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Effects of particle size optimization of quartz sand on rheology and ductility of engineered cementitious composites

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Abstract: In this study, the effect of particle size of quartz sand on the fresh and hardened properties of engineered cementitious composites (ECC) was investigated. For this purpose, three ECC mixtures that are identical except for the gradation of quartz sands used in their composition were designed. One of the mixtures includes a combination of quartz sands with amounts determined by the Andreasen and Andersen particle size optimization model while the remaining two have a finer and a coarser gradation. In the fresh state, mini slump, mini V-funnel and bleeding tests were applied, and rheological parameters were determined according to Bingham and modified Bingham models by using a rotational viscometer. In the hardened state, flexural strengths, mid-span deflections and numbers of microcracks formed under flexural loading were determined at 7 and 28 days. It was observed that the particle size optimization of the quartz sand can provide a balance between flow and bleeding characteristics of ECC mixtures. Although a reduction in flexural strength occurred at both ages in the optimized ECC mixture, the deflection capacity and the crack formation capacity under loading were significantly increased, reaching a deflection value of over 10 mm with at least 11 cracks formed during the test. As a result, it was revealed that particle size optimization can yield a mixture with the highest ductility without compromising the workability of ECC.

Keywords: Engineered cementitious composites, particle size, optimization, rheology, ductility.

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1. Introduction

Engineered cementitious composites (ECC), which is an advanced type of fiber-reinforced cementitious composites (FRCC), is especially recognized for their superior mechanical performance. Moreover, the design of reinforced ECC as structural members is also focused on in the literature (Sivasubramanian, Singh, and Rajagopalan 2016). ECC exhibits strain-hardening and multiple self-controlled micro-cracking (steady-state crack propagation) behavior under tensile and flexural loads which allow the material to possess a ductile behavior. The strain capacity of ECC, including polyvinyl alcohol (PVA) fibers at a volume fraction of 2%, is about 300-500 times higher than that of conventional concrete and fiber reinforced concrete (FRC) (Li, Wang, and Wu 2001). High ductility and micron-sized crack widths of ECC also lead to a superior durability performance under severe environmental conditions such as freezing-thawing and chloride exposure (Li and Li

2011; Özbay et al. 2013; Sahmaran, Li, and Li 2007). These enhanced features of ECC are provided by micromechanical-based design theory which lays down the basics of obtaining a strain hardening composite through multiple cracking and, in practice, requires a special type of PVA fiber to tailor the matrix-fiber interface to meet these fundamentals. Aside from the fiber characteristics, the efficiency, hence the dispersion of fibers directly affects the performance of the composite in terms of multiple self-controlled crack widths and strain-hardening behavior (Li and Li 2013). Nevertheless, the ingredients used in the ECC mix design were shown to have a significant effect on the microstructure and tensile properties of ECC (Tahmouresi et al. 2021).

The workability and rheology of cementitious composites are highly effective on the hardened properties due to their influence on mixing, pumping, casting, molding, and compaction (Rehman et al. 2017). Therefore, well understanding of the fresh state properties of cementitious composites is crucial to obtain high-quality composites. Typically, the consistency of cementitious composites is evaluated by a slump or slump flow test. However, it is not always possible to assess the workability of cementitious composites solely by slump characteristics since there are numerous types of cementitious composites in use that may have specific workability requirements which cannot be measured by these methods (González-Taboada et al. 2017). Therefore, it is necessary to have sufficient information on the rheological properties of a cementitious composite to get a better understanding of their behavior in the fresh state.

Rheology enables the comparison of the properties of different cement-based materials quantitatively, as it is possible to establish relationships by using the obtained shear stress and shear rate values. Although, there are several models used to evaluate the rheology of fluids like cementitious composites, the most widely used ones are the Bingham and modified Bingham models (Yahia and Khayat 2001). When it comes to FRCC, the significance of controlling rheological properties becomes more apparent due to the effect of fiber dispersion on the performance of the final product.

The rheology of a cement-based material can be affected by several internal and external factors such as water content, ambient temperature, and the existence of mineral and chemical admixtures. Nevertheless, aggregate properties have a significant effect on the fresh and hardened properties of cementitious composites. In the design of ECC generally, quartz sand with a specific size range is used. Although limitations set forth by the micromechanical design theory are considered in the selection of the maximum aggregate size, this range may differ among ECC mixtures due to the variability of locally available sand in the production. Although some studies concentrating on the effect of maximum aggregate size over the ECC properties have been published (Sahmaran et al. 2009; Sahmaran et al. 2013) there is almost no information about how the aggregate size distribution and packing density would influence the properties of ECC which form the rationale of this study.

The packing density is interrelated with the particle size distribution (PSD) of the aggregate and determines the volume of voids needed to be filled with cement paste. Kennedy (1940) stated that the amount of paste should be more than adequate to fill the spaces between the aggregate particles so that there would be excess paste enabling a thin film around the aggregate particles to lubricate the fresh concrete. Therefore, it can be concluded that the higher the packing density is, the more the volume of excess paste becomes for a constant paste volume, and this results in improved workability (Kwan and Li 2012). For fiber reinforced cementitious composites like ECC, optimal workability and rheology can facilitate achieving desired fiber dispersion (Li and Li 2013). In addition to enhanced fresh properties, high packing density may also enable the production of more durable, stronger, and economical cement-based materials according to their intended use. Therefore, many researchers have been focused on the application of particle packing methods (PPMs) on different concrete types.

The basic idea of PPMs is minimizing the void between the solid particles (generally aggregates) of the cementitious system. In other words, it is an optimization method that theoretically determines the optimal combination of different sized solid particles in hand to attain the densest possible granular skeleton. Although it has been a known fact that the gradation of ingredients can affect the properties of cementitious systems, the first particle size optimization model aiming for a denser combination of ingredients was suggested and formulated as in Equation 1 by Fuller and Thompson (1907):

$$P(d) = \left(\frac{d}{d_{\max}}\right)^{q} \tag{1}$$

where P(d) is the ratio of particles finer than “d”, d_{max} is the maximum particle size and q is the distribution modulus which is generally accepted as 0.5 for concrete aggregates. Later on, several particle size optimization models were developed such as the modified Andreasen and Andersen (A&A) model (Equation 2) proposed by Funk and Dinger (1994) which has been widely used to optimize particle size distribution of different concrete types including self-compacting concrete (SCC) (Brouwers and Radix 2005; Mueller, Wallevik, and Khayat 2014), ultra-high-performance concrete (UHPC) (Yu, Spiesz, and Brouwers 2015a) and high-performance fiber reinforced concrete (UHPFRC) (Hunger 2010). The mathematical expression of the model is given in Equation 2; where P(d) is the ratio of the particles smaller than d in total solid materials by volume, d_{min} is the minimum, d_{max} is the maximum particle size and q is the distribution modulus.

$$P(d) = \frac{d^q - d_{min}^q}{d_{max}^q - d_{min}^q} \tag{2}$$

It should be noted that unlike the model suggested by Fuller and Thompson, the modified A&A model requires a more careful selection of distribution modulus value (q). In the literature, different q values are used to design different types of concrete: while Hunger (2010) proposed that “q” value should be between 0.22 and 0.25 for SCC, Mueller et al. (2014) stated that 0.27 for “q” value is more suitable if the SCC production is to be made by using less powder content. In another study conducted by Wang et al. (2014), a distribution modulus in the range of 0.23-0.29 is proposed to be more suitable. Moreover, a value of 0.23 is encountered for HPFRCC design in the literature (Yu, Spiesz, and Brouwers 2014; Yu et al. 2015a; Yu, Spiesz, and Brouwers 2015b). Consequently, it can be stated that it is more realistic and efficient to determine a distribution modulus according to the particle size distribution of the solid particles included in the design process.

Although there are several studies regarding different types of concrete designed based on PPMs, the design of ECC by particle size optimization of aggregates is a novel subject. Essentially, ECC requires certain rules to be followed during the design process and is a very sensitive material to the alterations made in the ingredients. However, different researchers have managed to design and produce different ECC mixtures by altering the original design (Keskin 2012; Lepech et al. 2008; Qian et al. 2009; Sahmaran et al. 2009; Sahmaran, Yildirim, and Erdem 2013; Sahmaran and Li 2009; Wang and Li 2007). Nevertheless, the effects of PPMs on rheology and mechanical properties of ECC have not been investigated in the literature. Considering the possible benefits of PPMs, in this study, optimization work was conducted on the aggregate portion of a commonly used ECC mixture in the literature based on modified A&A model by using 3 different quartz sands.

2. Materials and methods

The experimental study is divided into three parts to assess the effect of particle size optimization on the rheology of ECC and ductile behavior of ECC. The first part is the preparation of ECC mixtures with two different particle size ranges, namely fine and coarse, of quartz sands and with particle size optimization of the aggregate portion of standard ECC mixture. The second part includes the determination of fresh properties of ECC matrix by using mini slump test, mini V-funnel test, mini V-funnel T_{5min} test, bleeding test and rheological properties with a rotational viscometer. The third part covers the determination of flexural behavior and micro cracking of ECC mixtures by using four-point bending test and visual inspection at 7 and 28 days. Finally, the effects of particle size optimization on the rheological and ductile behavior of ECC were evaluated.

2.1. *Materials*

CEM I 42.5 R ordinary portland cement (PC), Class F fly ash (FA) which meets the requirements of ASTM C-618 (ASTM C618-17a 2017) standard, quartz sands (QS) with three different particle size ranges (with maximum sizes of 45 µm, 200 µm and 300 µm), polycarboxylate ether-based high range water reducing admixture (HRWRA) and polyvinyl alcohol (PVA) fiber were used to produce ECC mixtures. To prevent the increase in fracture toughness, the maximum aggregate size was restricted to 300 µm in accordance with micromechanics-based material theory (Sahmaran et al. 2013). Particle size distributions of quartz sands and binders are given in Figure 1.

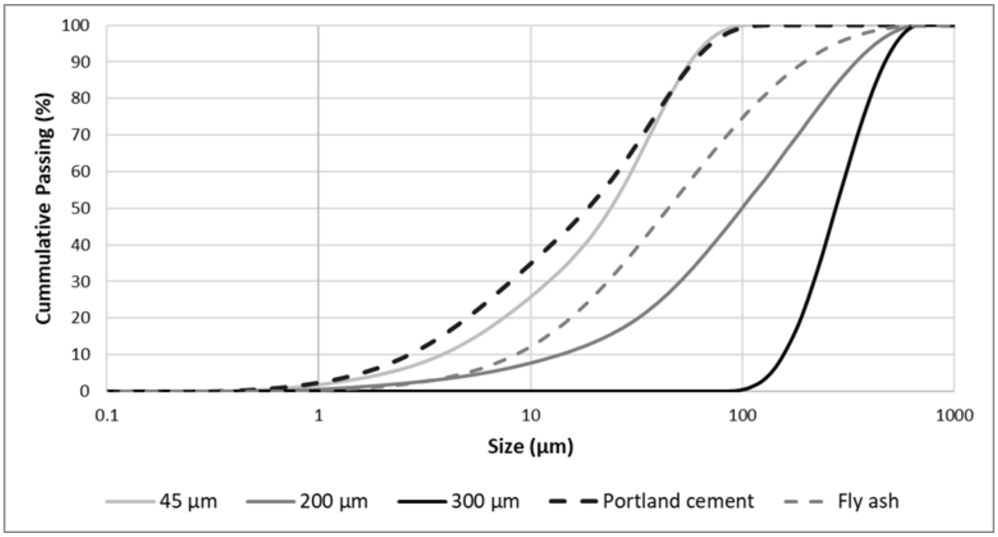


Figure 1. Particle size distribution of quartz sands and binders.

The length and diameter of the PVA fibers used in the production of ECC are 8 mm and 39 µm, respectively. The nominal tensile strength and elongation capacity of PVA fibers are also 1600 MPa and 6%, respectively. Besides, it should be noted that each ECC mixture was produced with three different HRWRA dosages (0.7%, 1.0% and 1.3% of the total binder by mass) to observe rheological behavior at different HRWRA dosages. Maximum HRWRA dosage was determined in such a way that no visual segregation would occur. The chemical composition and physical properties of PC, FA and QS are given in Table 1.

Table 1. Chemical composition and physical properties of PC, FA and QS.

Chemical Analysis	PC (%)	FA (%)	QS (%)
SiO_2	18.69	50.04	99.370
Al_2O_3	4.74	22.85	0.160
Fe_2O_3	3.37	8.02	0.027
CaO	61.87	11.21	0.140
MgO	3.36	2.23	-
Na_2O	0.19	0.27	0.030
K_2O	0.63	2.5	0.030
SO_3	2.93	0.78	-
Physical properties			
Specific gravity	3.15	2.28	2.60
Blaine fineness (cm^2/g)	3420	2850	-

2.2. *Specimen preparation and mixing procedure*

Since the rheology and workability of cementitious composites may be affected by ambient conditions, mixing procedures and mixer type (Yang et al. 2009), the same conditions were provided for all mixtures during the production process. A planetary mixer with a 20-liter mixing capacity was used to produce ECC mixtures. The casting process was conducted at 23±2°C and 50 ± 5% RH. First, all dry materials (PC, FA and QS) are added into the mixer and mixed for 1.5 minutes, then the mixing water is introduced and all materials are mixed for 1 minute. HRWRA is added into the mixture together and all materials are mixed for another 1 minute. Finally, PVA fiber is added to the mixture and the mixing process is resumed for additional 1.5 minutes as shown in Figure 2.

Figure 2. Production of ECC mixtures.

Throughout the determination of workability and rheological properties, ECC matrices without PVA fiber addition were used. It should be noted that only mini slump test was conducted on both with and without PVA fiber addition. To perform four-point bending test, prismatic specimens with dimensions of 22x75x360 mm were used. The test specimens were kept in the molds for the following 24 hours after casting. Then, they were demolded and moisture-cured in plastic bags at 23±2°C and 95 ± 5% RH until the testing ages. The flexural tests were conducted at the ages of 7 and 28 days.

2.3. *Testing methods*

Cementitious composites are generally described as non-Newtonian fluids (Li and Li 2013). In the case of ECC, adequate plastic viscosity is needed to maintain the stability of the matrix and provide uniform fiber dispersion resulting in more efficient fiber-bridging of PVA fiber (Sahmaran et al. 2013). To describe rheology, numerous rheological models like Bingham, modified Bingham, Herschel and Buckley, and Casson models were used in the literature (Yahia and Khayat 2001) while the Bingham model which predicts linear equation stands apart from the others as being the most widely used one (Li and Li 2013). However, fitting a line to the non-linear portion of the stress-shear rate curve at low shear rates is not possible by using the Bingham model. Furthermore, shear stress cannot be predicted precisely for fluids exhibiting shear-thickening behavior by using the Bingham model. To prevent these shortcomings of the Bingham model, the modified Bingham model was proposed which is the extension of the Bingham model by second-order term. The mathematical expression of the Bingham model and modified Bingham model are given in Equation 3 and Equation 4, respectively (Yahia and Khayat 2001).

$$\tau = \tau_0 + \mu\dot{\gamma} \tag{3}$$

$$\tau = \tau_0 + \mu\dot{\gamma} + c\dot{\gamma}^2 \tag{4}$$

In Equations 3 and 4, τ is the shear stress (Pa), τ_0 is the yield shear stress (Pa), μ is the plastic viscosity (Pa.s), , $\dot{\gamma}$ is the shear rate (s^{-1}) and c is the regression constant.

In the scope of this study, both models were used to decide whether the model represents the behavior in a more accurate way. A rotational viscometer was used for the determination of the rheological parameters. Measurements were recorded for eight different rotational speeds corresponding to shear rates between 20.40 s^{-1} to 0.102 s^{-1}. The tests were performed at 23 ± 2 °C and 50 ± 5% RH and the temperature of mixing water was kept constant as the rheological properties of cementitious materials are highly influenced by these parameters (Yang et al. 2009).

Although rheological parameters can be determined by using a rotational viscometer, the data should be correlated to evaluate realistic conditions by workability tests. In the literature, it is already stated that mini slump test can give a view on the yield stress of composite (Yang et al. 2009). Since stability is crucial for uniform PVA fiber dispersion, mini V-funnel, mini V-funnel T_{5min} and bleeding tests were also conducted to evaluate how segregation resistance and stability of matrix are affected by different rheological parameters.

To determine the workability properties of ECC matrix, mini slump, mini V-funnel and bleeding tests were performed. Mini slump values were determined by a mini slump cone with a height of 60 mm, a bottom diameter of 100 mm and a top diameter of 70 mm. For the mini slump test, two diameter measurements were taken in mutually perpendicular directions and the average of measurements was recorded as mini slump value. Mini V-funnel test is a commonly used method to measure the flow time of mortar type cementitious composites. Mini V-funnel T_{5min} test is also employed to have an understanding of the segregation resistance of the composites produced. For both tests, a funnel which is a smaller version of the standard V-funnel described in (EFNARC 2002) guidelines is used. After the funnel is filled with the ECC matrix, the nozzle is opened to allow the flow of the matrix. For the mini V-funnel T_{5min} test, mixtures were kept in the funnel for 5 minutes in addition to the mini V-funnel test. Then, flow duration is measured by using a stopwatch and recorded.

A bleeding test was also performed in accordance with ASTM C 232 standard (ASTM C232-04 2004). For the bleeding test, a cylindrical steel container with a diameter of 255 mm and a height of 280 mm that meets the requirements of the standard was filled with the mixture, and the container was covered with a lid. The bleeding water was drawn off by a pipette at pre-determined time intervals specified in the standard until the bleeding stopped. To prevent evaporation of bleeding water, the lid of the container was only opened while drawing off the bleeding water. It should be noted that the bleeding test was conducted for a single HRWRA dosage (1%) as in the determination of flexural properties of the mixtures.

Flexural properties including the flexural strength, deflection capacity and micro-crack distribution under loading were determined by using an electromechanical universal test machine under four-point bending setup with a displacement-controlled loading at a rate of 0.005 mm/s. For the four-point bending test, three prismatic specimens were prepared to test at the ages of 7 and 28 days. In order to evaluate the micro-cracking behavior, the images of specimens were captured to observe the number and distribution of cracks at the failure point.

2.4. *Particle size optimization*

Particle size optimization is a method that aims to obtain the highest packing density by filling up the voids between large particles with suitable sizes and proportions of smaller particles (Kumar and Santhanam 2003). In this study, the modified Andreasen and Andersen model which is proposed by Funk and Dinger (Funk and Dinger 1994) was employed to re-design the aggregate portion of an ECC mixture.

In the literature, different cementitious composites have been designed for different purposes by using different q values as mentioned previously. Accordingly, different q values were recommended for the materials with different particle size characteristics. After constructing a target aggregate distribution using the modified Andreasen and Andersen model, the volume fractions of the aggregates available are adjusted so that the actual aggregate distribution fits the model as much as possible. Then, the success of the fit is evaluated by calculating the residual sum of squares (RSS). Herein, the minimum possible RSS value indicates the maximum fit to the target curve and hence the highest possible packing density provided that an adequate distribution modulus “q” is selected for the model.

It should be noted that two conditions should be satisfied to attain the highest actual packing density: the q value should be suitable for the size range of aggregates in hand and the lowest RSS value between the model and the aggregate mixture should be attained by mixing these aggregates. In this study, instead of a fixed q value, different q values between 0 and 0.6 were tried to obtain the minimum possible RSS value. In other words, the minimum RSS value for each q value between 0 and 0.6 was calculated and the q value corresponding to the minimum possible RSS value was selected to constitute the model. For the optimization process, quartz sands with three different particle size ranges (maximum sizes of 45 µm, 200 µm and 300 µm) were used. After the optimization process, the model and the mixture converged at a q value of 0.53 which corresponds to the minimum possible RSS value of 691.9. Corresponding volumetric proportions to 45 µm, 200 µm and 300 µm maximum sized quartz sands were determined as 27.2%, 11.7% and 61.1%, respectively. Particle size gradations of the target curve and the combination of quartz sands are given in Figure 3.

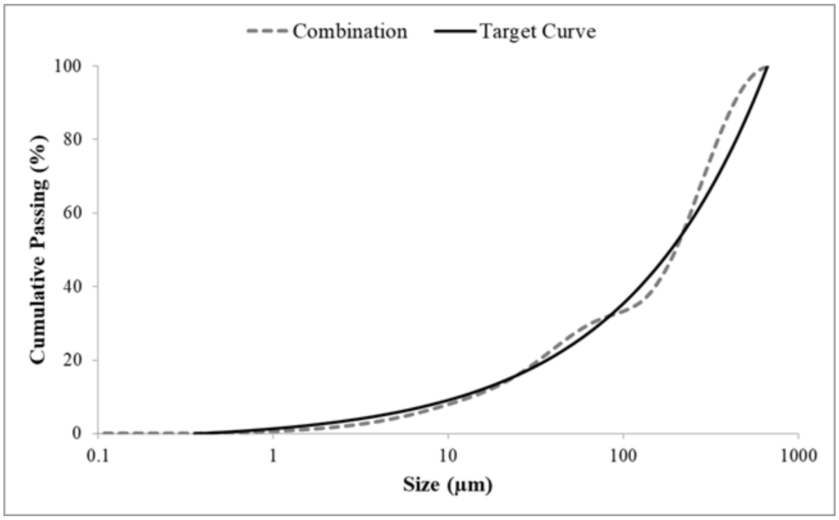


Figure 3. Particle size distributions of the target curve and combination of quartz sands

2.5. *Mixture proportions*

To evaluate the effects of particle size optimization on the rheological properties and ductility of ECC, three different mixtures, two of which with fine and coarse quartz to represent the limiting sizes of aggregates used in standard ECC mixture, were prepared. The third mixture, on the other hand, was prepared by using a combination of quartz sands with 3 different gradations in which the relative quantities of the sands were determined by optimization of the aggregate portion of the standard ECC mixture according to modified Andreasen and Andersen particle size model. Three different HRWRA dosages which are 0.7%, 1.0% and 1.3% of the total binder by mass were used for each mixture. Nomenclatures and proportions of the mixtures are given in Table 2 where FM, CM and OM represent the mixtures with fine, coarse and optimized quartz sand, respectively.

Table 2. Proportions of mixtures

Mixture ID	Ingredients (kg/m^3)						
	Cement	Fly Ash	Water	0-45	0-200	100-300	PVA
FM	560	672	333	442	-	-	26
CM	560	672	333	-	-	442	26
OM	560	672	333	120	52	270	26

3. Experimental results and analysis

The test results of three different ECC mixtures with different aggregate gradings are presented and discussed in the following three sections as workability properties (mini slump, mini V-funnel, V-funnel T_{5min} and bleeding tests), rheological parameters (assessment of regression coefficient, plastic viscosity and yield stress of mixtures), and flexural properties (four-point bending test and visual inspection).

3.1. *Workability properties*

As given in Table 3 the maximum HRWRA dosage was determined as 1.3% beyond which visual bleeding was observed for all mixtures. This phenomenon is also called as saturation point (Sahmaran et al. 2013) and 1.3% can be said to be the saturation point of the HRWRA for the mixtures considered in the scope of the study. As expected, a gradual increase of HRWRA dosage caused a rise in mini slump flow diameter of all mixtures. Regardless of the HRWRA amount, OM had the highest mini slump flow diameters while FM exhibited the least mini slump flow diameters. Although the most effective parameter on mini slump flow diameters of mixtures seems to be HRWRA dosage, it can be also stated that particle size optimization of aggregate portion has an additional beneficial effect on mini slump test results. As the same amount of paste was used in all ECC mixtures, the part of paste that was not used to surround and fill the gaps between aggregate particles constitutes the excess paste. This positive effect of optimization is attributed to the increase in the excess paste amount in the composite, as lower amount of paste is required to fill the gap between aggregate particles due to compact packing achieved by optimization. On the other hand, the use of quartz sand with a maximum particle size of 45 µm alone resulted in a relatively limited mini slump flow diameter. This may be a consequence of higher water demand of the FM due to the relatively higher specific surface area of used quartz sand in the mixture.

Table 3. Mini slump test results.

Mixture ID	Mini slump flow diameter (cm)		
	HRWRA dosage (%)		
	0.7	1.0	1.3
FM	36.0	39.0	41.0
CM	37.0	41.5	43.0
OM	37.0	42.5	43.5

To evaluate the segregation resistance of ECC mortars, mini V-funnel test was also employed. For this purpose, after performing the mini V-funnel test according to EFNARC guidelines (EFNARC 2002) the funnel was refilled with mortar and flow time was measured 5 minutes after filling the funnel, similar to the conventionally used V-funnel T_{5min} test. In other words, V-funnel T_{5min} test described in the guidelines was conducted in the same manner by using the mini V-funnel instead. The test results of V-funnel and V-funnel T_{5min} along with the differences between the flow durations are given in Table 4 which also includes the bleeding test results for HRWRA dosage of 1%.

As seen in Table 4, the highest flow durations for both V-funnel and V-funnel T_{5min} were obtained from the FM mixture. Even though the V-funnel test and the slump flow test produced parallel results, V-funnel test's conclusions are more obvious. Although a substantial decrease in flow durations and differences was observed with increasing HRWRA dosage, it is clear that the use of quartz sand with finer quartz resulted in limited flow characteristics in comparison with the use of coarser and combined quartz sands. The shortest flow time for all HRWRA dosages was obtained by the use of coarse quartz sand. For all HRWRA dosages, flow durations and differences between V-funnel and V-funnel T_{5min} tests were in the recommended range for conventional V-funnel tests according to EFNARC guidelines. However, the lowest difference between V-funnel and V-funnel T_{5min} tests which is an indicator for the highest segregation resistance was achieved by the OM mixture.

Table 4. Mini V-funnel and mini V-funnel T_{5min} (in seconds) and bleeding test results.

Mixture	HRWRA dosage (%)									Bleeding (%)
	0.7			1.0			1.3			
	V-funnel	T_{5min}	Diff.	V-funnel	T_{5min}	Diff.	V-funnel	T_{5min}	Diff.	
FM	47.0	64.3	17.3	29.2	36.9	7.7	16.0	21.2	5.2	0.71
CM	5.5	9.3	3.8	5.4	8.3	2.9	4.9	7.6	2.7	9.15
OM	9.8	12.0	2.2	7.6	10.5	2.9	7.0	9.4	2.4	4.51

The bleeding tendency of mixtures was evaluated following method A described in ASTM C 232 standard and the results are given in Table 4. This test was conducted for an HRWRA dosage of 1% which is also the dosage used in the mixtures tested for hardened behavior. Contrary to the V-funnel test, the lowest bleeding ratio was observed in the mixture FM while the CM mixture exhibited the highest bleeding tendency. Besides, the bleeding ratio of the OM mixture remained between FM and CM mixtures. Since the amount and fineness of binder materials, w/c ratio, HRWRA content, and fiber ratio are all identical in the three mixtures, bleeding is majorly affected by the fineness and gradation of the aggregate portion. The bleeding tendency increases when the mixture gets coarser while optimized aggregates yielded in-between results. As the specific surface area of the sand is increased, more water is required for wetting the surfaces, and hence more water is held by the aggregates and thus less bleeding is observed. Although the mixtures containing single sized quartz sands performed sufficiently in terms of either bleeding tendency or flowability, neither of them showed a stabilized performance in all respects when fresh tests are evaluated together. However, the optimized mixture exhibited high flowability without any potential risk of bleeding or segregation.

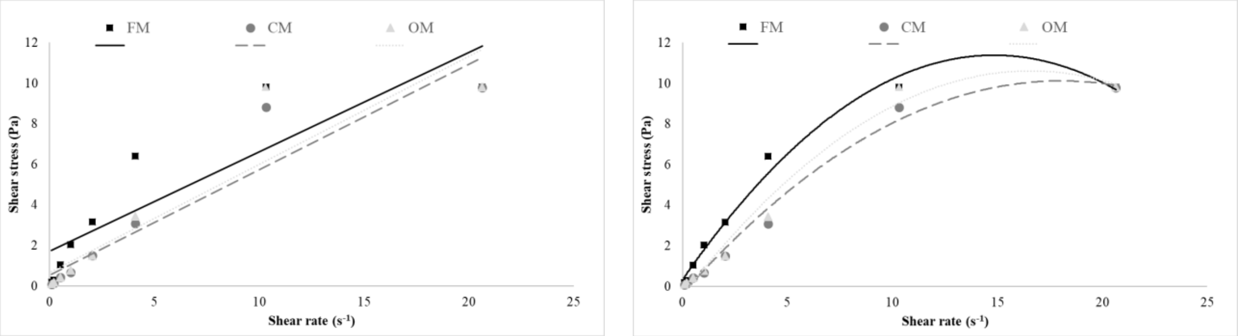
3.2. *Rheological properties*

Bingham and modified Bingham models for cementitious composites were developed for the ECC matrices and the success of the models is defined by regression coefficient (R^2). In the scope of this study, both the Bingham and the modified Bingham models were constituted to determine the model providing the best correlation for each mixture and each HRWRA dosage. Regression coefficients for each model are given in Table 5.

Table 5. Regression coefficients for Bingham and the modified Bingham models.

Mixture ID	HRWRA Dosage (%)	R^2 values	
		Bingham model	Modified Bingham model
FM	0.7	0.571	0.863
CM		0.850	0.985
OM		0.837	0.994
FM	1.0	0.769	0.989
CM		0.894	0.988
OM		0.851	0.982
FM	1.3	0.798	0.997
CM		0.931	0.996
OM		0.867	0.980

Regression coefficients calculated for the modified Bingham model are significantly higher than those calculated for the Bingham model for any individual mixture type and HRWRA dosage. For both Bingham and modified Bingham models, flow curves at the HRWRA dosage of 1% are given in Figure 4. It is apparent that the modified Bingham model provides a more accurate fit, especially in the low shear rate region of the flow curve. This situation was also stated in the literature (Yahia and Khayat 2001). Consequently, among the two models studied, the more proper model was determined as the modified Bingham model to constitute the flow curve.



(a) (b)

Figure 4. Flow curves for (a) Bingham and (b) modified Bingham models.

Rheological parameters, regression equation and regression coefficient of mixtures obtained by the modified Bingham model are given in Table 6, for each mixture and HRWRA content. Yield stress which is the threshold stress level for initiation of plastic deformation can be explained in analogy with the slump flow test. After the removal of the slump cone, flow initiates due to the self-weight of the matrix, and then, the flow continues until the weight of the matrix produces shear stress lower than the yield stress (Yang et al. 2009). Accordingly, the lower yield stress of composites results in higher slump flow values (Sahmaran et al. 2013). As seen in Table 6, negative values of yield stress (encountered in CM and OM mixtures) were assumed as zero (Sahmaran et al. 2013). The matrices like the ECC matrix possess much less yield stress than plain concrete as a result of very fine materials and a high amount of HRWRA in their composition (Feys, Verhoeven, and De Schutter 2008). It should also be noted that the ECC matrix which does not contain PVA fibers, is used to determine the rheological parameters without any reduction in HRWRA dosage which can lead to very low or even zero yield stress values. As expected, a gradual increase of HRWRA dosage in the FM mixture caused a reduction in the yield stress level. Besides, considering workability test results (mini slump and mini V-funnel) and yield stress values it can be inferred that the use of quartz sand with finer particle size causes a significant increase in the yield stress level of ECC.

Greater HRWRA dosage resulted in reduced plastic viscosity, for the same aggregate gradation indicating a mixture less resistant to flow. Adjustment of plastic viscosity is vital for the stability of the matrix and uniform fiber dispersion (Sahmaran et al. 2013). That is, a sufficient level of plastic viscosity is necessary to maintain the stability of the matrix while preserving flow ability for uniform fiber dispersion. Thus, plastic viscosity and workability properties (mini slump, mini V-funnel and bleeding tests) should be evaluated simultaneously. As can be understood from mini slump and mini V-funnel test results, the use of quartz sand with a maximum particle size of 45 µm led to an increase in plastic viscosity for each HRWRA dosage while the lowest plastic viscosity values were obtained by use of quartz sand with the maximum particle size of 300 µm.

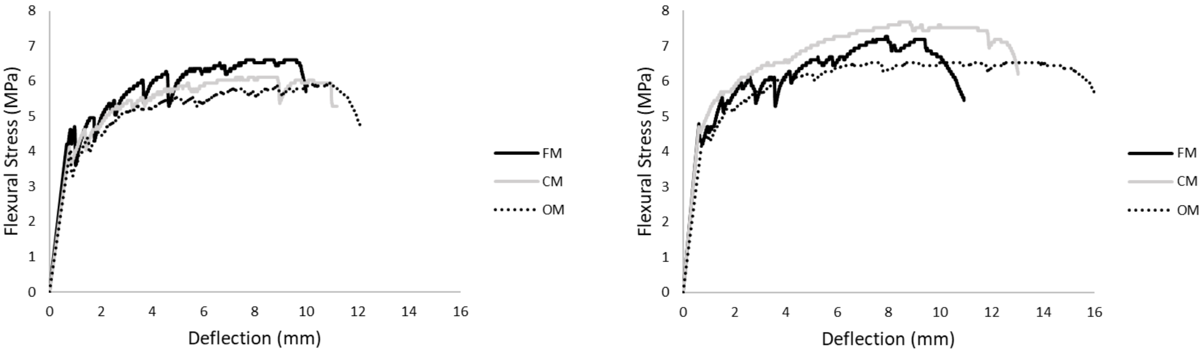
Besides, plastic viscosity values of OM mixture remained between FM and CM mixtures as in the workability test results. Herein, although CM mixture seems to provide the best flowability properties, it is an incontrovertible fact that the coarser sand causes a significant amount of increase in the bleeding rate, resulting in insufficient fiber dispersion in the matrix. Also, low slump flow values, high plastic viscosity and flow duration of FM mixture is a sign of inadequate flow characteristics despite having the lowest bleeding ratio. Accordingly, optimum rheological properties were achieved by particle size optimization of the aggregate portion in the ECC matrix. Furthermore, enhanced deflection capacity and micro-cracking of OM mixture which can be attributed to uniform PVA fiber distribution as a result of optimum rheological properties support this outcome. Moreover, it should also be stated that although viscosity can be correlated to both, it is in line with the results of the mini V-funnel test more than mini slump flow.

Table 6. Rheological parameters of ECC mixtures.

Mixture ID	HRWRA Dosage (%)	Yield stress (Pa)	Plastic viscosity (Pa.s)	Regression equation	R^2
FM	0.7	1.82	1.53	$\tau = 1.82 + 1.53\dot{\gamma} - 0.056\dot{\gamma}^2$	0.863
CM		0	1.32	$\tau = -0.30 + 1.32\dot{\gamma} - 0.040\dot{\gamma}^2$	0.985
OM		0	1.39	$\tau = -0.25 + 1.39\dot{\gamma} - 0.043\dot{\gamma}^2$	0.994
FM	1	0.33	1.49	$\tau = 0.33 + 1.49\dot{\gamma} - 0.050\dot{\gamma}^2$	0.989
CM		0	1.16	$\tau = -0.35 + 1.16\dot{\gamma} - 0.032\dot{\gamma}^2$	0.988
OM		0	1.33	$\tau = -0.44 + 1.33\dot{\gamma} - 0.040\dot{\gamma}^2$	0.982
FM	1.3	0.29	1.43	$\tau = 0.29 + 1.43\dot{\gamma} - 0.047\dot{\gamma}^2$	0.997
CM		0	1.01	$\tau = -0.24 + 1.01\dot{\gamma} - 0.020\dot{\gamma}^2$	0.996
OM		0	1.26	$\tau = -0.50 + 1.26\dot{\gamma} - 0.030\dot{\gamma}^2$	0.980

3.3. *Flexural performance*

In the literature, studies are revealing the effect of rheology on the fiber dispersion of cementitious composites (Fischer, Wang, and Li 2003; Li and Li 2013; Ozyurt, Mason, and Shah 2007). In the case of ECC, achieving well distributed fibers through the material is essential since the characteristics of ECC largely depend on post-cracking behavior. In cases where the fibers are not effectively dispersed in the composite, weak regions may prevent the fulfillment of the energy criterion (Li et al. 2001), hence may lead to low strain capacities accompanied by potential strain-softening behavior. Therefore, the mixtures used in the study were also evaluated under four-point bending test. The flexural stress-deflection diagrams obtained at 7 and 28 days for each mixture are given in Figure 5. Besides, the average values of flexural strength, mid-span deflection, and crack numbers are also presented in Table 7. It can be seen that the beneficial effect of aggregate optimization on the fresh properties is also reflected positively on mid-span deflection values. As expected, mid-span deflections decreased at 28 days as the hydration proceeds and the mixtures gain strength. Even though flexural strength of the optimized mixture is slightly lower than other mixtures, it has the highest deflection capacity among all mixtures at both ages. For ECC, the key parameter to attain desirable ductile behavior is formation of multiple self-controlled crack widths. Thus, the number and distribution of the cracks form on the specimens is a good indicator to assess the success of aggregate optimization in terms of ductile behavior. Nevertheless, tight cracking is also an indicator of a better durability. Number of cracks on OM specimens is also higher than that of the mixtures including single aggregate fraction. Moreover, the cracks appear to be well distributed through the composite compared to beam specimens belonging to other mixtures as seen from the close-up views of the crack zones of the specimens provided in Figure 6.



(a) (b)

Figure 5. Stress-deflection diagram of mixtures at the age of (a) 7 days and (b) 28 days.

Table 7. Four-point bending test results and micro-cracking evaluation of mixtures.

Mixture	Flexural strength (MPa)		Mid-span deflection (mm)		Average crack number	
	7-days	28-days	7-days	28-days	7-days	28-days
FM	6.6	7.3	8.61	7.93	9	7
CM	6.1	7.7	8.81	8.52	10	8
OM	6.0	6.5	10.92	10.47	12	11

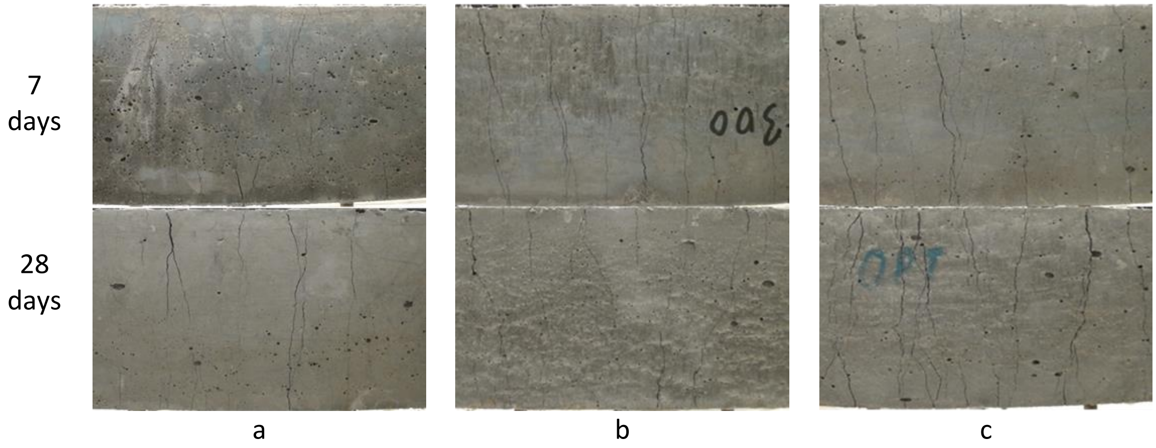


Figure 6. Pictorial view of flexural beams at failure: micro-cracking of (a) FM; (b) CM; (c) OM mixtures at the ages of 7 and 28 days

4. Conclusions and comments

In the scope of this study, the effects of the particle size optimization of ECC aggregates and the use of quartz sands with different particle size range on the workability, rheology and ductile behavior of ECC were examined. Besides, the connections between the workability, rheological parameters, four-point bending tests and visual inspection were established to evaluate how the ductile behavior of ECC is affected by its fresh properties. The following conclusions based on the test results can be drawn:

1. The workability and rheology are highly affected by the gradation of aggregate. While the use of a coarser particle size range has a beneficial effect on flow properties, bleeding problems may arise. On the other hand, while the use of a finer particle size range of aggregate significantly reduces the bleeding, it adversely affects the flow properties. Particle size optimization of the aggregate portion is very useful to balance the workability related properties of ECC;
2. Yield stress and plastic viscosity increase as aggregate particle size gets finer. In other words, a finer particle size range of aggregate causes higher resistance to flow. Although production of ECC with a coarser particle size range of aggregate provides a significant amount of decrement in plastic viscosity, particle size optimization of aggregates can help maintain superior ductility while achieving the preferable rheological parameters;
3. The beneficial effects of particle size optimization can be attributed to the lubrication effect resulting from the decrease in the required volume of paste to cover aggregates. Namely, more paste can be available to lubricate the matrix due to particle size optimization;
4. It is also revealed that enhanced ductile and micro-cracking behaviors can be achieved as a consequence of the valuable effects of particle size optimization on workability and rheology of ECC that might result in a more uniform PVA distribution.

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