Viskomat NT) rheometer. It consists of a cylindrical container with radius $r_c = 70$ mm and height $h_c = 85$ mm where a sample fluid of 1.0 liter approximately is introduced. The measurements are assessed by two different geometry devices: Ball measuring system and Vane system, Figure 4.3. Equations that govern the flow are different for each device and they are explained in detail in the following steps.

Figure 4.3 – Rheometer for mortars. a) Rotating ball configuration; b) Vane measuring cell. Both tests use a cylindrical container with the same volume of 1.3 L.

The comparison between both geometry devices is possible by using the same test program, Figure 4.4. This test program is the final result of a series of tests based on previous studies using the same equipment (LOWKE, 2013). The dynamic shear stress and viscosity are measured at descending rotational velocities 45 rpm to 5 rpm and 45 rpm to 0.05 rpm for Phase I and Phase II respectively. Every rotational velocity is hold for 12 s and slowed down to the next lower shear rate over 1 second. Subsequently, the thixotropy A_{thix} is measured in the same test, Phase III. Therefore, the static yield stress is determined at 15 s, 30 s, 60 s, 120 s, 240 s and 600 s after the ending of Phase II. Static yield stress is determined at a constant rotational velocity of 0.05 rpm as the maximum measured value in a period of 3 s. The mortar is

kept at rest between the static yield stress measurements. Thixotropy A_{thix} corresponds to the gradient of the static yield stress values versus time at rest.

Figure 4.4 – Rheometer test program.

4.2.3.1 Ball measuring system

The ball measuring system used in the present study consists in measuring the torque applied to a sphere with diameter $D = 20$ mm, fixed onto a thin holder. According to the literature (SCHATZMANN, 2005; LEEMANN; WINNEFELD, 2007), the governing equations of the ball measuring system are as follows. The sphere movement through a Newtonian fluid for Reynolds numbers $Re < 1$ can be expressed by the Stokes equation. It assumes that Stokes equation is also valid in the case of a non-Newtonian medium like a cement particle suspension:

$$F = 3. \pi. \eta. v. D \quad (4.4)$$

Where F is the force, η the dynamic viscosity of the fluid, v the velocity and D is the sphere diameter.

The Reynolds number is:

$$Re = \frac{v \cdot D \cdot \rho}{\eta} < 1 \quad (4.5)$$

Where Re is Reynolds number and ρ is the fluidity density. Torque and velocity are given as:

$$T = F \cdot L_h \quad (4.6)$$

$$v = \varpi \cdot L_h \quad (4.7)$$

Where T is the torque, L_h the distance between sphere and container center, and ϖ is the angular velocity.

From equations (4.4), (4.6) and (4.7) the torque T is given as:

$$T = 3 \cdot \pi \cdot \eta \cdot \varpi \cdot D \cdot L_h^2 \quad (4.8)$$

Dynamic viscosity is given as

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (4.9)$$

From equations (4.8) and (4.9) the linear relationship for shear rate (v/D) and shear stress can be derived.

The force of resistance F_{max} needed to move the ball through the mortar suspension is measured after different waiting periods during which the mixture is at rest. Each measurement is performed at the low rotational speed of 0.05 rpm. The resistance force F_{max} measured is due to the yield stress of the material. Neglecting the shear force due to the rotation motion, static yield stress τ_{max} is calculated using $\tau = Y_G F / (2\pi r^2)$. Through the correlation between the drag coefficient and Y_G (yield-stress parameter) using plastic boundary-layer analysis (BERIS et al., 1985) showed that when the drag coefficient tends to infinity the sphere ceased to move at a value $Y_G \approx 0.143$. The first derivative of the static yield stress as a function of time is a measure of the thixotropy (LOWKE et al., 2012).

4.2.3.2 Vane test

The Vane measuring cell (Schleibinger, Viskomat NT) is another configuration able to determine rheological parameters of cementitious materials, Figure 4.3b. The shear stress is measured at descending shear rates. The viscosity and yield stress

are analyzed by using the Bingham model (equation 4.10) based on the Reiner-Riwlin equation and the assumption that the gap is fully sheared (KOEHLER; FOWLER, 2004; ESTELLÉ et al., 2008).

$$\tau = \tau_0 + \mu_p \cdot \dot{\gamma} \quad (4.10)$$

For this equation, τ is the shear stress (Pa), τ_0 is the yield stress (Pa), μ_p is the plastic viscosity (Pa.s), and $\dot{\gamma}$ is the shear rate (s^{-1}). When the rheological measurement is performed with a coaxial cylinder rheometer, such as the Vane configuration, torque (T) and rotational velocity (N) are measured. Shear rate and shear stress data are derived from the torque and rotational velocity. When the torque is at equilibrium at each shear rate step, the rheological properties are calculated by the Reiner-Riwlin equation. If the torque is not at equilibrium at any step, this data point should be eliminated from the results (GHAFARI et al., 2016). The Reiner-Riwlin equation transforms the parameters G and H, from the equation 4.11, defining a linear relationship between T and N, into the Bingham parameters, equations 4.12 and 4.13.

$$T = G + H \cdot N \quad (4.11)$$

$$\tau_0 = \frac{G}{4\pi h} \cdot \left(\frac{1}{R_i^2} - \frac{1}{R_o^2} \right) \cdot \frac{1}{\ln(R_o/R_i)} \quad (4.12)$$

$$\mu_p = \frac{H}{8\pi^2 h} \cdot \left(\frac{1}{R_i^2} - \frac{1}{R_o^2} \right) \quad (4.13)$$

Where R_i is the inner cylinder, R_o is the outer cylinder, and h is the height of the cylinder. The shear stress and shear rate are calculated according to procedure described by (ESTELLÉ et al., 2008). Static yield stress is calculated using equation (4.14):

$$\tau_s = \frac{T}{2\pi r^2 h} \quad (4.14)$$

Where τ_s = static yield stress (Pa) and r = radial parameter and corresponds to the spread between inner radius R_i and outer radius R_o .

4.3 RESULTS AND DISCUSSION

4.3.1 Mini Slump Flow

Figure 4.5 show that the spread measured by average of slump flow values increases with admixture content. Consequently the higher the SP/binder relation the more fluid is the mortar. Regarding fibers addition, the relation between reinforced systems with 2% PVA fibers and reference mortar without fibers is around three times. Spread reduces around one third for PVA-ECC mortars.

Figure 4.5 – Increasing of spread with SP content measured by mini slump flow. Comparison for both systems with and without fibers.

The mixing time with fibers has no significant effect in terms of fluidity measured by slump flow especially for low fluidity mortars, Figure 4.6. However a slight decrease of spread values with increasing of mixing time can be seen.

Figure 4.6 – Effect of mixing time with fibers on slump flow values. B : Ball measuring system. V: Vane system. LF: low fluidity, MF: moderate fluidity, HF: high fluidity.

The yield stress values calculated according to Roussel model equation (4.1) decrease with the increase in fluidity and is proportionally inverse to the spread values, Figure 4.7. So higher flow mixtures led to reduced τ_0 response.

Figure 4.7 – Yield stress calculated from mini slump flow.

Nevertheless the slump-flow value is considered necessary but not a sufficient measurement to characterize the rheological properties as reported by

Mahmoodzadeh and Chidiac (MAHMOODZADEH; CHIDIAC, 2013). Since it is a single measurement used to evaluate a complex fluid such as concrete. On the other hand, many constitutive equations have been proposed in the literature in order to characterize the rheological behavior of cementitious materials. In this work the equations related to ball measuring system and vane test are studied.

4.3.2 Ball measuring system

The results in Figure 4.8 are obtained from the test program shown in Figure 4.4. This is only one example of curve. The remaining results can be found in Appendix A. The rheological parameters analyses are related to data from phase I, Figure 4.8. Phase II has a role for structure breakdown before starting the thixotropy measurements during phase III.

Figure 4.8 – Typical curve obtained from rheometer tests.

4.3.2.1 Torque vs. time profiles recorded using ball measuring system

Figure 4.9 shows the torque vs. time curves for reinforced mortars (2%) compared to the reference mortar (0% fiber). Firstly it is observed an increase of

torque levels for reinforced mortars compared to mortar control. Moderate fluidity (MF) and high fluidity (HF) mixtures have similar behaviors showing torque levels in the same range. With regard to low fluidity (LF) mortars there is an increase of torque values almost twice than the torque levels of MF and HF. Thus it is possible to identify how the admixture affects the mortar fluidity since all other parameters are kept constant.

Figure 4.9 – Comparison among the different fluidity levels for 2% PVA fibers and reference curves (0% fibers).

4.3.2.2 Flow curves

Flow curves (torque x rotational speed) are illustrated in Figure 4.10. As it can be seen, low fluidity LF shows high torque levels than MF and HF even for reference mortar (0% fiber). Additionally torque increases linearly with rotational speed and curves well fitting the Bingham model.

Figure 4.10 – Flow curves torque vs. rotational speed, LF, MF and HF. Comparison between different fluidity level for systems with and without fibers (dashed lines).

This same tendency is found comparing the fluidity levels of each mixing time (Figure 4.11). MF and HF curves are very close to each other in terms of torque level except for a mixing time of 180 s (Figure 4.11c), since it seems mixing time with fibers promotes a rising of MF torque compared to HF.

Figure 4.11 – Influence of superplasticizer content on flow curves torque vs. rotational speed for different mixing times a) 60 s, b) 120 s, c) 180 s measured using ball measuring system (B). The relation superplasticizer binder (SP/binder) for each fluidity level is LF = 0.636%, MF = 0.773% and HF = 0.909%.

A second analysis shows how mixing time influences each fluidity level (Figure 4.12). In LF curves it is observed an increase of torque with mixing time (Figure

4.12a). MF shows more stability between the curves as well as a slight torque increase for 180 s, Figure 4.12b. Although HF mortars show decreasing torque with mixing time (Figure 4.12c) once more differences exist but they are minimal.

Figure 4.12 – Influence of mixing time on flow curves torque vs. rotational speed a) LF, b) MF, c) HF measured using ball measuring system (B). The relation superplasticizer binder (SP/binder) for each fluidity level is LF = 0.636%, MF = 0.773% and HF = 0.909%.

Based on the flow curves analysis, the mixing time with fibers has no significant influence on the rheological behavior. However more investigation and further analysis are conducted in this chapter to confirm this assumption.

4.3.2.3 Rheological parameters analysis

4.3.2.3.1 Apparent yield stress and viscosity

As a first rheological analysis, the apparent viscosity and yield stress are calculated through linear regression of flow curves torque vs. rotational speed. The results show a decrease in the apparent yield stress and viscosity with SP/binder increase, Figure 4.13.

Figure 4.13 – Apparent yield stress and viscosity. a), b) Reference mortar 0% fiber. b), d) Reinforced mortar with 2% PVA fibers. B: Ball measuring system.

From a physical point of view the yield stress is linked to the filling ability. Consequently a LF composition should be more difficult to cast than (MF and HF). It is assumed that the stress created by gravity force during casting the concrete

decreases until it reaches the yield stress of the material, at which point the flow stops (ROUSSEL; CUSSIGH, 2008).

4.3.2.3.2 Shear stress and viscosity

The shear stress, viscosity and shear rate are calculated according to equations from section 4.2.3.1 by ball measuring system. Figure 4.14 shows how ECC mortar fluidity and mixing time affect shear stress and viscosity. As discussed earlier, LF mortars have a behavior that differs from MF and HF compositions. ECC mortars prepared with low superplasticizer content show a big increase in shear stress and viscosity. It means that more work is necessary to cast this material for instance. Besides that, the shear stress and viscosity increase with mixing time. Consequently, increased mixing time does not contribute to either better rheological properties or fiber homogenization.

Figure 4.14 – Effect of mortar fluidity and mixing time with fibers on the shear stress and viscosity. a); d) 60 s, b); e) 120 s, c); f) 180 s mixing time.

In addition, Phan et al. (PHAN; CHAOUCHE; MORANVILLE, 2006) state that by increasing the shear rate, the paste first shows a shear thinning behavior, then changes to a Newtonian behavior and then finally displays a shear thickening behavior. Indeed similar behavior can be seen from our results in Figure 4.14 independently of SP content. Thus the rheological behavior changes according to shear rate range. This information is very useful under application conditions where the shear rate can be adjusted to optimize the final result.

Furthermore, three fluidity zones are identified regarding SP content and shear rate measurement, Figure 4.15. Low SP content shows high viscosity with low shear rate. On the other hand, high SP contents show low viscosity in high fluidity zone. For all SP content observes that mortar flows in the high fluidity zone concerning in the shear rate range of 3.0 s^{-1} to 7.1 s^{-1} . This shear rate range is below the maximum shear rate estimated of 10 s^{-1} for casting as reported by (ROUSSEL, 2006). In practice, different ECC mortars applications must take into account the shear rate.

Figure 4.15 – Fluidity zone for ECC mortars.

Regarding material applications, a relative low viscosity is required at high shear rates in order to properly process or apply the product. Once applied, the material should either stop flowing or flow slowly under any low stress that the product may still be subjected to, as mentioned by Mewis and Wagner (MEWIS; WAGNER, 2009).

Also with increased shear rate small agglomerates can be broken down. According to (PHAN; CHAOUCHE; MORANVILLE, 2006) the higher the shear rate the smaller are the flocks that can be broken down by the viscous forces and viscosity then decreases, as seen in Figure 4.15.

4.3.2.4 Static yield stress and Thixotropy

Figure 4.16 shows the static yield stress evolution in time at different fluidity levels revealing the influence of mixing time over static yield stress. There is a clear tendency of rising up yield stress as mixing time with fibers increases for all evaluated fluidity levels. Yield stress also increases when SP content reduced.

Figure 4.16 – Static yield stress. a) Low fluidity – LF, b) Moderate fluidity – MF, c) High fluidity – HF.

Although the results show no direct correlation between yield stress and bleeding as reported by (PERROT et al., 2012), a minimum yield stress value must

be taken into account. This minimum yield stress value refers to a condition under which bleeding does not occur or can be neglected. Regarding the fluidity level range evaluated in this study it seems that yield stress below 3 Pa increases the risk of bleeding for ECC mortars. Moreover, during the mini slump flow test paste bleeding in HF mortars is observed. This analysis also reinforces the idea of a minimum yield stress value necessary to avoid cement paste bleeding.

Looking at the initial yield stress values one observes its reduction with SP content Figure 4.17. This behavior is the same for all mixing times with fibers: 60 s, 120 s and 180 s. The initial yield stress value for 120 s of mixing time is twice the value correspondent to 60 s considering a low SP/binder.

Figure 4.17 – Correlation between SP/binder, yield stress and thixotropy for different mixing time with fibers.

Thixotropy is related to the stability of a composite. It means that stability can be improved when the cement paste is highly thixotropic. From the results showed in Figure 4.17, thixotropy decreases with SP for a mixing time with fibers equals to 60 s. For mixing times equal to 120 s and 180 s this tendency is not observed.

Figure 4.18 shows more clearly those differences. For LF mortars there is an A_{thix} decrease with mixing time while MF mortars follow the opposite direction. Moreover a distinct behavior is observed in HF mortars which show very low A_{thix} value independent of mixing time. Seemingly, this low A_{thix} is associated to the mortar bleeding behavior.

Figure 4.18 – Thixotropy analysis evolution with SP/binder and mixing time with fibers.

Furthermore, Figure 4.18 also shows how the superplasticizer content affects ECC mortar stability. Three different behaviors are identified according to the fluidity level. Lower thixotropic behavior is seen in high SP content (HF mortars) suggesting high mortar segregation risk while MF mortars show more stable behavior. For MF mortars the higher the mixing time the more thixotropic it becomes. On the other hand, LF mortars show high flocculation rate A_{thix} value for a reference mixing time of 60 s.

Additionally, although MF and HF have similar values for apparent yield stress and apparent viscosity, their thixotropic behaviors are very different leading to structuration rates from 0.001 Pa/s to 0.007 Pa/s (i.e. from 0.06 Pa/min to 0.42 Pa/min). In practice the more stable an ECC is the more useful it is for application conditions.

4.3.3 Vane test

4.3.3.1 Torque vs. time profile recorded using vane device

Mortar fluidity governs the ECC rheological properties as an intrinsic material characteristic, independently of the rheological measurement system. As can be seen in Figure 4.19, torque levels double when compared to a reference mortar without fibers.

Figure 4.19 – Effect of fluidity level and fiber content on torque vs. time curve.

Figure 4.19 shows high torque levels fluctuation during the rheological measurement with vane test configuration. These torque fluctuations caused by PVA fibers perturb the test results using Bingham model equations, since in order to reach a steady state a constant torque average is required at each rotational speed. Besides that it is possible to measure the rheological parameters using a specific shear rate range for each condition.

4.3.3.2 Flow curves

Flow curves measured by a vane test device are seen in Figure 4.20. They show that torque increases with rotational speed except for LF-2% which has small variation of torque values with increasing rotational speed. LF-2% has low SP/binder

content. Compared to others it is a more stiff material. During the vane test measurement it has been noted that mortar accumulates above the vane device. Not only this, but also at the beginning of the test the device cuts the mortar and sometimes looks like it creates a gap between the vane blades and mortar. This is noted at the end of the test as sometimes the device blades do not touch the mortar. Consequently, the low variation of torque values (0.10 to 0.12 Nm) can be attributed to less contact surfaces capable of capturing the totality of stress across the surface circumscribed by the vane device. This analysis promotes an understanding of the rheological behavior response as well as the difficulties found during the measurement of reinforced mortar using vane configuration.

Figure 4.20 – Flow curves measured by vane configuration. Comparison between PVA-ECC mortars and reference mortars without fibers.

By comparing the fluidity levels LF, MF and HF, torque decreases with increase of SP content, Figure 4.21. The same tendency is observed for all tested mixing times (60, 120 and 180 s). Additionally, SP content influence is higher than mixing time as noted in Figure 4.22. Low fluidity (LF) has a more cohesive matrix where both the fiber-fiber and fiber-matrix contacts are more intense. For high fluidity mortars (HF) the fiber-fiber contact remains but the fiber-matrix contact is impaired. The mortar fluidity level is so high that it has not enough viscosity to keep the fibers disperse through the suspension. Consequently fibers entangle with each other and

segregation occurs. Regarding moderate fluidity (MF) it is an intermediary behavior between LF and HF. MF shows enough fluidity to flow like a self-compacting matrix and at the same time fibers get well dispersed in the mortar.

Figure 4.21 – Influence of superplasticizer content on flow curves torque vs. rotational speed for different mixing times a) 60 s, b) 120 s, c) 180 s measured using vane configuration (V). The relation superplasticizer binder (SP/binder) for each fluidity level is LF = 0.636%, MF = 0.773% and HF = 0.909%.

This tendency of increasing torque values with decreasing SP-content is also observed for the different mixing times evaluated, Figure 4.21. Additionally the SP content influence is higher than the mixing time influence as it can be noted in Figure 4.22.

According to the mixing procedure used, mortar is first prepared without fibers to reach a good matrix homogenization. Fibers are then added during a period of 20 s. In sequence fibers are dispersed in mortar and three mixing times with fibers are analyzed: 60 s, 120 s and 180 s. This set up is the same of Figure 4.2. As can be seen in Figure 4.22 the effect of mixing time with fibers on the flow curves is minimal. For this reason 60 s is considered a sufficient enough mixing time to promote a good fiber homogenization in this set up (mixer, velocity, time, matrix composition).

Figure 4.22 – Effect of mixing time with fibers. a) Low fluidity – LF, b) moderate fluidity – MF, c) high fluidity – HF. Influence of mixing time on flow curves torque vs. rotational speed a) LF, b) MF, c) HF measured using vane configuration (V). The relation superplasticizer binder (SP/binder) for each fluidity level is LF = 0.636%, MF = 0.773% and HF = 0.909%.

4.3.3.3 Rheological parameters analysis

4.3.3.3.1 Apparent yield stress and viscosity

The apparent yield stress and the viscosity of the mortars are calculated directly from torque vs. rotational speed flow curves. As it can be seen in Figure 4.23, there is a decrease in both yield stress and viscosity for increasing SP/binder contents for reference mortars Figure 4.23 a,b and ECC mortars Figure 4.23 c. However the viscosity of ECC mortars increases with SP/binder content, Figure 4.23d.

Figure 4.23 – Apparent yield stress and viscosity measured from flow curves torque vs. rotational speed. a) Yield stress of reference mortar without fibers (0%), b) viscosity of reference mortar without fibers (0%), c) Yield stress of composite 2% of fibers, c) Viscosity of composite 2% of fibers.

The rheological measurement of PVA fiber reinforced mortars using vane test configuration presents some difficulties. As mentioned before, at low fluidity (LF) the device creates gaps between the blades. When fluidity increases, there are no more gaps between the blades which may explain the behavior in Figure 4.23d (i.e. viscosity increases with superplasticizer content).

4.3.3.3.2 Reiner-Riwlin equation

Reiner-Riwlin equation is the method to calculate the rheological parameters using concentric cylinders. Figure 4.24 and Figure 4.25 show the dynamic yield stress and viscosity calculated trough Reiner- Riwlín equation.

Figure 4.24 – Dynamic yield stress (a) and viscosity (b) calculated using Reiner-Riwlin equation for reference mortar without fibers.

For the reference mortar, both yield stress and viscosity decreases with increase of SP/binder content Figure 4.24.

PVA-ECC mortars exhibit a decrease in yield stress with increase of SP content as plotted in Figure 4.25a. By definition yield stress is the minimum stress for irreversible deformation and flow to occur (ROUSSEL; COUSSOT, 2005).

Consequently it is expected that $LF > MF > HF$ in terms of yield stress since yield stress decreases with fluidity level.

Figure 4.25 – Dynamic yield stress (a) and viscosity (b) calculated using Reiner-Riwlin equation.

Regarding the viscosity results in Figure 4.25b, the LF mortar viscosity values are lower than MF and HF due to the effects of sheared gaps during the measurements. These results are contrary to the literature where the superplasticizer addition usually decreases the viscosity of cement pastes (NG; JUSTNES, 2015). So it means that variations in viscosities of ECC mortars are not directly proportional to the added SP dosages. On the other hand the vane configuration used can be not suitable to measure PVA-ECC mortars at least for low fluidity levels.

4.3.3.3.3 *Shear stress vs. shear rate*

Shear stress and shear rate are calculated as a Bingham model using the Reiner-Riwilin equation and also the Estellé approach (ESTELLÉ et al., 2008). The reference mortar without fibers (0% fiber) Figure 4.26a shows two distinct behaviors: one for MF and HF and another one for LF. MF and HF have similar features such as low shear stress and almost the same shear rate range. Additionally, for those fluidity levels, the increase from shear stress to shear rate is smooth. In other words the fluidity levels show low viscosity. In the meantime, since LF has less admixture than MF and HF, LF shows a strong shear stress increase with shear rate. Consequently LF viscosity is higher than MF and HF viscosity.

It seems that for reinforced mortars (2% fiber) (Figure 4.26b) the behavior changes completely. High viscosity is achieved for MF-2% and HF-2% instead of LF-2%. This behavior can also be explained by the relatively higher SP adsorption which may cause a disorder state rising energy dissipation and increasing viscosity (YAHIA, 2014). In addition the shear stress values for reinforced mortars (2% fiber) are above the maximum measured in the reference mortar (0% fiber).

Figure 4.26 – Shear stress vs. shear rate. a) Reference mortar (0% fiber), b) PVA-ECC mortar (2% fiber).

PVA-ECC mortars show a rheological behavior that is close to shear thinning. However MF and HF have similar behavior with an increase in shear stress and shear rate exhibiting higher viscosity than LF which shows slight variation in shear stress for a high range of shear rate.

With regard to the mixing time with fibers Figure 4.27, a slight viscosity reduction with increase in mixing time for MF and HF mortars is observed. For LF mortars although they keep the same shear stress range there is a huge variation of shear rate mainly after 180 s of mixture with fibers.

Figure 4.27 – Shear stress vs. shear rate for different mixing times with fibers. (a) 60 s, (b) 120 s and (c) 180s.

4.3.3.4 Static yield stress and thixotropy

The evolution of static yield stress is nearly linear with the resting time (Figure 4.28). As it can be seen, there is an increase of static yield stress with the resting time mainly for MF mortars (Figure 4.28b). This analysis also brings a clear

distinction in terms of mixing time with fibers. Static yield stress increases as mixing time rises up. This behavior is clearly noted for LF and MF Figure 4.28a, and Figure 4.28b respectively. However this tendency is not seen in HF mortars. Additionally a slight static yield stress reduction with resting time is noted but is too small to be significant (Figure 4.28c). This difference can be explained considering the formation of dense and firm precipitate at the bottom of the rheometer, suggesting a mortar segregation behavior. The yield stress result from the competition between Brownian motion and colloidal interactions whereas the stability against segregation results from the competition between colloidal interactions and gravity (PERROT et al., 2012). Although the correlation between yield stress and segregation is not as direct as reported by Perrot et al. (PERROT et al., 2012).

Figure 4.28 – Static yield stress as a function of the resting time. Analysis of mixing time with fibers and mortar fluidity a) LF, b) MF and c) HF. The results show average values of a test series.

Regarding the static yield stress values, there is a reduction of yield stress with SP/binder increase, Figure 4.29. In terms of thixotropy, MF shows higher thixotropy values than LF and HF. By comparison, between LF and MF there is an increase of SP content equal to 0.137% which leads to a reduction from 65 to 21 Pa of the initial

yield stress $\tau_{\max 15s}$ after 15 s and a thixotropy increase A_{thix} from 0.07 Pa/s to 0.09 Pa/s.

Figure 4.29 – Evolution of yield stress and thixotropy with SP/binder content.

4.3.3.5 Relation between yield stress and spread

The yield stress measured using different approaches such as Roussel, von Misès and Murata was compared to the yield stress measured by Viskomat rheometer with Vane configuration, Figure 4.30. Roussel's model refers to the spread flow for very large spreads (> 200 mm) (ROUSSEL; STEFANI; LEROY, 2005), while von Misès and Murata approaches are more suitable for small spreads (< 200 mm) according to (FLATT; LAROSA; ROUSSEL, 2006).

Figure 4.30 – Relation between yield stress and spread diameter calculated using Roussel's model, Von Mises, Murata's approach and through vane configuration in the Viskomat rheometer.

Figure 4.30 shows that von Misès and Murata approaches have similar curves with calculated values. Yield stress calculated by Vane configuration fits quite well these models especially for MF mortars. On the other hand, high fluidity mortars HF are more similar to the Roussel model as expected since this model is suitable for high spread flow values. Notwithstanding, since those models are validated for cement paste, it can be assumed that they also apply for PVA- ECC mortars.

4.3.4 Comparison Ball versus Vane measurements

For mini-slump flow measurements, there is a good correlation between the batches prepared for both ball and vane test, Figure 4.31. As expected the spread values increase with increased SP content and mortar fluidity. These results confirm that changes between devices are only due to device configuration, since the composition fluidity is constant for both measurements.

Figure 4.31 – Mini slump flow measured before vane and ball tests in the rheometer.

In terms of rheometer measurements, the first comparison analysis among ball and vane test configuration is regarding torque levels (Figure 4.32). It can be seen that there are some differences obtained using these two rheometer devices. A linear relation among torque and rotational speed for ball measuring system is observed, which can be more easily characterized by Bingham model. On the other hand vane test shows more instability of torque values with increasing in rotational speed.

In relation to fluidity levels the same tendency is seen in both measurement devices, i.e. a decreasing of torque levels with increase in SP content.

Figure 4.32 – Comparison between vane (solid line) and ball (dotted line) configuration. a) 60 s, b) 120 s, c) 180 s mixing time with fibers.

The values of apparent yield stress and viscosity measured by sphere and vane device are compared in Figure 4.33. Yield stress measurement shows a good correlation between both devices for HF and as well as MF. In spite of dispersion increases with SP content reduction. With regard to apparent viscosity no correlation is found.

Figure 4.33 – Apparent yield stress (a) and viscosity (b) calculated from torque vs. rotational speed curves.

With regard to shear stress a good relation is observed between the values calculated from ball geometry and vane tests. Variations are more evident for LF mortars since they show higher shear stresses values Figure 4.34.

Figure 4.34 – Comparison between ball and vane in terms of shear stress.

On the other hand static yield stress measurements show considerable differences between values calculated by vane and ball devices. Ball measurement system response is very low compared to vane's, Figure 4.35. From geometrical point of view they are completely different as well as the equation used to calculate the rheological parameters.

Figure 4.35 – Static yield stress measured by vane and ball geometry in rheometer.

For comparison, the main characteristics of vane and ball measurement tests are listed in Table 4.3. Please notice that this is a qualitative comparison between vane and ball devices.

Table 4.3 – Main differences between vane and ball rheometer devices.

Analysis	Vane	Ball
Torque vs. rotational speed curves	Intense torque values fluctuations	Stable curves
Apparent yield stress and viscosity	Decreasing with SP content for REF mortars. For PVA-ECC viscosity increases with SP.	Decreasing with SP content
Yield stress and viscosity from Reiner-Riwlin equation	Decreases with SP content. For PVA-ECC viscosity increases with SP.	Non- measurable
Shear stress x shear rate	Shear rate changes according to material	Shear rate constant

Viscosity x shear rate	Non- measurable	Viscosity decreasing with shear rate
Static yield stress	- Decreases with SP content - Higher than measured by Ball	Decreases with SP content
Thixotropy	MF higher than HF and LF	- Changing according to mixing time with fibers - MF higher than HF and LF for 120 and 180 s of mixing time with fibers.

4.4 CHAPTER CONCLUSIONS

This chapter investigated the influence of two different rheometer devices (vane test and ball measuring system) to evaluate the rheological behavior of PVA-ECC mortars. The rheological parameters from each analysis were compared to each other.

With respect to material composition as well as the fluidity levels evaluated, it was found that MF and HF have similar behaviors, since they show torque levels in the same range. With regard to low fluidity mortars (LF), torque values are almost two times higher than those of MF and HF mortars. Low fluidity (LF) mortars have a more cohesive matrix and the fiber–fiber and fiber–matrix contact are more intense. For high fluidity mortars (HF), the fiber–fiber contact remains but the fiber–matrix contact is impaired. The mortar fluidity level is so high that it has not enough viscosity to keep the fibers disperse through the suspension. Consequently fibers entangle themselves and segregation occurs. Moderate fluidity mortars (MF) have intermediary behavior compared to LF and HF. MF shows enough fluidity to flow like a self-compacting matrix and at the same time fibers are well dispersed within the mortar.

ECC mortars prepared with low superplasticizer content show a great increase in shear stress and viscosity. It means, for instance, that more effort is required to cast this material. Besides that, shear stress and viscosity increase with mixing time.

Consequently, an increase in mixing time is not linked to better rheological properties or fiber homogenization improvement. However, static yield stress evolution with time analysis reveals that mixing time with fibers has great influence in terms of static yield stress. For all fluidity levels evaluated there is a clear tendency of yield stress rising up as mixing time with fibers increases. Yield stress also increases with SP content reduction.

The devices analyses show that torque increases linearly with rotational speed and curves, well fitting the Bingham model for ball measuring systems. While on the contrary, vane tests have high fluctuation of torque levels during rheological measurement. These torque fluctuations caused by PVA fibers perturb the test results using Bingham model equations since a constant torque average at each rotational speed to reach steady state is necessary.

Regarding the fluidity level range evaluated with ball measuring system, it is shown that yield stress below 3 Pa increases the risk of bleeding for ECC mortars. While for vane measurements the risk of bleeding occurs when static yield stress is less than 20 Pa. Those differences of yield stress values are due to the effect of measurement device. Moreover, for both device analyses paste bleeding in HF mortars is observed during the mini-slump flow test confirming that a minimum yield stress value is necessary to avoid cement paste bleeding.

Furthermore, three different behaviors are identified according to the fluidity level. Lower thixotropic behavior is shown in high SP content (HF mortars) suggesting high segregation risk. While, LF mortar shows high flocculation rate A_{thix} value for 60 s decreasing the thixotropy value with time meaning an unstable ECC mortar. With respect to the MF mortars, a more stable behavior among fluidity levels evaluated is shown.

Finally it can be concluded that both devices are able to measure the rheological behavior of PVA-ECC mortars. However, ball-measuring system shows more efficiency to evaluate systems reinforced by fibers since it measures torque values more precisely.

5

Effect of mixing procedure on fibers homogeneity

5.1 INTRODUCTION

This chapter investigates the mixing process effect regarding its effect on fiber homogeneity when producing ECC mortars. Two fluidity levels (LF and MF) with different superplasticizer content are evaluated. The Polyvinyl alcohol (PVA) fiber volume fraction is 2.0%, which is the amount, typically used for strain hardening cement-based composite (SHCC). The mixture composition is the same used in chapter 4. Parameters such as fiber addition at different points during the mixing process, mixing time and intensity are evaluated as well as their effect on fiber homogenization. A special grid formwork is used to evaluate the fiber homogenization in the fresh state. Additionally, rheological measurements are obtained in the rheometer after the mixture process.

By comparing the results obtained from rheological behavior, mixing power consumption and fiber homogenization after the mixing process, this chapter aims to answer the following questions:

- Does mixing intensity influence fiber homogenization?
- Is better fiber homogeneity achieved by changing the fibers addition procedure?
- Is there a correlation among mixing power consumption, fiber homogenization and rheological behavior?

5.2 EXPERIMENTAL

The material compositions used here are the same as shown in Chapter 4. Polyvinyl alcohol (PVA) fibers volume is 2% and the high range water reducer (HRWR) admixture content (SP) is 0.014 and 0.017 by weight for low fluidity (LF) and moderate fluidity (MF). Table 5.1 depicts the setup of the experimental tests. Two different stirrer mixing intensities are studied: high speed 4.9 m/s and low speed 2.5 m/s. The container speed is fixed equal 1 m/s. The mixer is set in counter current configuration, i.e. the stirrer and container rotate in opposite directions. The mixing method REF refers to reference mortar without fibers. Fibers are added at the beginning before mixing process (ECC-Bef) or after 180 s of mortar mixing (ECC-Aft). This mixing time (180 s) is kept constant for both mixing speeds (4.9 m/s and 2.5 m/s). In addition two hybrid mixing procedure low/fast (ECC-Aft-2.5-4.9 m/s) and fast/low (ECC-Aft-4.9-2.5 m/s) are investigated.

Table 5.1 – Experimental tests setup.

Mixing method	Mixing stirrer speed (m/s)	Fibers	(SP content)	
REF	4.9	0%	LF (0.014)	MF (0.017)
	2.5			
ECC-Bef	4.9	2%		
	2.5			
ECC-Aft	4.9			
	2.5			
ECC-Aft-2.5- 4.9m/s	2.5, 4.9			
ECC-Aft-4.9-2.5m/s	4.9, 2.5			

ECC – 2% fiber by volume;

Bef – fibers addition before mixing process;

Aft – fibers addition after mortar mixing;

An intensive inclined drum mixer with 75 L capacity and controllable stirrer speed (that can be varied continuously from 1 to 20 m/s) was available to prepare ECC mortars for those experiments, Figure 5.1. The batch volume is kept constant and equal to 25 liters. By using the Eirich Mixer, Figure 5.1, was possible to record the power consumption by the mixing stirrer during the mixing process.

Figure 5.1 – Intensive Eirich mixer with 75 L volume capacity. Photo: Mazanec (2010).

The mixing procedures are shown in Figure 5.2. Fibers are added at the beginning (ECC-Bef) together with dry material before the mixing process starts or after 180 s of mixing (ECC-Aft). In this case (ECC-Aft) the fibers are added during 20 s of continuous mixing. For all mixing procedures shown in Figure 5.2, dry material is mixed during 30 s. After that, water, previously mixed with superplasticizer, is added during 15 s. The mixing process continues until 330 s (total mixing time for all configurations).

Figure 5.2 – Mixing sequences evaluated in this study (W + SP = water + superplasticizer).

After the mixing process was finished, the rheological behavior was evaluated via a Viskomat NT rheometer using ball measuring system configuration. The rheological tests began 13 min after mixing. The same test program and methodology for results analysis of used in chapter 4 was applied here as well. In the meantime, mini slump-flow spread, density and air content by pressure method and fiber homogenization tests were performed. Figure 5.3 shows the sequence of tests used for this experimental work.

Figure 5.3 – Experimental test setup.

5.2.1 Stabilization time calculation

As reported elsewhere (CHOPIN; DE LARRARD; CAZACLIU, 2004; CHOPIN et al., 2007; CAZACLIU; ROQUET, 2009; MAZANEC; SCHIEßL; GEHLEN, 2011), the stabilization time is defined as the time necessary for the applied power to reach a determined limiting value. In this sense optimal fresh concrete properties are obtained as soon as the applied power decreases below a certain rate. Stabilization time T_s , also named as fluidity point, is calculated from the power curves obtained by the Erich mixer during mixing following the mixing procedures shown in Figure 5.2. The mixing energy required by each procedure can also be retrieved by calculating the area below the power consumption vs. time curve.

In order to calculate the stabilization time, the power value is given by the difference between mixing recorded power and that supplied to an empty mixer. This way the applied power is normalized in terms of peak power. This normalized curve is divided into two parts: before and after maximum peak power, which are fitted by linear and exponential mathematical functions respectively. The methodology is similar to that adopted by (CHOPIN; DE LARRARD; CAZACLIU, 2004; CHOPIN et al., 2007; MAZANEC; LOWKE; SCHIEßL, 2010).

An exponential fitting equation (Equation 5.1) is used to describe the power behavior for longer frames.

$$\frac{P}{P_{max}} = P_0 + P_1 e^{-\frac{t}{t_1}} + P_2 e^{-\frac{t}{t_2}} \quad (5.1)$$

The parameters P_0 , P_1 and P_2 describe the power range while t_1 and t_2 are the time dependence of the fitted power curve. The stabilization time is defined by (CHOPIN; DE LARRARD; CAZACLIU, 2004) as the time where the power-time curve reaches an asymptote. This value is given as the time needed for the gradient $\varepsilon = dP/dt$ to reach a value of (-0.0004) Figure 5.4. This criteria has been previously used for ultra-high performance concrete (UHPC) and self-consolidated concrete (SCC) and is adopted in this work as well.

Figure 5.4 – Definition of stabilization time based on the power curve gradient.

Once the stabilization time is reached, the mixed concrete exhibits optimal workability indicating that the mixing components are well dispersed. At this stage, different mixture procedures can also be compared.

5.2.2 Fiber homogenization test

A grid formwork is used to produce specimens for determining the fibers distribution, Figure 5.5. The methodology was adopted from Mazanec et al. (MAZANEC; SCHIEßL; GEHLEN, 2011). The fresh mixing is poured into the middle of

the formwork until a height of 50 mm is reached Figure 5.5a. Then a steel grid containing 16 open rectangular compartments is pushed into the fresh material. Finally the base of the formwork is removed to fill the containers (l/w/h = 125/125/150 mm³) below each compartment, Figure 5.5b.

Figure 5.5 – Grid formwork to determine fibers distribution in fresh ECC mortars (MAZANEC; SCHIEßL; GEHLEN, 2011).

The individual specimens in each container are weighted, loaded into buckets and mixed with water. Fibers are washed out of the mortar, collected by a fine sieve and weighted after they are washed. The fibers are then dried in climatic chamber at 105 °C during two days and after that they are burned at 450 °C in an oven so that fiber content can be calculated for each specimen. As shown in Figure 5.6, the sixteen specimens are grouped in four regions of interest to make the burning process easier and faster.

B-13	C-14	C-15	B-16
D-9	A-10	A-11	D-12
D-5	A-6	A-7	D-8
B-1	C-2	C-3	B-4

Figure 5.6 – Layout showing four different regions for fibers homogenization analysis: A, B, C, D.

Figure 5.7 shows the experimental setup used during the fiber homogenization tests. The fiber distribution over the four regions shown in Figure 5.6 is quantified by calculating the coefficient of variation. It is assumed that lower coefficient of variation indicates better distribution and homogenization of fibers within the mixture. For this reason, the different mixing procedures shown in Figure 5.2 are compared from the fibers homogenization point of view.

Figure 5.7 – Fiber homogenization test setup.

5.3 RESULTS AND DISCUSSION

5.3.1 Mixing behavior

With regard to mixing behavior, it is observed that power consumption increases with water addition (after 30 s of mixing) until a maximum peak is reached. After that the power consumption decreases exponentially with mixing time. Figure 5.8 shows the mixing curves obtained according to the mixing methods showed in Figure 5.2. First LF (low fluidity) mixtures are shown followed by MF (moderate fluidity) analysis.

As expected, lower power consumption is required by the reference mixture (REF) without fibers. It is seen that fiber addition at the beginning (ECC-Bef) increases the power consumption compared to other mixing procedure curves. When fibers are added after 180 s of mortar mixing (ECC-Aft) there is a power increase from this point on due to the fibers. Power fluctuation with fiber addition is also observed from this point on but it practically has no difference in terms of power level when compared to ECC-Bef. While the (ECC-Aft-4.9-2.5 m/s) mixing curve shows the best behavior meaning that hybrid mixing procedure has an important role in fibers distribution. ECC-Aft-4.9-2.5m/s procedure consists in mixing mortar under fast speeds (4.9 m/s) during 180 s. Right after that fibers are added during 20 s and mixed with mortar for another 150 s.

Figure 5.8 – Low fluidity mixing power consumption curves for high mixing intensity 4.9 m/s.

Figure 5.8 depicts the mixing curves obtained using a fast blade speed of 4.9 m/s. The Figure 5.9 shows the mixing curves for low speed blade 2.5 m/s. Observe that the maximum power peak is lower than those of curves shown in Figure 5.8. The previous analysis is also applied here for ECC-Bef and ECC-Aft showing similar mixing behavior. However with respect to the hybrid mixing procedure ECC-Aft-2.5-4.9 m/s

the effort applied to migrate from a low speed to a high one is higher than any procedure evaluated. Besides that the power fluctuation is also higher than what is seen in the others curves. This behavior in particular shows that more power consumption is required to reach a stable mixing level compared to other mixing procedures.

Figure 5.9 – Low fluidity mixing power consumption curves for low mixing intensity 2.5 m/s.

In terms of hybrid mixing procedure, the results show that it is more effective to provide more energy during the matrix dispersion so that fibers can be distributed using lower speeds without impairing the mixture homogenization. A hybrid mixing sequence has also been used in self-compacted concretes as reported by (LOWKE D; SCHIESSL P, 2005) through which the authors managed to further reduce mixing time.

Figure 5.10 shows a comparison between both mixing speeds. One should notice that power consumption increases with mixing intensity. This behavior is more visible when fibers are added before mixing Figure 5.10b. In terms of hybrid mixing procedure (Figure 5.10d), the comparison between fast-low and low-fast processes show that a more stable mixture is achieved when mortar is prepared at high speeds

and fibers added using low mixing speed. As mentioned by (MAZANEC; LOWKE; SCHIEßL, 2010), at the beginning of the mixing process high mixing intensity is required in order to rapidly distribute water and superplasticizer thus optimizing the mixing procedure. After that the material can be processed at lower speeds without impairing the mixing process.

Another aspect of mixing considered from this hybrid mixing process refers to the size and strength of agglomerates. Han and Ferron (HAN; FERRON, 2016) compared different mixing speeds looking into the microstructural changes of cement pastes via fresh state particle size measurements. They concluded that cement pastes prepared with high mixing intensity contained larger but weak agglomerates compared to pastes prepared at lower mixing intensity. From physics point of view, fibers get dispersed through the mixture when they are added using low mixing intensity (2.5 m/s) and the mortar was previously prepared under high mixing intensity. However when mortar is prepared at low mixing intensity and fibers added at higher mixing speed the fibers addition will increase the mixing effort. These results from two phenomena: 1) the agglomeration process which creates new bridges between fibers and reminiscent agglomerates and 2) by the agglomerate themselves breaking down. Both phenomena result in high power consumption fluctuations.

Figure 5.10 – Comparison between mixing intensities 4.9 m/s and 2.5 m/s.

Once the fluidity level increases, the mixing behavior also changes as to be seen in the next graphs. It is observed that either for high speed (Figure 5.11) or low speed (Figure 5.12) the differences between mixing processes are minimized. This behavior confirms that more fluidity means more distance between particles and thus fibers have more freedom to move through the mixture. For this reason fibers do not change the mixing behavior no matter what the fiber sequence addition may be.

Figure 5.11 – Power consumption curves for moderate fluidity MF. High mixing intensity 4.9 m/s.

The same analysis is also applied to low mixing speeds, Figure 5.12. The curves follow the same tendency of power increase with liquid addition until the maximum power consumption is reached. After this point, the power consumption decreases and a second peak is reached for (ECC-Bef) probably because fibers generate additional effort during the mixing process. Additionally, fibers can also contribute to create new agglomerates and the competition between forces of agglomeration and dispersion can cause this second power peak. This interpretation is in accordance with previous results on power consumption evolution with mixing time for concretes (CAZACLIU; ROQUET, 2009).

Figure 5.12 – Power consumption curves for low mixing intensity 2.5 m/s. Moderate fluidity mortars.

The comparison between the mixing intensities for moderate fluidity level is seen in Figure 5.13. This second peak after maximum cohesion point is observed for both mixing speeds since fibers are added at the beginning as shown in Figure 5.13b. In terms of fluidity level, this second peak is more evident for MF (Figure 5.13b) than for LF (Figure 5.10b) mortars.

Figure 5.13 – Comparison between mixing speeds for MF mortars.

Reference mortars REF (Figure 5.13a) show that high mixing intensity of 4.9 m/s achieves power stabilization level before low mixing speed 2.5 m/s. Also, the low speed curve (REF-2.5m/s) maintains its power decreasing tendency even when mixing ends.

5.3.2 Mixing stabilization time

The mixture stabilization time is calculated for REF and (ECC-Bef) procedures. Figure 5.14 shows the relation among mixture stabilization time, SP/binder content and mixing intensity. For both REF and reinforced mortars the stabilization time decreases with both mixing intensity and SP content. Except for (REF-2.5 m/s), Figure 5.14a, where stabilization time slightly increases with SP content.

Figure 5.14 – Relation between stabilization time (T_s) and SP/ binder for reference mortars REF a), and reinforced mortars b). Mixing intensity influence in the stabilization time. Low fluidity mortars c), and moderate fluidity mortar d).

Mixing intensity plays an important role in the stabilization time as mentioned elsewhere (MAZANEC; LOWKE; SCHIEßL, 2010). As an example for reinforced mixtures (ECC-Bef), it cuts down the mixing stabilization time in 70 s by just changing the mixing speed from 2.5 m/s to 4.9 m/s.

In addition to that, the stabilization time is higher for REF mortars than for reinforced mortars (ECC-Bef). One of the reasons for that is the air content increase with fiber addition, which is about three times higher in reinforced mortars as shown in Table 5.2.

5.3.3 Consistency, air content and mixing energy

The mortar fresh properties such as spread measured by mini slump-flow, air content and mixing energy per ton of composite are shown in Table 5.2. The diameter measured from the mortar spread in slump-flow test is greater for higher superplasticizer contents, i.e. MF mortars. Regarding the different mixing processes and their influence in the slump flow results, the slump flow coefficient of variation is higher for fast speed (4.9 m/s) than for low speed (2.5 m/s). This behavior means that most of the differences between mixing procedures are observed in high mixing speeds and low fluidity mortars.

Table 5.2 – Average spread measured from mini slump flow, air content by the pressure method and mixing energy per ton calculated from the area below the power consumption curve. LF- low fluidity; MF – moderate fluidity.

Mixing Process		Slump flow (mm)		Air Content (%)		Mixing Energy / ton [kWh/t]	
		LF	MF	LF	MF	LF	MF
4.9 m/s	REF	368	422	1.30%	0.90%	1.35	1.68
	ECC-Bef	217	193	3.10%	4.00%	1.73	1.75
	ECC-Aft	181	217	3.60%	4.30%	1.67	1.62
	ECC-Aft-4.9-2.5m/s	213	228	3.30%	3.50%	1.33	1.31
2.5 m/s	REF	393	400	1.60%	0.90%	0.74	0.80
	ECC-Bef	205	220	3.80%	2.90%	1.01	0.99
	ECC-Aft	229	243	2.90%	3.90%	0.93	0.95
	ECC-Aft-2.5- 4.9m/s	223	237	3.20%	4.00%	1.30	1.28

Reference mortars show higher slump flow and low air content compared to reinforced mortars, since the fibers increase the air content by almost three times for the ECC mortars in comparison to the reference (REF) counterpart. The mixing intensity also increases the air content but this tendency is not clear by looking at the values in Table 5.2.

In terms of energy spent during mixing process, the fibers addition before mortar mixing (ECC-Bef) increase the energy amount when compared to other mixing procedures. Therefore this procedure demands more energy of the mixer equipment. The hybrid mixing process (ECC-Aft-2.5- 4.9m/s) shows that more energy is also required to disperse fibers when the mortar is prepared in low speed and fibers are added in fast speed. Regarding the mixing process (ECC-Aft), less mixing energy is required than (ECC-Bef) for both parameters analyzed: fluidity levels and mixing speeds. This behavior not only means that ECC-Aft is the most efficient mixing procedure but also promotes good performance even for low speeds.

5.3.4 Rheological behavior

As mentioned before, rheological behavior is measured in the rheometer using a ball measuring system. Figure 5.15 shows torque vs. rotational speed curves for the reference mortars. Mixing intensity does not cause a significant difference in the rheological behavior of MF mortars, although mixing intensity influence is more noticeable for low fluidity (LF) mortars with torque levels higher than MF mortars.

Figure 5.15 – Torque vs. rotational speed curves for reference mortars without fibers.

Low torque values for reference mortars are present since high mortar fluidity is required in order to guarantee an acceptable flow with fibers. As seen in Figure 5.16,

the torque level of reinforced mortars is about three times higher than the reference mortar.

Figure 5.16 shows the comparison among the different mixing procedures in terms of ECC mortars rheological behavior. Low fluidity mortars (LF) show no difference between mixing procedure using low mixing intensity (2.5 m/s) since the flow curves are practically at the same level Figure 5.16b. For high mixing intensity (4.9 m/s) there is a slight torque level increase in (ECC-Bef) compared to others mixing procedures Figure 5.16a. Those results follow the same tendency found in the mixing behavior analysis.

Figure 5.16 – Comparison among different mixing procedures. Low fluidity a) and b). Moderate fluidity c) and d).

The curves of moderate fluidity mortars (MF) and high mixing intensity show that (ECC-Bef) and (ECC-Aft) are similar to each other while hybrid mixing procedure (ECC-Aft- 4.9-2.5 m/s) promotes an increase in torque level, Figure 5.16c. The higher torque level can be associated to remaining agglomerates from the mixture that are broken down during rheological tests since for this hybrid procedure fibers are dispersed using low mixing intensity.

Regarding low mixing speed (2.5 m/s) and moderate fluidity mortars (MF), the mixing procedure (ECC-Bef) differs from others by a slight increase of torque level. This behavior indicates that it is not useful to prepare the mixture with fibers addition at the beginning if low mixing intensity is used.

In the end, it is seen that (ECC-Aft) promotes a more reproducible rheological behavior among all the procedures evaluated. Furthermore, (ECC-Aft) procedure is not directly affected by different parameters such as mixing intensity or matrix fluidity, which shows its robustness.

When focusing on mixing intensity analysis both low fluidity (LF) and moderate fluidity (MF) mortars show an increase in torque levels and consequently in the composite yield stress for low mixing intensity (2.5 m/s), Figure 5.17.

Figure 5.17 – Comparison between mixing intensities 4.9 m/s (fast speed) and 2.5 m/s (low speed). For low fluidity mortars a), b) and c). Moderate fluidity d), e) and f).

The apparent yield stress and apparent viscosity are calculated from the flow curves torque vs. rotational speed and the results are shown in Table 5.3. For low fluidity mortars (LF) and low mixing intensity (2.5 m/s) there is an increase in both viscosity and yield stress. ECC-Aft procedure shows low yield stress values for all conditions evaluated which means that the material requires less stress to initiate flow.

Table 5.3 – Rheological parameters from flow curves torque vs. rotational speed.

Mixing Process		LF			MF		
		Yield stress - 10^{-3} (Nm)*	Viscosity - 10^{-3} (Nm.s/rad)	R ²	Yield stress - 10^{-3} (Nm)*	Viscosity - 10^{-3} (Nm.s/rad)	R ²
4.9 m/s	REF	~0	10.84	0.97	~0	4.24	0.97
	ECC-Bef	35.20	47.12	0.98	30.10	40.05	0.99
	ECC-Aft	28.20	45.71	0.98	26.70	41.00	0.97
	ECC-Aft-4.9-2.5m/s	28.10	42.41	0.99	37.50	47.59	0.98
2.5 m/s	REF	~0	8.48	0.97	~0	3.77	0.97
	ECC-Bef	35.70	57.49	0.95	35.70	52.31	0.99
	ECC-Aft	34.10	64.56	0.99	29.70	45.71	0.95
	ECC-Aft-2.5-4.9m/s	31.10	61.73	0.98	27.20	51.36	0.97

*Negative yield stress values are assumed to zero.

The yield stress is also calculated through mini slump flow values using Roussel's model (ROUSSEL; STEFANI; LEROY, 2005). According to Figure 5.18 yield stress values decrease exponentially with slump diameter. It seems that LF and MF show similar behavior of decreasing yield stress with slump diameter. REF mortars show very low yield stress values and high diameter spread. On the other hand while ECC mortars are within the slump flow diameter range of 150 to 250 mm the yield stress shows high variation (from 15 to 70 Pa).

Figure 5.18 – Yield stress calculated through Roussel's model.

Comparing the different mixing procedures, it is seen in Figure 5.19 that the yield stress calculated by Roussel's model decreases with mixing intensity decreasing for both fluidity levels MF and LF except for ECC-Bef-LF. It was expected a behavior similar to ECC-Bef-LF for all mixing procedures since by increasing the mixing speed the particles dispersion should improve and consequently cause a decrease in yield stress. However the opposite is observed in this analysis.

Figure 5.19 – Yield stress calculated according to Roussel's model. Comparison among different mixing processes.

Since the yield stress results calculated using Roussel's equation differ from results obtained in the rheometer, a possible explanation for it is that either the spread flow diameter is not high enough to fit a Roussel model or the slump flow measurement features are inconsistent. Slump flow is a qualitative measure of workability and it measures only one point that represents everything about sample while rheological measurements in the rheometer can evaluate the composite at different rates. For this reason the values from rheometer are being taken into account in order to evaluate the sample rheological properties.

5.3.4.1 Shear stress and viscosity

For all mixing procedures evaluated, the flow curves are shown in Figure 5.20. Low fluidity (LF) mortars are shown on the Figure 5.20 left side while moderate fluidity (MF) on its right side.

For low fluidity mortars (LF) viscosity decreases with shear rate but the differences between mixing procedures are not pronounced even though composites prepared at low speed show a slight viscosity increase, Figure 5.20c. The shear stress shear rate curves show more dispersion which can be associated to each mixing procedure. This analysis also shows that shear stress values are higher at low mixing

intensity (2.5 m/s) Figure 5.20a. Also MF and LF mortars follow the same tendency for shear stress and viscosity Figure 5.20.

Figure 5.20 – Shear stress a) and b), viscosity c) and d). Low fluidity mortars in left side. Moderate fluidity mortars in right side.

The yield stress and viscosity parameters are calculated by linear regression from shear stress vs. shear rate curves from Figure 5.20. The results in Table 5.4 were generated using Bingham model, which establishes a linear relation between shear stress and shear rate. Also, a good relation expressed by R-square is found when using the Bingham model to calculate rheological parameters.

For reference mortars (REF) and also for reinforced mortars the viscosity decreases with SP content. This decrease is stronger for reference mortar by an order of 60% while for reinforced mortars the difference between low and moderate fluidity is around 10%.

Table 5.4 – Yield stress and viscosity calculated from shear stress shear rate curves.

Mixing Process		LF			MF		
		Yield stress (Pa)	Viscosity (Pa.s)	R ²	Yield stress (Pa)	Viscosity (Pa.s)	R ²
4.9 m/s	REF	~0	13.76	0.97	~0	5.33	0.97
	ECC-Bef	311	59	0.98	266	50	0.99
	ECC-Aft	249	57	0.98	236	52	0.97
	ECC-Aft-4.9-2.5m/s	248	53	0.99	331	59	0.98
2.5 m/s	REF	~0	10.58	0.97	~0	4.46	0.97
	ECC-Bef	316	72	0.95	318	65	0.99
	ECC-Aft	301	67	0.98	262	57	0.95
	ECC-Aft-2.5-4.9m/s	275	77	0.98	241	64	0.97

*Negative yield stress values are assumed to zero.

Yield stress also decreases with SP content except for (ECC-Aft-4.9-2.5 m/s) and (ECC-Bef-2.5 m/s) mixture procedures. The latter (ECC-Bef) shows higher yield stress value compared to the others mixing procedures emphasizing that fibers addition at the beginning of mixture is the worst practice when preparing reinforced mortars.

The hybrid mixing process (ECC-Aft-4.9-2.5 m/s) is deemed very suitable since it shows low yield stress and viscosity values mainly for low fluidity mortars. However, after further analysis of all mixing conditions, the (ECC-Aft) procedure is seen to promote low yield stress as well as low viscosity independently of mortar fluidity or mixing intensity. This result once more confirms that ECC-Aft provides better performance in terms of rheology and mixing behavior. Additionally, this mixing procedure tolerates changes and variations in materials (i.e. SP content) as well as in the process (i.e. mixing intensity). It also shows more robustness which is a very important property in order to expand the use of this composite.

5.3.4.2 Static yield stress and thixotropy

As discussed in Chapter 4, a minimum static yield stress value of 3 Pa is necessary to maintain a stable composite. Values below 3 Pa increase the risk of bleeding for ECC mortars. The static yield stress curves in Figure 5.21 and Figure 5.22

show that the minimum criteria is reached for LF mortars Figure 5.21 but not completely for all MF mortars Figure 5.22.

Figure 5.21 – Static yield stress for low fluidity (LF) mortars. Low mixing intensity curves are drawn with solid lines and high mixing intensity (4.9 m/s) with dashed lines.

For LF mortars, one can see a static yield stress increase with time, Figure 5.21. This behavior shows a flocculation phenomenon and it is associated to mortar stability. Despite that, no flocculation is observed with superplasticizer content increase. The static yield stress decreases with the rest time instead Figure 5.22. This behavior results from mortar segregation during the rheological tests. When comparing fluidity levels for both LF and MF, low fluidity mortars show more stability than moderate fluidity mortars for this experimental setup.

Figure 5.22 – Static yield stress for MF mortars.

Thixotropy (A_{thix}) is also calculated from the gradient of static yield stress vs. resting time curves. As it is seen in Table 5.5, for low fluidity mortars (LF) a linear correlation is found and the structural rate is higher for mixtures prepared using high mixing speed (4.9 m/s). Despite that, according to Roussel's criterion (ROUSSEL, 2006) all mixtures are considered non-thixotropic due to their low flocculation rate (less than 0.1 Pa/s). This means that the composite is suitable for slab casting which is acceptable for this kind of application.

Table 5.5 – Thixotropy behavior of LF and MF mortars.

Mixing Process		LF		MF	
		$A_{thix} (x10^{-3})$ Pa/s	R^2	$A_{thix} (x10^{-3})$ Pa/s	R^2
4.9 m/s	REF	1.8	0.972	3.00E-05	0.70
	ECC-Bef	8.5	0.970	2.00E-01	0.22
	ECC-Aft	8.4	0.990	~0	0.89
	ECC-Aft-4.9-2.5 m/s	9.4	0.991	~0	0.73
2.5 m/s	REF	0.6	0.956	8.00E-03	0.95
	ECC-Bef	4.4	0.999	~0	0.07
	ECC-Aft	5.7	0.998	~0	0.08
	ECC-Aft-2.5- 4.9 m/s	6.0	0.997	3.00E-01	0.31

*Negative yield stress values are assumed to zero.

For moderate fluidity mortars (MF), the static yield stress does not increase linearly with resting time. Lower flocculation rate is also found which can be related to paste segregation.

The thixotropy analysis shows that low fluidity mortars are more stable than moderate fluidity mortars. Indeed, mixing intensity has more influence in the flocculation rate (A_{thix}) than mixing procedure, i.e. the moment at which fibers are added.

5.3.5 Fiber Homogenization Test (FHT)

Fresh state fiber homogenization test analysis is shown in Table 5.6. The coefficient of variation is calculated for each mixing process. The lower the coefficient of variation the more homogenized are fibers in the mortar. This means that the mixing procedure is more efficient to promote fibers homogenization and dispersion. From this analysis one concludes that the mixing procedure (ECC-Aft), which consists in preparing the mortar first and then adding fibers afterwards, is seen to be the best procedure in terms of fibers homogenization among all of them.

Table 5.6 – Standard deviation (SD) and coefficient of variation (CV) for fiber homogenization test analysis.

Mixing Process		LF		MF	
		SD	CV	SD	CV
4.9 m/s	ECC-Bef	3.20	0.03	4.11	0.04
	ECC-Aft	5.19	0.05	3.91	0.05
	ECC-Aft-4.9-2.5m/s	8.07	0.07	-*	-*
2.5 m/s	ECC-Bef	10.87	0.10	5.45	0.05
	ECC-Aft	2.25	0.02	3.44	0.04
	ECC-Aft-2.5- 4.9m/s	12.07	0.11	-*	-*

*Results were impaired due to fibers burn process.

Comparing mixing intensities it is observed that for LF mortars the low mixing intensity (2.5 m/s) standard deviation is 53% higher than high mixing intensity (4.9 m/s). On the other hand, this difference reduces to 10% for MF mortars.

Since mixing intensity is high, it does not matter if fibers are added after (ECC-Aft) or before (ECC-Bef) preparing the mortar. However, for low mixing intensity the fiber addition sequence is very important. That is the reason why the ECC-Aft procedure improved fiber homogenization. This analysis also proves that the ECC-Aft procedure can be used independently of mixer capacity.

Furthermore a mechanical performance improvement is expected if fibers are well homogenized and dispersed after the composite mixing process. As mentioned by (LARANJEIRA et al., 2012), the mixing process as well as the concrete fresh-state properties are stages of interaction in which fiber orientation is modified and both have influence on the fiber orientation in the hardened-state.

5.3.6 Correlation between mixing behavior, rheology and fibers homogenization

A correlation among mixing behavior, rheology and fiber homogenization is evaluated in this subsection. At first analysis, mixing behavior is related to the fiber homogenization as illustrated in Figure 5.23. High mixing intensity (4.9 m/s) shows higher mixing energy and better fiber homogenization which is indicated by low

coefficient of variation. By contrast low mixing intensity (2.5 m/s) shows that fibers homogenization becomes worse with mixing energy increase.

Figure 5.23 – Mixing energy influences on the fibers homogenization coefficient of variation.

Regarding mixing procedures Figure 5.23 also shows that (ECC-Aft) mixing procedure improves the fibers homogenization even for low mixing speeds. This result has an important role in terms of application since it determines that the simple action of adding fibers after preparing the mortar can improve fibers homogenization. This effect is more visible for low capacity mixer.

If the mixer is powerful enough to prepare mixtures at high speeds, it does not matter when fibers are added, i.e. at the beginning or after mortar preparation.

Regarding the rheological behavior, the yield stress and viscosity parameters are related to fibers homogenization as shown in Figure 5.24. For low mixing intensity (2.5 m/s) there is no clear correlation between rheological behavior and fibers homogenization. Despite of that it is seen that low yield stress values increases the coefficient of variation which means that fibers are not well dispersed in this condition, Figure 5.24a.

Figure 5.24 – Relation between rheological parameters and fibers homogenization.

In terms of viscosity, a linear correlation between viscosity and fibers homogenization was found, Figure 5.24b. The coefficient of variation for fibers homogenization increases with viscosity for high mixing intensity (4.9 m/s). Low mixing intensity (2.5 m/s) does not follow this behavior since viscosity increases with coefficient of variation instead. Additionally, for low mixing intensity (2.5 m/s), a slight increase in viscosity causes a high coefficient of variation, i.e. fibers are not very well dispersed. In summary, a qualitative comparison between mixing behavior, rheology and fiber homogenization is found in Table 5.7. It is also shown that some parameters have more influence on the fresh properties than others, or at least they affect one property particularly.

Table 5.7 – Relation between parameters of analysis and fresh-state properties.

Parameters	Mixing behavior	Rheology	Fiber homogenization test
SP content	Differences between mixing procedures decrease with SP content	Viscosity and yield stress decrease with SP content	Improves fiber homogenization.
Mixing intensity	More stable mixing for high mixing intensity	Increases viscosity and yield stress	Low mixing intensity decreases fibers homogenization
Fibers addition before mortar mixing	Increases mixing efforts	Increases viscosity and yield stress.	No improvement
Fibers addition after mortar mixing	Reduced mixing energy	Reduces viscosity and yield stress. Improving the composite robustness	Improves fiber homogenization.
Hybrid mixing process (low-fast)	Increases mixing energy	Increases viscosity	No improvement
Hybrid mixing process (fast-low)	Reduced mixing energy and air content	Decreases yield stress and viscosity for low fluidity mortars.	No improvement

5.4 CHAPTER CONCLUSIONS

First of all, mixing intensity influences fiber homogenization and high mixing intensity promotes more uniform dispersion of fibers. It is shown that for LF mortars the low mixing intensity (2.5 m/s) standard deviation is 53% higher than for high mixing intensity (4.9 m/s). On the other hand, this difference reduces to 10% for MF mortars. Moreover, the fiber addition sequence plays an important role mostly for low mixing intensity, which is also associated to mixer power. When mixing intensity is high (4.9 m/s), it does not matter if fibers are added after or before preparing the mortar. However for low mixing intensity the fiber addition sequence is very important. That is the reason why the ECC-Aft procedure improved fiber homogenization. This analysis also proves that the ECC-Aft procedure can be used independently of mixer capacity. Furthermore this mixing process, where fibers are added after preparing the mortar (ECC-Aft), reduces mixing energy by around 8% compared to the other mixing process.

In terms of viscosity and yield stress for low mixing intensity (2.5 m/s), there is no clear correlation between rheological behavior and fibers homogenization. However, for fibers homogenization, a coefficient of variation increase with yield stress decrease is observed, which means that fibers are not well dispersed in this condition. Also, for viscosity analysis a linear relation between viscosity and fiber homogenization was found. For low mixing intensity (2.5 m/s), a slight increase in viscosity causes a high coefficient of variation thus impairing fiber homogenization.

Finally, there is a correlation between mixing behavior and fiber homogenization. High mixing intensity (4.9 m/s) shows higher mixing energy and better fiber homogenization which is indicated by a low coefficient of variation. By contrast, low mixing intensity (2.5 m/s) shows that fibers homogenization becomes worse with mixing energy increase. So if the mixer is powerful enough to prepare mixtures at high speeds, it does not matter if fibers are added at the beginning or after the mortar preparation. But for low capacity mixers the sequence of fiber addition has a large impact on fibers homogenization.

6

Link between rheological and mechanical behavior of PVA-ECC mortars

6.1 INTRODUCTION

The link between rheological and mechanical performance of fiber reinforced mortars is the target of this chapter. The influence of parameters like mixing procedure, mixer type and mortar fluidity on the composite mechanical performance and rheological behavior are investigated. The mechanical performance is evaluated in terms of flexural behavior, compression strength and fracture energy using bending tensile tests while the rheological properties are measured after the mixing process using a rheometer equipped with ball measuring system.

By means of the combined analysis of those different parameters, correlations between mechanical performance and rheological behavior are analyzed. At the same time attempts are made to identify if changes in the mixing process significantly affect the composite rheology and its mechanical performance.

6.2 EXPERIMENTAL PROGRAM

All the raw materials used for these experiments were discussed in Chapter 4, along with the fiber type and volume. Apart from that two different mixers were used to prepare the mixtures, Figure 6.1. A comparison between both mixers in terms of rheological behavior and mechanical properties was also performed.

Figure 6.1 – Mixer type. a) Intensive mixer EIRICH-75, with volume capacity of 75 L. b) Small mixer EIRICH-5 with volume capacity of 5 L.

An intensive inclined drum mixer (Eirich-75) with 75 L of capacity and controllable tool speed was available to prepare ECC mortars for these examinations, Figure 6.1a. The batch volume was kept constant and equal to 25 liters. Different mixing procedures, two fluidity levels LF and MF with superplasticizer/binder content of 0.636% and 0.773% respectively, and two mixing speeds (4.9 m/s and 2.5 m/s) were evaluated using this mixer as shown in Chapter 5 and in Figure's 6.2 left side. The mixing sequence details for this mixer type are presented in Chapter 5.

Figure 6.2 – Experimental setup using both mixers. Big mixer with 75 L capacity (on the left) and small mixer with 5 L capacity on the right.

Additionally, a second mixer called Eirich-5 with 5 L capacity was used, Figure 6.1b. All tests using this mixer (Eirich-5) were carried out with a vessel velocity of $v_1 = 45$ rpm and the impeller set at velocity $v_2 = 200$ rpm (1.05 m/s). The mixer was configured in counter current configuration; i. e. the vessel and device rotate in opposite direction. The batch volume kept constant and equal to 2 liters in all tests. Batches were carried out at a temperature of 20 ± 3 °C. Solid materials were loaded before starting the mixer. After mixing the solid constituents during 30 s to ensure proper materials dispersion and homogenization, water containing dissolved liquid admixture was progressively added during the next 10 s. The solid materials and liquids were then mixed for another 3 min. Next, fibers were added during 20 s and mixed with mortar during 60 s. The total mixing time without fibers (REF) was constant and equal to 5 min.

Therefore, as seen in Figure 6.2 (left), two fluidity levels (LF and MF) and four mixing procedures are evaluated using Eirich-75 while for Eirich-5 the mixing procedure is fixed (fibers are added only after the mortar has been prepared) and three fluidity levels (low fluidity - LF, moderate fluidity - MF and high fluidity - HF) are studied Figure 6.2 (right). The superplasticizer / binder relation (SP/binder) for each fluidity level is as follows: LF = 0.636%, MF = 0.773% and HF = 0.909%.

6.2.1 Rheological measurements

Ball Measuring System (BMS) is the rheometer configuration type used to measure rheological behavior. The test program as well as the equations used to calculate the rheological parameters are shown in Chapter 4. All rheological measurements were performed 13 min after the first contact between water and solid materials.

6.2.2 Casting and curing

The mixture was prepared according to the mixing process under evaluation and then placed horizontally into molds without applying vibration. The specimens casts were covered with lids and stored in a climatic chamber for 24 h at temperature of 23 °C. After that, specimens were demolded and cured underwater at 21 °C temperature until 28 days. Afterwards the mechanical tests were performed.

6.2.3 Test methods

The flexural properties were measured 28 days after the specimens (without notch) were cast. A three-point bend configuration was used to measure the flexural strength (MOR) and fracture energy. For this setup a servo-controlled test machine *Walter + bai ag* model is used with loading rate of 0.005 mm/s. For each condition a total of six specimens (40 x 40 x 160 mm) undergone flexural tests. After flexural tests, the specimens were used to evaluate the compressive strength.

The flexural strength was calculated according to equations (6.1):

$$MOR = \frac{3Pl}{2bd^2} \quad (6.1)$$

P is the maximum load, l is a support span of 100 mm, b and d are respectively the specimen's width and depth (measured at the region of failure).

Through integration of load-deflection curve measures the fracture energy to the point corresponding to a reduction in load carrying capacity to 50% of the

maximum observed. According to (SAVASTANO JR.; WARDEN; COUTTS, 2000), the fracture toughness is given by the relation between fracture energy and fracture area as follows:

$$\text{Fracture toughness} = \frac{\text{fracture energy}}{bd} \quad (6.2)$$

An overview of the main experimental steps is seen in Figure 6.3.

Figure 6.3 – Overview of experimental steps.

6.3 RESULTS AND DISCUSSION

6.3.1 Rheological behavior

The rheological behavior in terms of yield stress and viscosity can be used to evaluate the material final structure after mixing giving an indication of the mixer efficiency as mentioned by (DILS; DE SCHUTTER; BOEL, 2012). Thus not only mixer types are compared but also different mixing processes can be investigated via rheological measurements.

The results for shear stress in Figure 6.4a as well as viscosity in Figure 6.4b show that by using a small mixer (Eirich-5) the SP/binder ratio strongly affects the mortar rheological behavior. A large difference between LF and MF mortar fluidity is

also observed. Besides that, the apparent yield stress calculated via flow curves (torque vs. rotational speed) shows that the apparent yield stress of LF mortars is 2.25 times higher than MF mortars.

On the other hand, the rheological behavior of mixtures prepared using a bigger mixer (Eirich-75) shows a slightly difference between fluidity levels (LF and MF) as well as between the mixing speeds evaluated. In addition the shear stress and viscosity curves (Eirich-75) are situated between the LF and MF curves of Eirich-5. This behavior indicates that mixer capacity has a big influence in terms of materials dispersion since better dispersion leads to more stable systems. In addition, from the practical point of view is also possible to decrease the superplasticizer content if the mixer efficiency increases accordingly.

Figure 6.4 – Shear stress (a) and viscosity (b). Grey dashed curves refers to mixtures prepared using the big mixer (Eirich-75) while continuous lines refers to the small mixer -MF2% and LF2% - (Eirich-5).

The rheological properties of those mixtures are found in Table 6.1. According to the results shown in Table 6.1, mixtures prepared using low mixing speeds (ECC-Aft-2.5m/s) or low mixer capacity (Eirich-5) present higher yield stress. And because yield stress is a measure of the shear stress required to initiate flow (MAHMOODZADEH; CHIDIAC, 2013; COUSSOT, 2014; DINKGREVE et al., 2016) the higher it is the yield stress value more difficult is to use this material. Another fundamental characteristic of yield stress is it prevents the material flowing from unless it is submitted to a stress greater than a critical value (COUSSOT, 2014).

As reported previously, particle dispersion increases by increasing the mixing intensity. In this way, agglomerates are also reduced promoting a decrease in both inter-particle surface contacts and friction forces which increases fluidity (VANCE; SANT; NEITHALATH, 2015) and consequently reducing yield stress and viscosity values.

Viscosity is a measure of the material resistance to flow after it begins to flow (MAHMOODZADEH; CHIDIAC, 2013). Consequently, viscosity increases when

mixtures are prepared in low mixing speed. However viscosity values also are higher for high mixer capacity (Eirich-75) than for low mixer capacity (Eirich-5).

Furthermore, as expected, all ECC mixtures with fibers show higher yield stress and viscosity when compared to REF mortars. With respect to LF mortars, the viscosity factor of 2% vol. fiber (Viscosity Factor [VF] = reinforced mortar viscosity / reference mortar viscosity) is 1.5 times higher than that of the reference mortar when the mixture is prepared using an Eirich-5 mixer. For Eirich-75 this factor is much higher (about six times more). Additionally, for MF mortars the viscosity factor increases by a magnitude of 3 compared to LF mortars for mixtures prepared using EIRICH-5 and almost 2 for EIRICH-75. As consequence, the viscosity factor is high the higher the mortar fluidity and mixer efficiency.

Table 6.1 – Rheological properties calculated from shear stress vs. shear rate curves.

Mixer	Mixing Process	LF			MF		
		Yield stress (Pa)	Viscosity (Pa.s)	R ²	Yield stress (Pa)	Viscosity (Pa.s)	R ²
Eirich-75	REF-4.9m/s	~0	13	0.97	~0	5	0.97
	REF-2.5m/s	~0	10	0.97	~0	4	0.97
	ECC-Aft- 4.9 m/s	249	57	0.98	236	51	0.97
	ECC-Aft-2.5 m/s	301	66	0.98	262	57	0.95
Eirich-5	REF	84	37	0.99	9	7	0.99
	ECC-Aft	482	56	0.98	214	36	0.95

*Negative yield stress values are assumed to be zero.

6.3.2 Mechanical performance

6.3.2.1 Influence of mixing procedure

Different mixing procedures were investigated using the big mixer (Eirich-75) and their influence on the mechanical performance, presented via load vs. deflection curves, are seen in Figure 6.5 and Figure 6.6 for LF and MF mortars respectively. Since for every mixing procedure six specimens were evaluated, each load deflection curve corresponds to the result of one specimen tested.

Figure 6.5 – Load deflection curves for different mixing procedures using Eirich-75. High mixing intensity (4.9 m/s) are shown on the left side while low mixing intensity curves (2.5 m/s) on the right.

According to Figure 6.5a,b, fiber addition at the beginning of the mixing process increases dispersion for both LF and MF mortars. A hybrid mixing procedure shows good results for ECC-Aft-2.5-4.9 m/s since it decreases dispersion among all measured curves, Figure 6.5f. Thus, when fibers are added in a high speed mixture process the fibers dispersion increases which improves the mortar mechanical performance.

Moreover, an up-close view of all mixing procedures indicate that when fibers are added after the mortar is prepared (ECC-Aft) its mechanical performance improves independently of either mixing speed or mortar fluidity.

One should note that for MF mortars (Figure 6.6) the dispersion among the load vs. deflection curves is low than for LF mortars. Not only this but the maximum load is also lower.

Figure 6.6 – Effect of mixing procedure on the load deflection curves for moderate fluidity (MF) mortars.

Figure 6.7 shows a comparison among mixing procedures in terms of representative curve. One curve of each group is chosen to represent the overall mixing procedure so that they can be easily compared this way. Looking at the low fluidity mortars (left side of Figure 6.7), ECC-Aft and ECC-Aft-4.9-2.5m/s procedures are very similar while ECC-Bef shows a decrease in both deflection at ultimate load and deflection at failure. For moderate fluidity MF mortars (right side of Figure 6.7) it is seen that the mixing procedure has no significant influence on the load deflection

curves, except for ECC-Bef (Figure 6.7d) which shows a slight difference with decrease of deflection at ultimate loading.

Figure 6.7 – Comparison among mixing procedures in terms of representative curve. Low fluidity mortars a) and b). Moderate fluidity mortars c) and d).

Regarding the ECC-Aft procedure where fibers addition occurs after mortar mixing, it is seen that high mixing intensity (4.9 m/s) produces a more reproducible mechanical behavior independent of mortar fluidity Figure 6.7a,c. For low mixing intensity (2.5 m/s) and low fluidity mortars this procedure achieves better mechanical performance Figure 6.7b. Moreover, the load increase is higher than other mixing procedures as shown in Figure 6.8. Besides that fiber addition after the mortar mixture is prepared, (ECC-Aft procedure) comes on top as the most robust procedure amongst all mixing procedures evaluated. Also, for this procedure the different changes in the fresh state such as mixing intensity and fluidity level do not have a big impact on the mechanical performance.

Figure 6.8 – Influence of mixing design on the bending test for mixing procedure ECC-Aft.

The flexural strength as well as the compressive strength for the mixtures prepared using Eirich-75 are seen in Table 6.2. Please note that the compressive strength for LF mortars is 8% higher than for MF mortars while for flexural strength this difference decreases to 5%.

Table 6.2 – ECC mortars properties prepared using Eirich-75 mixer.

Mixing intensity	Mixing procedure -	Flexural strength (MPa)	Coefficient of variation	Compressive strength (MPa)	Coefficient of variation
LF_ 4.9 m/s	ECC-Bef	11.60	0.12	72.76	0.02
	ECC-Aft	14.13	0.10	76.37	0.03
	ECC-Aft-4.9-2.5m/s	14.37	0.17	76.93	0.03
LF_ 2.5 m/s	ECC-Bef	10.56	0.11	75.33	0.03
	ECC-Aft	14.37	0.12	73.40	0.02
	ECC-Aft-4.9-2.5m/s	14.87	0.06	73.18	0.03
MF_ 4.9 m/s	ECC-Bef	11.96	0.10	65.92	0.03
	ECC-Aft	11.95	0.09	67.60	0.02
	ECC-Aft-4.9-2.5m/s	11.24	0.04	68.24	0.04
MF_ 2.5 m/s	ECC-Bef	14.91	0.05	69.61	0.03
	ECC-Aft	13.02	0.15	70.33	0.02
	ECC-Aft-4.9-2.5m/s	12.55	0.06	69.40	0.04

Regarding mixing intensity there is no difference between 2.5 m/s and 4.9 m/s for LF mortars in terms of compressive and flexural strength. Although for MF mortars low mixing intensity (2.5 m/s) improved its mechanical performance when compared to high mixing intensity (4.9 m/s), the flexural strength increased by 13% for all mixtures prepared using low mixing intensity. This can be attributed to increased air content for high mixing intensity MF mortars which increases porosity and impairs its mechanical performance.

6.3.2.2 Influence of mixer type

First the load vs. deflection curves for mixtures prepared using the small mixer (Eirich-5) are shown in Figure 6.9 and then the results for both mixers are compared. As seen in Figure 6.9 (left side) the reference mortar without fibers exhibits a brittle behavior losing their tensile load-carrying capacity almost immediately after the first crack, whereas fibers addition significantly improves the post-cracking flexural response, Figure 6.9 (right side).

Regarding the mortar fluidity results, Figure 6.9 shows that for the reference mortar, this parameter practically has no influence on the load vs. deflection curve while for reinforced matrix there is an improvement in both deflection at ultimate load and deflection at failure when the super-plasticizer content increases. Conversely, dispersion increases among the curves with an increase in mortar fluidity.

Figure 6.9 – Influence of mortar fluidity on the load deflection curves. Mixtures prepared using Eirich5. Reference mortar presented on the left side and ECC mortars on the right.

In the analysis of mortar fluidity in terms of representative curve for each group of results, it is observed that toughness increases with mortar fluidity, Figure 6.10. This behavior can be attributed to the bonding between matrix and fibers as well as by to the matrix strength. As reported by (QIU; LIM; YANG, 2016), the quality of interfacial bond between fibers and matrix determines the reinforced composite post-cracking behavior. Due to the presence of the hydroxyl (OH) group in PVA fiber the

chemical bond is high (LI et al., 2002) which when associated to a high strength matrix can lead to fibers rupture before they are pulled out (KANDA; LI, 2006).

The aforementioned increase in SP content improved the interface between fibers and matrix improving the composite toughness although reducing its compressive strength as seen in Table 6.3. Similar behavior was also observed when the mixtures are prepared using an intensive mixer as shown in Figure 6.11.

Figure 6.10 – Mortar fluidity comparison in terms of representative curves.

Table 6.3 shows mechanical properties such as flexural behavior and compressive strength for mortars prepared using the small mixer (Eirich-5). Regarding compressive strength, reinforced mortars achieved a 10.6% increase compared to the reference mortar and the highest compressive strength was found in MF-ECC mortar. In terms of flexural strength, reinforced mortars exhibit an increase of 34% compared to reference. By the way the highest flexural strength was found in LF-ECC mortars. Additionally, it was observed that the matrix strength diminishes with an increase in SP content. As mentioned before, this behavior has direct impact on the mortar toughness.

Table 6.3 – Flexural strength and compressive strength of REF and ECC mortars prepared using the small mixer Eirich-5.

Mixing procedure	Fluidity	Flexural strength (MPa)	Coefficient of variation	Compressive strength (MPa)	Coefficient of variation
REF	LF	8.79	0.09	69.99	0.03
	MF	9.50	0.05	69.90	0.04
	HF	8.91	0.08	58.73	0.16
ECC-Aft	LF	15.32	0.08	76.67	0.02
	MF	14.54	0.09	77.88	0.03
	HF	11.36	0.15	67.61	0.08

Comparing the values in Table 6.3 for Eirich-5 with those obtained using the Eirich-75 in Table 6.2, for the same fluidity level of LF and MF, composites prepared using a small Eirich-5 mixer show high compressive strength and flexural behavior but not necessarily better mechanical performance since different aspects like volume of material and mixing intensity for instance play an important role in particles dispersion. Another important aspect pointed out by (DILS; DE SCHUTTER; BOEL, 2012) is that admixture quantities can often be reduced because of the better mixing process found in an intensive mixer type.

Figure 6.11 shows the mixer influence on flexural behavior. Deflection-hardening behavior with improved post-cracking flexural strength is observed for mixtures prepared using the big mixer (Eirich-75). Only HF mixtures display similar behavior to the mixtures prepared in the big mixer.

Figure 6.11 – Mixer influence on flexural behavior. Gray curves for mortars prepared in the big mixer (Eirich-75).

The fracture toughness of the curves shown in Figure 6.11 is presented in Table 6.4. Mixtures prepared using the big mixer (Eirich-75) show fracture toughness higher than mixtures prepared using the small mixer (Eirich-5). Also, the higher the superplasticizer content the higher is the fracture toughness when using the small mixer. On the other hand, SP content has basically no influence on the fracture toughness when using the big mixer. Mixing intensity has more influence instead. Surprisingly, fracture toughness decreases with an increase in mixing intensity and this fact is more noticeable for LF mortars.

Table 6.4 – Fracture toughness of mixtures prepared using the small Eirich-5 mixer and the big one Eirich-75.

Mixer	Mixture	Fracture toughness (kJ/m ²)
Eirich-5	LF-2%	4.98
	MF-2%	5.69
	HF-2%	7.48
Eirich-75	ECC-AFT-2.5m/s_LF	8.79
	ECC-AFT-4.9m/s_LF	7.17

	ECC-AFT-2.5m/s_MF	7.26
	ECC-AFT-4.9m/s_MF	7.15

In general, it seems that the higher the mixer efficiency the more disperse are the particles, meaning more free water in the mixture. If on one side this contributes to improve the fluidity of material on the other side it may reduce its mechanical properties. This analysis also hints that the mix design can be improved in terms of superplasticizer content reduction or even mixing time as long as more robust mixer is used.

6.3.3 Link between rheological behavior and mechanical performance

Regression analysis were performed to find possible correlations between rheological and mechanical properties. The results show that the data approximately fits the quadratic function. As seen in Figure 6.12, curves move upwards for both yield stress and viscosity. Moreover, it is seen that by either doubling the yield stress or viscosity a variation of more than 50% in flexural strength is achieved.

Figure 6.12 – Correlation between rheological properties and flexural strength. a) Yield stress, b) viscosity.

A previous research work shows that plastic viscosity increased enhances mechanical properties since it can promote better fiber distribution and bridging

efficiency in mixtures (YANG et al., 2009). However, while high plastic viscosity is desirable for better fiber distribution, low yield stress is required for better ECC deformability although no correlation between yield stress and mechanical properties was found (YANG et al., 2009). In this work a range of variations among rheological and mechanical properties is proposed since parameter such as mortar fluidity, mixing procedure and mixer type are taken into account.

Regarding these correlations in terms of compressive strength, Figure 6.13 shows that both the yield stress and viscosity curves move downwards. It means that there is an increase of viscosity as well as yield stress with compressive strength (MF mortars) until reaching a certain point when the viscosity and yield stress begin to decrease with compressive strength (LF mortars).

Figure 6.13 – Correlation between rheological properties and compressive strength. a) Yield stress, b) viscosity

The correlation between yield stress and compressive strength shows lower compressive strength for MF mortars when compared to LF mortars but in terms of yield stress both are in the same range, Figure 6.13a. Thus yield stress by itself does not affect compressive strength. The results however possibly highlight two different aspects linked to the superplasticizer content. For instance, MF mortars containing more super-plasticizer in their formulation have low compressive strength due to increased air content. However due the high mixer efficiency the differences between LF (less superplasticizer) and MF in terms of yield stress become barely detectable. Conversely, mixtures prepared using the small mixer (Eirich-5) are more sensible to

the differences in SP content regarding rheological behavior although the mechanical properties are not too much affected. Furthermore, it was observed that even high variations in the mortar rheological properties due to increased SP content only cause small mechanical properties changes when mixtures are prepared using the small mixer (Eirich-5).

6.4 CHAPTER CONCLUSIONS

This chapter discussed an intensive work of comparison of mixers, mixing procedures and matrix fluidity in terms of their rheological and mechanical properties. It was found that using a small mixer (Eirich-5) the SP/binder content strongly affects the mortar rheological behavior. In addition a large difference between low fluidity (LF) and moderate fluidity (MF) mortars was observed where the apparent yield stress for LF mortars is 2.25 times higher than for MF mortars. On the other hand the rheological behavior of mixtures prepared using a big mixer (Eirich-75) shows almost no difference between fluidity levels (LF and MF) as well as between the mixing speeds evaluated. This behavior indicates that mixer capacity has a great influence on materials dispersion since better dispersion can create more stable systems. In addition, from a practical point of view it is also possible to decrease the superplasticizer content by improving the mixer efficiency.

An analysis of all mixing procedures evaluated using the big mixer indicates that when fibers are added after the mortars prepared (ECC-Aft) the mechanical performance of composite improves independently of mixing speed or mortar fluidity. Furthermore, mixtures prepared using the big mixer (Eirich-75) show higher fracture toughness than mixtures prepared using the small mixer (Eirich-5). Also, when using the small mixer the high superplasticizer content the higher is the fracture toughness.

In general it seems that the high the mixer efficiency the more disperse are the particles meaning more free water in the mixture. If on one side this helps improve the material fluidity on the other side it can impact its mechanical properties leading to the conclusion that the mix design can be improved in terms of superplasticizer content reduction or even mixing time as long as a more robust mixer is used.

7

Effect of mix design and fiber content on SHCC properties

7.1 INTRODUCTION

Mix design and fiber content effect on the properties of strain-hardening cement-based composites are investigated in this chapter. Three different mix designs and two fiber contents were studied. Batches were prepared in the same mixer and following the same mixing procedure. In sequence fresh properties were evaluated in terms of consistency and rheology. The rheological behavior was measured using a rheometer equipped with a ball measuring system. In the meantime the composite strain-hardening behavior as well as its mechanical properties such as flexural behavior and compression strength were evaluated.

Finding a correlation between strain-hardening behavior and rheological properties was the main target of such analysis. Besides that the impact of matrix characteristics and fiber content on the composite fresh and hardened properties were investigated.

7.2 EXPERIMENTAL PROGRAM

7.2.1 Materials and mixture proportions

The constituent materials used to prepare the mixtures investigated in this study are ordinary Portland cement, fly ash and fine quartz sand (0.125 mm average size and maximum grain size of 0.250 mm) with density of 2.65 g/cm³, Table 7.1. Superplasticizer (SP) MasterGlenium 51 polycarboxylic ether (PCE) and viscosity agent (VA) MasterMatrix SCC 2, both from BASF, were added to the mixture in order to adjust its rheological properties. Furthermore, polyvinyl alcohol (PVA) fibers, 8 mm in length, 40 µm thick and with a density of 1300 kg/m³ were used. Fiber content variation by volume was 2.0% and 2.5% of the total mixture.

Table 7.1 – Mixture design proportions by weight.

Composition	Cement	Fly Ash	Sand	Water	Water/binder*	[SP]	Viscosity Agent [VA]	Fiber content (%)	
C1	1	1.20	0.80	0.56	0.255	0.017	-	2	2.5
C2	1	2.33	1.67	1.04	0.312	0.022	0.01		
C3	1	1.21	1.06	0.64	0.290	0.015	0.01		

*Binder = cement + fly ash.

The experimental setup consists in the evaluation of three different mixture designs based on previous works from other researchers. All those compositions are very well understood from a mechanical performance point of view. However some small adjustments have been made to adequate them to the current materials. As seen in Table 7.1 the proportions are different for each composition but the materials used are the same. Composition C1 is based on results from (LI, 2008) and it has been used as reference composition in the previous chapters while composition C2 was studied by (JUN; MECHTCHERINE, 2010) and composition C3 comes from (MECHTCHERINE et al., 2011).

7.2.2 Mixing

A laboratory pan Eirich-5 mixer with 5 L capacity as shown in Chapter 6 was used to prepare the mixtures. In all tests, the batch volume was kept constant and

equal to 3.8 L. Batch experiments were carried out according to Table 7.1 at a temperature of 20 ± 3 °C. The solid materials were loaded before starting the mixer. After mixing the solid constituents for 30 s to ensure materials dispersion and homogenization, water containing dissolved liquid admixture was progressively added during the next 10s. Then the solid materials and liquids were mixed together for 3 min. Last, fibers were added during 20 s and the total mixing time with fibers was 60 s.

7.2.3 Method

7.2.3.1 *Mini slump-flow*

Right after the mixing process finished the flowability was measured using a mini-slump flow test. The mini-slump cone had a top diameter of 70 mm, a bottom diameter of 100 mm and height of 60 mm with a volume of 0.344 L. The cone was then lifted to allow the mortar to flow without shocking. Once the flowing stopped, the mortar spread was recorded as the average of the diameters measured along two diagonals.

7.2.3.2 *Rheology*

The rheological behavior was measured using a rheometer equipped with a Ball Measuring System (BMS). The test program as well as the equations used to calculate the rheological parameters are shown in Chapter 4. The rheological measurements were performed 13 min after the first contact between water and solid materials.

7.2.3.3 *Casting and curing*

After the mixtures were prepared they were then placed horizontally into molds without applying vibration. The specimens were covered with plastic foils and stored in a climatic chamber for 24 h at a temperature of 23 °C. After that the specimens were demolded, sealed in plastic bags and stored at room temperature until 28 days. Then the mechanical tests were performed.

7.2.3.4 Bending tensile tests

The flexural properties and compressive strengths were measured 28 days after casting. A three-point bending configuration was used to determine the flexural strength (MOR) and fracture energy. A servo-controlled test machine *Walter + bai ag* model was used with a loading rate of 0.005 mm/s. For each composition a total of three specimens measuring 40 x 40 x 160 mm each were used to perform flexural tests. The test procedure is the same as shown in Chapter 6.

7.2.3.5 Uniaxial tension tests

The uniaxial tension test setup for this study is shown in Figure 7.1. This setup is based on the one reported in (JUN; MECHTCHERINE, 2010) comprising of 5 dumbbell shaped prisms with total length of 250 mm and cross sectional dimensions (measured at the narrow middle) of 24 mm by 40 mm, Figure 7.1a. The uniaxial tension test was conducted in a 10 KN load capacity *Wolpert* universal testing machine using a deformation controlled mode with a displacement rate of 0.005 mm/s. Non-rotatable boundary conditions were ensured by gluing steel supports at both ends, which are then bolted to the testing machine. The deformations were measured on a 100 mm gauge length in the middle of the specimen using a Linear Variable Differential Transformer (LVDT) fixed on the specimen, Figure 7.1b. For each SHCC composition four specimens were tested.

Figure 7.1 – Direct tensile tests. a) Dimensions of the dumbbell shaped specimen (values in mm) (JUN; MECHTCHERINE, 2010). b) Test setup.

An overview of the experimental program is shown in Figure 7.2.

Figure 7.2 – Experimental setup.

7.3 RESULTS AND DISCUSSION

The composites consistency was measured through mini slump-flow. Figure 7.3 shows the mini slump-flow values of each composition. A slump-flow decrease with an increase in fiber content is observed. C1 is the composition with lowest slump-flow probably because of the low water binder ratio in its formulation. Composite C3 also shows low slump-flow values when fiber content is at 2.5%.

Figure 7.3 – Mortar consistency in terms of mini slump-flow.

7.3.1 Rheological behavior

Flow curves (torque vs. time) highlighting the differences among the compositions are shown in Figure 7.4 while the influence of fiber content is given by Figure 7.5. Please note that there is a correlation between slump-flow values and flow curves since high slump-flow are related to low torque levels.

Figure 7.4 – Influence of mixing composition. a) 2% fiber content. b) 2.5% fiber content.

From Figure 7.5a one sees that fiber content does not significantly influence the shape of C1 flow curve besides decreasing its slump-flow. However, for compositions C2 and C3 an increase of torque level with fiber content is observed. Their flow curves agree with the Bingham model via linear regression and its parameters such as apparent yield stress and viscosity are seen in Table 7.2.

Figure 7.5 – Influence of fiber content. a) Composition C1, water/binder (W/B) equal to 0.25. b) C2 with W/B = 0.31. c) C3, W/B = 0.29.

From Table 7.2, which lists rheological parameters as apparent yield stress and viscosity, it is safe to assume that the composition presents different behavior with increased fiber content. Indeed, for all compositions the apparent viscosity increases with fiber content while the yield stress increases for C2 and C3 but not for C1 where a 26% decrease in yield stress is observed.

Table 7.2 – Rheological parameters yield stress and apparent viscosity calculated from flow curves torque vs. rpm.

Composition	2% fiber content			2.5% fiber content		
	Yeld stress (Nmm)	Apparent viscosity (Nmm/rpm)	R ²	Yeld stress (Nmm)	Apparent viscosity (Nmm/rpm)	R ²
C1	41.2	0.89	0.97	30.43	1.13	0.96
C2	10.23	0.43	0.97	26.39	0.59	0.87
C3	26.86	0.52	0.95	43.42	1.09	0.97

Figure 7.6 shows the rheological behavior in terms of shear stress vs. shear rate and viscosity vs. shear rate curves. Composite C1-2% shows high shear stress (Figure 7.6a) as well as high viscosity (Figure 7.6b) compared to C2-2% and C3-2%. However for fiber volume content of 2.5%, C3-2.5% shows values higher than C1-2% meaning it is a more cohesive material. Conversely, the viscosity curves are very close to each other for high shear rates.

Figure 7.6 – Shear stress on the left side and viscosity on the right for mortars reinforced with 2% fiber volume content a), b) and 2.5% fiber volume content c), d).

In summary, the rheological behavior analysis shows that the composites are rheologically different and that an increase in fiber content increases the composite viscosity and yield stress. In the next section, the influence of those rheological properties on the mechanical behavior is evaluated.

7.3.2 Mechanical Performance

7.3.2.1 Bending tensile tests

For the mechanical performance analysis the compressive strength Figure 7.7b is presented from the lowest to highest: C2 < C3 < C1. Mixture composition C2 shows the lowest compressive strength and besides that its compressive strength decreases by 6.5% when the fiber content increases. The same behavior is also observed during the flexural strength analysis, Figure 7.7a.

Figure 7.7 – Influence of mixture composition on a) flexural strength and, b) compressive strength.

Flexural behavior increases with fiber content except for composite C2 where a slight decrease was observed. Furthermore, composites with low compressive strength such as composite C2 also display low flexural strength. Regarding composite C3, where almost no difference in terms of compressive strength with fiber content variation was shown, a huge increase in the flexural strength with fiber content was measured. This behavior can be associated to the composite rheological properties since for C3-2.5% the apparent yield stress is almost 40% higher than C3-2% while the difference in viscosity is more than 50% as shown in Table 7.2. In other words, the rheological behavior of composite C3 shows that an increase in yield stress and viscosity improves flexural behavior without affecting compressive strength.

The composite mechanical properties are important to, for instance, predict strain-hardening behavior. As reported elsewhere, compressive strength is related to tensile strength. Moreover, strength determines if the composites will exhibit strain-hardening behavior and the length of the strain hardening branch (LIN et al., 2017).

7.3.2.2 Tensile behavior

Tensile stress-strain curves are seen in Figure 7.8. In order to achieve strain-hardening in a cementitious composite the load carrying capacity of the fibers must exceed the first-crack strength of the composite (PARRA-MONTESINOS; REINHARDT; NAAMAN, 2012). In addition to that, for the same load carrying

capacity of the fibers, it is more likely to achieve strain-hardening in a low strength matrix than in a high strength matrix, (LIN et al., 2017).

With 2% fiber reinforcement, composite C1-2% practically only exhibits single-cracking strain-softening behavior as shown in Figure 7.8a. This means that the fibers load carrying capacity is not sufficient to exceed the first crack strength. Due to their relative lower tensile strength (1600 MPa) and strong bond with the matrix, PVA fibers tend to rupture instead of pulling out during the composite tensile failure.

On the other hand, composite C3-2% which is slightly different from C1-2% in terms of mix design and compressive strength, exhibits strain-hardening branches under direct tension as shown in Figure 7.8a.

Finally, in comparison with high strength composites (C1-2% and C3-2%), composite C2-2%, which can be considered a normal strength composite, exhibits higher ultimate strain and shows higher number of cracks when submitted to a centric tensile stress Figure 7.8a.

Figure 7.8 – Tensile stress-strain curves. a) 2% fiber volume content, b) 2.5% fiber volume.

Regarding fiber content, an ultimate strain improvement was observed for composite C1-2.5% but not in composites C2-2.5% and C3-2.5%, Figure 7.8b and Figure 7.9. For composite C1 an increase in fiber content favorably improves its performance by increasing first crack strength as well as ultimate strain, Figure 7.9a. While an increase in first crack strength was also observed for composite C2 its ultimate strain decreased by almost 50% compared to C2-2%, Figure 7.9b. Finally, composite C3 showed a decrease in first crack strength and a slight decrease in ultimate strain Figure 7.9c.

Figure 7.9 – Influence of fiber content on tensile stress-strain curves. a) Composition C1, b) C2, c) C3.

On the whole, the results show that mixture design and fiber content have an important role on the composite strain-hardening properties. It can also be concluded that high strength composites behave differently with fiber content increase as shown for composites C1 and C3. While for composite C1 an increase in fiber content led to an improvement in strain-hardening behavior, for composite C3 it did not improve the results.

In addition, for strain-hardening cement-based composites reinforced by PVA fibers it has been demonstrated that a volume fraction of 2% can enhance tensile strain capacity by about 3-5% (MAALEJ; HASHIDA; LI, 1995; LI; LI, 2013) which is higher than what was found in this work. Despite that important conclusions can be derived from the current study. For instance, a fiber content increase above 2% can improve the strain-hardening behavior for high strength matrices (C1). Conversely, for C2 and C3 fiber content has no effect on the strain capacity.

7.3.3 Correlations between rheological and mechanical behavior

This section tries to establish correlations between rheological properties and mechanical behavior. First, the mechanical performance in terms of flexural and compressive strength is related to mini slump-flow values, Figure 7.10 since those two parameters decrease with composite consistency. Aside from composite C3, increased fiber content promotes a decrease in mini slump-flow but the composite mechanical properties remain practically unaltered.

The composite C3 consistency decreases with fiber content increase. This behavior has no effect on its compressive strength Figure 7.10b but has a big impact on the flexural strength Figure 7.10a.

Figure 7.10 – Correlation between a) flexural behavior and mini slump-flow, b) and between compressive strength and mini slump-flow.

The relation between rheological properties and tensile behavior is seen in Figure 7.11. For a 2% fiber volume content a good correlation was found between yield stress and ultimate strain Figure 7.11a as well as between viscosity and ultimate strain Figure 7.11b. The lower is the composite yield stress and viscosity the higher is its ultimate strain. However for 2.5% fiber volume content no relation was found using this experimental setup.

Figure 7.11 – Relation between rheological behavior and strain-hardening.

Furthermore, Figure 7.11 shows that for composite C3 the variation of ultimate strain during tension tests is minimal and equal to 3.6% besides the yield stress for C3-2.5% being 38% higher than the yield stress of C3-2%. At the same time, viscosity increases more than 50% with fiber content but also no significant change was observed in terms of ultimate strain. However when compared to C3-2% the composite C3-2.5% showed a decrease in first-crack strength.

7.4 CHAPTER CONCLUSIONS

This chapter discussed the correlation between rheological behavior and mechanical performance of different mixture designs using two fiber contents. The results show that the composites are rheologically different and that an increase in fiber content increases the composite viscosity and yield stress.

Furthermore, mixture design and fiber content have an important role on the composite strain-hardening properties. High strength composites behave differently with fiber increased content, as shown for composites C1 and C3. Also, the rheological properties decisively contribute to understand the composite mechanical behavior response. Finally, for 2% fiber volume content a good correlation between yield stress and ultimate strain as well as between viscosity and ultimate strain was found: lower values for yield stress and viscosity are linked to higher ultimate strain. On the other hand, for a 2.5% fiber volume content no relation was found using this experimental setup.

8

Conclusions

The measurement and analysis of the rheological behavior of Engineered Cementitious Composites (ECC) reinforced with polyvinyl alcohol (PVA) fibers was the main topic of this research followed closely by techniques to improve the composite mixing process as well as evaluating the impact of fresh properties on its mechanical performance. Together, those factors contributed to understand the composite rheological behavior and globally optimize its performance by studying the correlation among mixing, rheology and mechanical properties.

This research has covered three distinct and at the same time interconnected areas: mixing behavior and processing, rheology, and mechanical behavior. The main conclusions can be summarized as follows:

- Mixing process has influence on the rheological behavior of PVA reinforced mortar especially regarding the moment at which fibers are added;
- ECC mortars prepared with low superplasticizer content show a significant increase in shear stress and viscosity. This means, for instance, that more effort is required to cast the composite. Besides that, shear stress and viscosity increase with mixing time. Consequently, an increase in mixing time is not linked to better rheological properties or an improvement in fiber homogenization;
- The devices (Vane and Ball Measuring System) used to measure the composite rheological behavior show that torque increases linearly with rotational speed and curves well-fitting the Bingham model mainly for ball measuring system. Conversely, vane tests show high fluctuation of torque levels during the rheological measurements. Those torque

fluctuations caused by PVA fibers disturb the test results using Bingham model equations since a constant torque average at each rotational speed is necessary to reach a steady state;

- Despite of that, both devices are able to measure the rheological behavior of PVA-ECC mortars. However, ball measuring system shows more efficiency when evaluating systems reinforced by fibers since it measures torque values more precisely;
- Mixing intensity affects fiber homogenization and high mixing intensity promotes a more uniform dispersion of fibers. It is shown that in low fluidity mortars the standard deviation of the fiber homogenization test for low mixing intensity (2.5 m/s) is 53% higher than that for high mixing intensity (4.9 m/s). On the other hand, this difference decreases to 10% for moderate fluidity mortars;
- Fiber addition sequence plays an important role mainly for low mixing intensity and it is also associated to the mixer capacity. Once the mixing intensity is high (4.9 m/s), it does not matter if fibers are added after or before the mortar is prepared. But for low mixing intensity the fiber addition sequence is very important;
- The mixing procedure where fiber addition occurs after mortar mixture (ECC-Aft procedure) improves fibers homogenization and reduces mixing energy by around 8% compared to the other mixing processes studied;
- If the mixer has enough power to prepare mixtures at high speeds it does not matter when fibers are added: if in the beginning or after the mortar is prepared. However, for low speed mixers, the sequence of fiber addition has a big impact on fiber homogenization;
- Mixer capacity has also a big influence in terms of materials dispersion and better dispersion lead to more stable systems. In addition, from a practical point of view it is also possible to decrease the super-plasticizer content by increasing mixer efficiency;

8.1 PERSPECTIVES FOR FUTURE STUDIES

The results of this research point to future studies regarding the remaining gaps in the theoretical and experimental work that may lead to further research, such as:

- To research the effect of matrix optimization on the composite rheology using the concepts of particle packing density;
- To implement alternatives to improve the composite strain-hardening behavior;
- To compare strain-hardening results obtained via different probe geometries;
- To develop new methods to evaluate the segregation of plastic fibers;
- To build a model for dynamic segregation.

9

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Appendix A

1. Admixture saturation curve.

From this data the three fluidity levels LF, MF and HF evaluated in chapter 4 were chosen.

Figure A 1 – Spread flow diameter

2. Torque vs. time curves for ball measuring system.

Figure A 2 – Reference mortar without fibers.

Figure A 3 – PVA-ECC mortar. Mixing time with fibers is 60s.

Figure A 4 – Mixing time with fibers is 120 s.

Figure A 5 – Mixing time with fibers is 180 s.

3. Torque vs. time curves for vane test.

Figure A 6 – Reference mortar without fibers measured via vane test configuration.

Figure A 7 – PVA-ECC mortar. Mixing time with fibers is 60 s.

Figure A 8 – 120 s of mixing time with fibers

Figure A 9 – 180 s of mixing time with fibers.