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Rheological Control in Production of Engineered Cementitious Composites

by En-Hua Yang, Mustafa Sahmaran, Yingzi Yang, and Victor C. Li

*This paper reports on a study of rheological control of fresh properties during processing of engineered cementitious composites (ECC) for the purpose of more effectively realizing mechanical properties optimized through micromechanical design theory. Four factors (Class C fly ash [FA] to Class F FA ratio, water-binder ratio [*w/b*], amount of high-range water reducer [HRWR], and amount of viscosity-modifying admixture) were investigated to determine their effects on the fresh and hardened properties of ECC. Test results indicated that among the investigated factors, the* w/b *most strongly affects the plastic viscosity of ECC mortar (without fiber), which in turn have a significant impact on the ECC composite ultimate tensile strength and tensile strain capacity. Marsh cone flow test and mini-slump flow test were demonstrated as simple and practical methods to characterize the rheological properties of ECC mortar. By complying with the recommendations of rheological control for producing ECC summarized in this paper, it is expected that self-consolidating ECC with optimum rheological properties that promote uniform fiber distribution throughout the matrix can be easily produced, and optimized tensile properties can be realized for micromechanics-based optimized ECC mixture design.*

Keywords: cementitious; fiber-reinforced concrete; process; rheology; tensile properties.

INTRODUCTION

Engineered cementitious composite (ECC) is a unique type of high-performance fiber-reinforced cementitious composite designed micromechanically for high ductility and damage tolerance under mechanical loading, including tensile and shear loadings with moderate fiber volume fraction, typically 2% by volume.[1-4] Unlike ordinary concrete materials, ECC strain-hardens after first cracking, similar to a ductile metal, and demonstrates a tensile strain capacity (tensile strain at peak load in a uniaxial tension test) of 3 to 5%, approximately 300 to 500 times that of normal and fiber-reinforced concrete (FRC). Even at large imposed deformation of several percent, crack widths of ECC remain small, less than 80 μm (0.003 in.). To accommodate large deformations, rather than forming a single crack that widens with increasing load as is typical in concrete or tension-softening FRC, ECC forms numerous microcracks that allow the material to undergo large tensile inelastic straining. The tight crack width of ECC is important to the durability of ECC structures as the tensile ductility is to the structural safety at ultimate limit state. These unique properties, together with a relative ease of production including self-consolidation casting[5,6] and shotcreting,[7] make ECC attractive to various civil engineering applications. ECC is currently emerging in full-scale structural applications, including bridges and buildings, as well as in infrastructure repair work in Japan and the U.S.[8,9]

In the micromechanics-based design theory of ECC, it is assumed that perfect fiber dispersion is attained in the composite. In practice, nonuniform distribution of fibers tends to degrade as well as introduce undesirable variability into the mechanical properties of this material. As a result, high variability in tensile properties is sometimes observed. This aim of this paper is to improve the robustness of the mechanical properties of ECC through rheological control of fresh properties during processing. Focus is placed on understanding the factors governing rheological parameters and the correlation between fresh and hardened properties. It is expected that this knowledge, along with micromechanics-based material design theory, will help maximize the performance of fresh and hardened ECC. Experimental design (ED) and statistical analysis (Taguchi method)[10] were used as tools to identify the correlation between governing factors and rheological and hardened properties of ECC. Specifically, the four factors are Class C fly ash (FA) to Class F FA ratio (*C*/*F*), water-binder ratio (*w*/*b*), the ratio of amount of HRWR to binder content (*HRWR*/*B*), and the ratio of amount of viscosity-modifying admixture to binder content (*VMA*/*B*).

RESEARCH SIGNIFICANCE

Although the mixture proportions of ECC have been well documented, only a limited number of laboratories and experienced researchers have consistently reproduced high ductility ECC. This is mainly due to two reasons. First, ingredients with inappropriate characteristics (type, size, and amount) as defined by micromechanical principles may negatively influence the microstructure of the composite and, therefore, the tensile ductility of ECC. The second reason may be that ingredients from different sources and/or processing procedures lead to a change in rheological properties of fresh ECC that results in poor fiber-dispersion characteristics and degrades the hardened properties. This research provides a statistically significant link between ECC rheological properties and hardened properties. The findings in this paper provide a rational foundation for the application of rheological control of the fresh properties of ECC matrix (mortar) as an effective tool to practically realize optimal hardened tensile properties predicted by micromechanics. Results from this study should assist the broader adoption of this relatively new material with more reproducible and robust properties.

MATERIAL PARAMETERS GOVERNING FRESH PROPERTIES OF ECC

The ingredients and mixture proportions of ECC are optimized through micromechanics-based material design theory to satisfy strength and energy criteria for attaining

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ACI member **En-Hua Yang** *is an Engineer at Exponent®, Inc. He received his MSE and PhD from the University of Michigan, Ann Arbor, MI. His research interests include the development and characterization of high-performance fiber-reinforced cementitious composites.*

ACI member **Mustafa Sahmaran** *is an Assistant Professor in the Department of Civil Engineering at the University of Gaziantep, Gaziantep, Turkey. He is a member of ACI Committee 237, Self-Consolidating Concrete. His research interests include advanced materials technology and composite materials development for sustainable infrastructure.*

Yingzi Yang *is an Associate Professor at the Harbin Institute of Technology, Harbin, China. Her research interests include durability of concrete at subzero temperatures and high-performance fiber-reinforced cementitious composites.*

ACI member **Victor C. Li** *is a Professor in the Department of Civil and Environmental Engineering at the University of Michigan. He is a member of ACI Committee 544, Fiber Reinforced Concrete. His research interests include micromechanics-based composite materials design, innovative structures design based on advanced materials technology, and sustainable infrastructure engineering.*

high composite tensile ductility.[1-4] The type, size, and amount of fiber; matrix ingredients; and interface characteristics are tailored for multiple cracking and controlled crack width in ECC. Specifically, high aggregate content and presence of coarse aggregates lead to a tough matrix that delays crack initiation and prevents steady-state flat-crack propagation in ECC, resulting in loss of tensile ductility. Therefore, instead of coarse aggregate, ECC incorporates fine silica sand with a sand-to-binder (cement + fly ash) ratio, by mass, (*S*/*B*), of 0.37 to maintain adequate stiffness and volume stability,[11] and to obtain the optimum gradation of particles to produce good workability.[12]

Fly ash (FA) has been used to replace cement in ECC mixture design for a number of reasons. The absence of coarse aggregate in ECC results in a higher cement content. Partial replacement using FA reduces the environmental burden. Further, it has been found that the addition of FA at high volumes improves the fresh and durability properities, reduces the drying shrinkage and matrix toughness, and improves the robustness of ECC in terms of tensile ductility.[13,14] Additionally, unhydrated FA particles with a small particle size (<45 μm) and smooth spherical shape serve as filler particles resulting in higher compactness of the fiber/matrix interface transition zone that leads to a higher frictional bond, which aids in reducing the steady-state crack width beneficial for the long-term durability of the structure.[13,14] Excessive FA, however, decreases the compressive strength of ECC. In this study, a high FA-to-cement ratio (*FA*/*PC*) of 2.8, by mass, was chosen to satisfy the above requirements, while still maintaining adequate compressive strength similar to that of normal-strength concrete.[14]

Table 1—Coded and absolute values for variables and orthogonal array for $L_9(3^4)$

Mix-ture no.	Factors, their coded (levels) and absolute values							
	C/*F*		*w*/*b*		*HRWR*/*B*, %		*VMA*/*B*, %	
	Coded	Abso-lute	Coded	Abso-lute	Coded	Abso-lute	Coded	Abso-lute
1	1	0	1	0.21	1	0.76	1	0.016
2	1	0	2	0.23	2	0.51	2	0.008
3	1	0	3	0.25	3	0.27	3	0.000
4	2	0.25	1	0.21	2	0.51	3	0.000
5	2	0.25	2	0.23	3	0.27	1	0.016
6	2	0.25	3	0.25	1	0.76	2	0.008
7	3	0.50	1	0.21	3	0.27	2	0.008
8	3	0.50	2	0.23	1	0.76	3	0.000
9	3	0.50	3	0.25	2	0.51	1	0.016

Although various fiber types have been used in the production of ECC, polyvinyl alcohol (PVA) fiber was adopted in the current version of ECC based on composite performance and economic considerations.[2] The dimensions of the PVA fiber are 8 mm (0.3 in.) in length and 39 μm (0.002 in.) in diameter. The nominal tensile strength and density of the fiber are 1600 MPa (235 ksi) and 1300 kg/m^3 (2190 lb/yd^3), respectively. A fiber content of 2% by volume is used in the mixture design. These decisions were made through ECC micromechanics material design theory and had been experimentally demonstrated to produce good ECC properties in previous investigations.[2,6]

From the previous discussion, *S*/*B*, *FA*/*PC*, fiber type, dimension, and content may be considered as constraints, and are treated as fixed parameters in the present study. Their specific details, summarized previously, should lead to optimal composite properties, according to micromechanics.[1] Improper processing, however, may lead to nonuniform fiber dispersion, inappropriate flaw size and distribution, or other microstructures not conducive to multiple cracking. Suitable processing control is necessary to realize composite materials with expected optimal properties. The following four parameters, not constrained or only loosely constrained by micromechanics, are considered important factors governing the rheological properties of ECC in the present study:

1. *C*/*F* (by mass), defined as the ratio of the amount of Class C FA to Class F FA, is used to approximate the variability of the quality of FA, widely known to vary from one source to another;
2. *w*/*b* (by mass), often used to adjust the workability of cementitious materials;
3. *HRWR*/*B* (by mass), reflecting the amount of HRWR, a commonly used admixture to enhance the fluidity of cementitious materials; and
4. *VMA*/*B* (by mass), reflecting the amount of VMA, sometimes used to enhance the stability of flowing cementitious materials, and to obtain better fiber distribution.[15]

These four factors are selected as “processing” variables in the following ED study.

STATISTICAL DESIGN APPROACH

In this experimental study, the Taguchi method[10] was adopted as a statistical method in ED. Taguchi[10] proposed a special set of orthogonal arrays to standardize fractional factorial designs and statistical methods to analyze the results. This approach can substantially reduce the number of experiments so that only a few tests with systematically chosen combinations of values of variables are required to separate and study the individual effects of each factor and their interactions. The theory behind this approach, which considers both the process of designing the experiment and the way of statistically analyzing the experimental data of response, is based on the usage of orthogonal array, the analysis of variance (ANOVA), and significance test with F statistic.[10,16] Taguchi’s[10] approach with an $L_9(3^4)$ orthogonal array was used herein to design the experiment with four factors with three levels each. The coded and absolute values for the variables are shown in Table 1. The values chosen represent those of a “standard” mixture ($C/F = 0$, $w/b = 0.21$, $HRWR/B$ (%) = 0.76, VMA/B (%) = 0) and its variations. Based on the balanced orthogonal array

(Table 1), the minimum required number of experiments for four factors at three levels is nine.

A four-factor ANOVA for the test results (response) was conducted by commercial statistical software. When the contribution of a factor is small, the sum of squares (*SS*) for the factor can be combined with the error SS_e, a procedure known as pooling. It is suggested that a factor can be pooled when the influence of the factor, that is, *SS*, is 10% or lower than the most influential factor.[17] After pooling, a test of significance can be executed based on the calculated *F*-ratio. The bigger the *F*-ratio, the larger the significant influence of the factor will be. The significance level is divided into three kinds:

1. $F \geq F_{0.01,f1,f2}$ Very significant
2. $F_{0.01,f1,f2} > F \geq F_{0.05,f1,f2}$ Significant
3. $F_{0.05,f1,f2} > F \geq F_{0.1,f1,f2}$ Normal

where f_1 and f_2 are the degrees of freedom *f*. Note that, physically, the value of *F* statistic represents the ratio of variance explained by factors to the unexplained variance by errors in the experiments. For some responses, pooling cannot be executed. In this scenario, the percentage contribution *P* was used to rank the influence of each factor on the response.

EXPERIMENTAL PROGRAM

Materials and mixture composition

The mixture proportions of all nine tests can be found in Table 2. The cement used in all mixtures was an ordinary portland cement (PC). Both Class C and Class F FA (C and F) used conformed to ASTM C618 requirements. The chemical composition and physical properties of the cement and FA are presented in Table 3. Scanning electron microscopy (SEM) images show that both FAs have spherically shaped particles with smooth surface characteristics. The particle size distributions of these materials (PC and FAs), obtained using a laser scattering technique, are given in Fig. 1. Fine silica sand with a maximum grain size of 250 μm (0.01 in.) and a mean size of 110 μm (0.004 in.) was adopted in ECC mixtures. The PVA fibers are purposely manufactured with a tensile strength, elastic modulus, and maximum elongation matching those needed for strain-hardening performance.[2] The PVA fiber tends to rupture instead of pull out in a cementitious matrix due to the strong chemical bonding with cement due to the presence of the hydroxyl group in its molecular chains.[2] Excessive fiber rupture limits the composite tensile strain capacity. For this reason, the surface of the PVA fibers is coated with a proprietary hydrophobic oiling agent of 1.2% by mass to tailor the interfacial properties between fiber and matrix for strain-hardening performance. Two chemical admixtures—a polycarboxylate HRWR with a solids content of 30%, and hydroxypropylmethylcellulose (VMA)—were used to control the rheological properties of ECC.

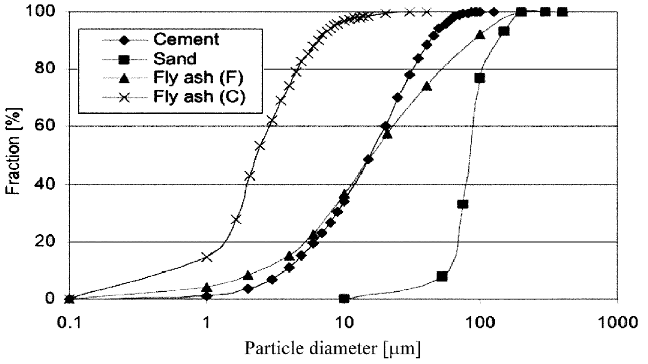


Fig. 1—Particle size distribution of cement, fly ashes, and sand.

Preparation, casting, and curing of specimens

As is well known, the mixer type, mixing procedure and speed, and temperature may affect the rheological properties of fresh cementitious material.[18] In this study, a paddle mixer was used in the preparation of all ECC mixtures. Solid ingredients, including PC, FA, and sand, were first dry mixed at 100 rpm for a minute. Water and chemical admixtures were then added and mixed at 150 rpm for 1 minute and then at 300 rpm for another 2 minutes to produce a consistent, uniform ECC mortar (ECC without PVA fiber). After measuring the fresh properties of each mortar, ECC mixture was produced by using the same amount of ingredients as mortar and PVA fiber was added in last and mixed at 150 rpm for an additional 3 minutes. From each ECC mixture, three 75 x 150 mm (3 x 6 in.) cylinder specimens were prepared for determining the compressive strength test at the age of 28 days and four 152.4 x 76.2 x 12.7 mm (6.0 x 3.0 x 0.5 in.) coupon specimens were prepared for conducting the uniaxial tensile test at the age of 28 days. All specimens

Table 2—Mixture proportions of ECC used in Taguchi method (dry basis)

Mixture no.	PC	Sand	F	C	Water	HRWR	VMA	PVA
	kg/m^3 (lb/yd^3)							
1	323 (544)	456 (768)	906 (1527)	0 (0)	262 (441)	9.3 (15.7)	0.2 (0.34)	26 (44)
2	323 (544)	456 (768)	906 (1527)	0 (0)	282 (475)	6.3 (10.6)	0.1 (0.17)	26 (44)
3	323 (544)	456 (768)	906 (1527)	0 (0)	302 (509)	3.3 (5.6)	0 (0)	26 (44)
4	323 (544)	456 (768)	680 (1146)	226 (381)	262 (441)	6.3 (10.6)	0 (0)	26 (44)
5	323 (544)	456 (768)	680 (1146)	226 (381)	282 (475)	3.3 (5.6)	0.2 (0.34)	26 (44)
6	323 (544)	456 (768)	680 (1146)	226 (381)	302 (509)	9.3 (15.7)	0.1 (0.17)	26 (44)
7	323 (544)	456 (768)	453 (763)	453 (763)	262 (441)	3.3 (5.6)	0.1 (0.17)	26 (44)
8	323 (544)	456 (768)	453 (763)	453 (763)	282 (475)	9.3 (15.7)	0 (0)	26 (44)
9	323 (544)	456 (768)	453 (763)	453 (763)	302 (509)	6.3 (10.6)	0.2 (0.34)	26 (44)

Table 3—Chemical composition and physical properties of portland cement and fly ashes

Chemical composition, %	PC	C	F
CaO	63.27	25.73	7.8
SiO_2	19.61	31.42	58.8
Al_2O_3	5.86	18.53	17.7
Fe_2O_3	3.40	5.19	5.4
MgO	0.95	6.44	—
SO_3	2.45	2.4	0.8
K_2O	0.54	0.48	0.56
Na_2O	0.47	2.24	1.54
Loss on ignition	3.02	0.17	0.3
Physical properties			
Specific gravity	3.18	2.78	2.35
Percent retained on No. 325 (45 μm) sieve	—	13.8	24.3

were demolded after 24 hours and cured in sealed plastic bags without external moisture supply at 23 ± 2 °C (73.4 °F) for 7 days. The specimens were then air cured at 50 ± 5% relative humidity, 23 ± 2 °C (73.4 ± 3.6 °F) until the age of 28 days for testing.

Test methods for determining fresh and hardened properties

The rheology of fresh mortar is most often described by the Bingham model.[19-22] According to this model, fresh mortar must overcome a limiting stress (yield stress) before it can flow. Once the mortar starts to flow, shear stress increases linearly with an increase in strain rate as defined by plastic viscosity. The plastic viscosity is defined as resistance to flow, once the yield stress is overcome. Therefore, to fully describe the rheological properties of fresh ECC mortar by the Bingham model, two parameters, namely, plastic viscosity and yield stress, are necessary. In this experimental study, rheological parameters are determined by a rotational viscometer. In addition, workability-related fresh properties of the ECC mortar were measured by means of mini-slump flow diameter, Marsh cone flow time. All tests were repeated twice with a new batch and the results indicate good repeatability with the coefficient of variance values less than 3%.

Rheological measurements—Rheological parameters of ECC mortar (plastic viscosity and relative yield stress) were measured using a rotational viscometer[22,23] at 23 ± 2 °C (73.4 ± 3.6 °F). This instrument with mortar probe (Fig. 2(a)) can accommodate mortars with a maximum particle size of 2 mm (0.08 in.). A rotation rate N is applied to the paddle, and the torque T of sample shear resistance is measured. The flow curve was recorded for both the ascending and descending legs of the shear stress-shear rate curve (Fig. 2(b)). The experimental shear stresses considered in this investigation were taken from the descending curve (dotted line in Fig. 2(b)). Assuming Bingham behavior, the flow curve can be represented by a straight line and the rheological parameters can be easily determined by linear regression

$$T = g + Nh \qquad (1)$$

where g (N·mm [lb·in.]) and h (N·mm·s [lb·in.·s]) are constants corresponding to yield stress and plastic viscosity, respectively. Because the data obtained from the rotational viscometer are not in proper rheological units, a calibration was performed by using the "General Purpose Silicone Oil (Brookfield)" to express h in proper plastic viscosity unit (Pa·s [psi·s]). Similar calibration has also been conducted by others,[24,25] and it has been shown that both torque and rotation obtained from the rotational viscometer are proportional to an average shear stress and shear rate. In this study, the constant g, found by fitting Eq. (1) to experimental data, is referred to as a "relative yield stress."

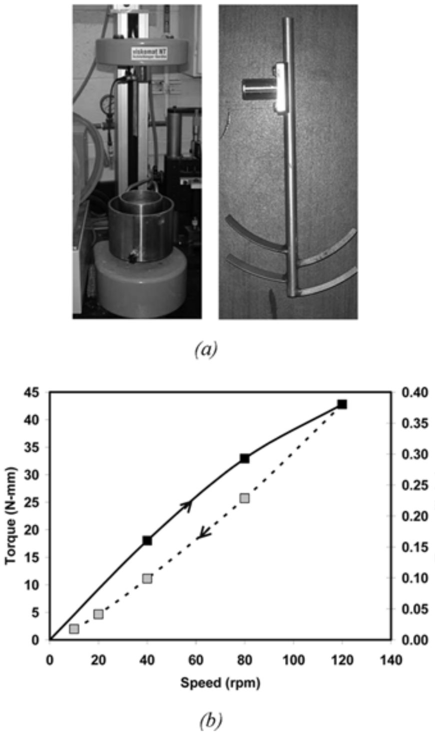


Fig. 2—(a) Rotational viscometer and mortar probe; and (b) typical flow behavior of ECC mortars obtained from rotational viscometer.

Mini-slump and funnel tests—The workability of fresh ECC mortar was evaluated by measuring the mini-slump flow deformation and Marsh cone flow time. In the mini-slump flow deformation test, a truncated cone mold with a diameter of 100 mm (3.94 in.) at the bottom and 70 mm (2.76 in.) at the top and a height of 60 mm (2.36 in.) was placed on a smooth plate, filled with mortar, and lifted upward. The slump flow deformation was defined as the dimension of the spread when the mortar stops flowing. The modified Marsh cone test was based on measuring the time necessary for the flow of a particular volume of mortar through a flow cone. A plastic funnel with a capacity of 1700 mL (0.06 ft^3) and an internal orifice diameter of 20 mm (0.787 in.) was used in this study. The cone was completely filled with mortar and the bottom outlet was then opened, allowing the mortar to flow out. The Marsh cone flow time of mortar was the elapsed time in seconds between the opening of the bottom outlet and the time when all mortar flow through the cone.

Mechanical properties—Mechanical properties were measured at the age of 28 days. The compressive strength was measured using 75 x 50 mm (3 x 6 in.) cylinder specimens. Uniaxial tensile tests with 152.4 x 76.2 x 12.7 mm (6.0 x 3.0 x 0.5 in.) coupon specimens were conducted using a servohydraulic testing system in displacement control mode. The loading rate used was 0.005 mm/s (0.0002 in./s) to simulate a quasi-static loading condition. Aluminum plates were glued on both sides at the coupon specimens' ends to facilitate gripping. Two external linear variable displacement transducers were attached to the specimen with a gauge length of approximately 50 mm (2 in.) to measure the specimen deformation. Complete tensile stress-strain curves were recorded. In this study, the tensile strain capacity of each ECC coupon specimen is defined as the strain value at peak load of the uniaxial tensile stress-strain curves.

RESULTS AND DISCUSSIONS

Table 4 presents the rheological and workability properties of nine ECC mixtures. The coefficients of correlation (R^2) of the Bingham model are also included in Table 4. The mechanical properties for all ECC mixtures are given in Table 5.

Analyses of rheological and mechanical properties

Rheological properties of ECC mortars and tensile properties of ECC mixtures were analyzed by means of ANOVA. This analysis was carried out to determine if the influences of the four selected variables on the rheological properties and mechanical properties of ECC were statistically significant or not. The analyses of test results enable the identification of major trends and predict the most promising direction for future ECC mixture optimization. In addition, these can reduce the cost and effort associated with the selection of trial batches.

Relative yield stress—Lower yield stress of the fresh mortar requires less stress to initiate flow (lower yield stress generally corresponds to higher slump flow). The fresh cementitious material stops slumping when the weight of the material (mass times gravitational forces) is lower than the yield stress.

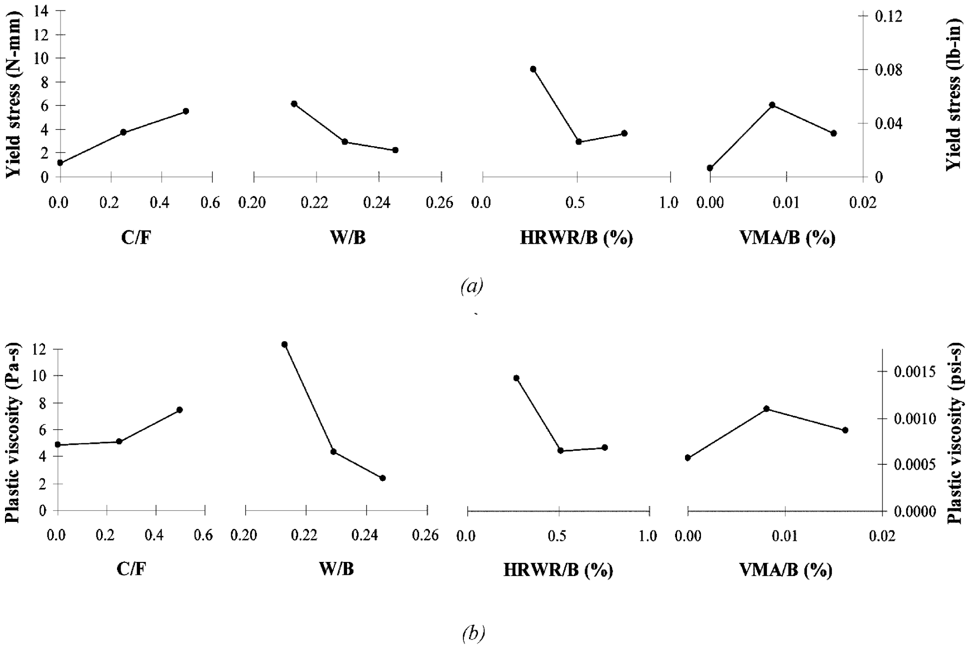
Figure 3(a) plots the effect of each factor on the relative yield stress of ECC mortar as measured by the rotational viscometer. As can be seen, an increase of *HRWR/B* and *w/b* reduces the relative yield stress of mortar. In contrast, an increase of *C/F* and *VMA/B* raises the relative yield stress. As seen from Fig. 3(a), however, at higher dosages of *HRWR/B* (more than 0.51% by mass) and *VMA/B* (more than 0.008% by mass), their influences on the relative yield stress of ECC mortar reverse the trends at smaller dosages. This suggests “saturation points” of the chemical admixtures. The issue of “saturation points” is discussed comprehensively in References 26 and 27. The ANOVA for mortar relative yield stress is shown in Table 6. No factor was pooled because none of them is considered an insignificant factor. *HRWR/B* has the highest percentage contribution and represents the most significant factor in altering mortar relative yield stress. This result is consistent with the well-known fact that polycarboxylate-based HRWR is an effective chemical admixture to reduce flocculation (aggregation) of cement particles and, therefore, lower the relative yield stress of fresh ECC mixture. Less known is the effect of *C/F* that is shown to substantially alter the rheological properties of ECC, but it is

Table 4—Fresh properties of ECC mortar (without PVA fibers) and ECC (with PVA fibers)

	ECC mortar (ECC without PVA fibers)					ECC
	Rheological parameters			Workability tests		
Mixture no.	Plastic viscosity, Pa·s (psi·s)	Relative yield stress, N·mm (lb·in.)	R^2, %	Marsh cone flow time, seconds	Mini-slump flow diameter, mm (in.)	Mini-slump flow diameter, mm (in.)
1	9.02 ($1.31E^{-3}$)	1.94 ($1.72E^{-2}$)	99.62	21.03	366 (14.41)	205 (8.07)
2	3.05 ($4.42E^{-4}$)	0.61 ($5.40E^{-3}$)	99.86	12.56	369 (14.53)	220 (8.66)
3	2.57 ($3.73E^{-4}$)	0.96 ($8.50E^{-3}$)	99.96	11.01	306 (12.05)	210 (8.27)
4	6.95 ($1.01E^{-3}$)	0.76 ($6.73E^{-3}$)	99.65	20.22	365 (14.37)	225 (8.86)
5	6.69 ($9.70E^{-4}$)	7.23 ($6.40E^{-2}$)	99.89	28.69	199 (7.84)	115 (4.53)
6	1.69 ($2.45E^{-4}$)	3.21 ($2.84E^{-2}$)	99.33	8.88	408 (16.06)	235 (9.25)
7	17.89 ($2.59E^{-3}$)	14.24 ($1.26E^{-1}$)	99.94	35.00	129 (5.08)	100 (3.94)
8	2.15 ($3.12E^{-4}$)	0.29 ($2.57E^{-3}$)	99.38	13.58	418 (16.46)	223 (8.78)
9	2.24 ($3.25E^{-4}$)	1.83 ($1.62E^{-2}$)	99.83	11.02	365 (14.37)	218 (8.58)

Table 5—Mechanical properties of ECC

Mixture no.	Compressive strength, MPa (ksi)	Ultimate tensile strength, MPa (ksi)	Tensile strain capacity, %
1	53.4 (7.74)	5.09 (0.74)	2.69
2	51.5 (7.47)	4.10 (0.59)	1.38
3	50.9 (7.38)	4.29 (0.62)	1.49
4	55.9 (8.11)	5.53 (0.80)	2.07
5	53.6 (7.77)	5.02 (0.73)	2.08
6	50.1 (7.26)	4.07 (0.59)	1.87
7	58.1 (8.42)	6.44 (0.93)	2.69
8	55.3 (8.02)	5.11 (0.74)	2.41
9	52.8 (7.66)	4.64 (0.67)	2.52



(a)

(b)

Fig. 3—The effect of each factor on: (a) relative yield stress; and (b) plastic viscosity of ECC mortar.

in agreement with research findings by Grzeszczyk and Lipowski.[28] An increase in the relative yield stress of mortar mixtures when *C/F* is increased can be attributed to the higher fineness of Class C FA (Fig. 1), resulting in higher particle surface area at constant *w/b*. The higher reactivity of Class C FA may also contribute to this phenomenon.

Plastic viscosity—Once the yield stress is overcome, plastic viscosity controls the spreading rate of the mortar flow. In self-consolidating concrete (SCC), viscosity is a dominant factor to prevent segregation caused by inhomogeneous flow between the ingredients of the fresh mixture and gravitational sedimentation. In the case of ECC, in addition to the segregation concern, viscosity of the fresh mortar may play an important role in fiber distribution, which affects fiber bridging efficiency, composite hardened properties in general, and tensile behavior in particular.

Table 6—ANOVA for relative yield stress of ECC mortar

Factors	Pooling	DOF *f*	Sum of squares *SS*	Variance *V*	F-ratio *F*	Pure SS *SS'*	Percentage contribution *P*, %
C/F	No	2	27.876	13.938	—	27.876	16.676
w/b	No	2	22.426	11.213	—	22.426	13.415
HRWR/B	No	2	73.718	36.859	—	73.718	44.1
VMA/B	No	2	43.14	21.57	—	43.14	25.807
Error	—	0	—	—	—	—	—
Total	—	8	167.162	—	—	—	100

Table 7—ANOVA for plastic viscosity of ECC mortar

Factors	Pooling	DOF *f*	Sum of squares *SS*	Variance *V*	F-ratio *F*	Pure SS *SS'*	Percentage contribution *P*, %
C/F	Yes	(2)	(11.905)				
w/b	No	2	140.033	70.016	11.762	128.128	58.363
HRWR/B	No	2	47.432	23.716	3.984	35.572	16.183
VMA/B	No	2	20.162	10.081	1.693	8.257	3.761
Error	—	2	11.904	5.952	—	—	21.693
Total	—	8	219.534	—	—	—	100

Note: $F_{0.01,2,2} = 99$; $F_{0.05,2,2} = 19$; $F_{0.1,2,2} = 9$.

Figure 3(b) plots the effect of each factor on ECC mortar plastic viscosity. In general, an increase of *w/b* and *HRWR/B* reduce the mortar plastic viscosity, and an increase of *VMA/B* and *C/F* elevate the viscosity. Similar to the relative yield stress test results, however, there are chemical admixture "saturation dosages" (HRWR and VMA) beyond which their influences on the plastic viscosity of ECC mortars reverse trend. The ANOVA for plastic viscosity of ECC mortar is shown in Table 7. As can be seen, *C/F* was considered insignificant and was pooled. The *F* statistic indicates that *w/b* has the most significant impact on mortar plastic viscosity. This observation is particularly beneficial because it provides insights to control plastic viscosity in producing ECC to obtain good fiber distribution and to avoid segregation.

From the above ANOVA for rheological properties, the relative yield stress and the plastic viscosity are determined to be mainly dominated by two different factors, namely, *HRWR/B* and *w/b*, respectively. For given ingredients, this observation suggests an easy way to control the relative yield stress and plastic viscosity of ECC independently in future mixture design.

Tensile strain capacity—ECC material ductility enhances structural performance in terms of load-carrying capacity and deformability and represents the most important characteristic of ECC mechanical properties.[1] As can be seen from Fig. 4(a), an increase of *C/F* and *VMA/B* tends to improve the tensile strain capacity. An increase of *w/b* tends to reduce the tensile strain capacity. Table 8 gives the ANOVA for the tensile strain capacity. Among the four factors, *C/F* has the largest contribution (positive) to the tensile strain capacity and *w/b* is the second important factor (negative) to affect the tensile strain capacity.

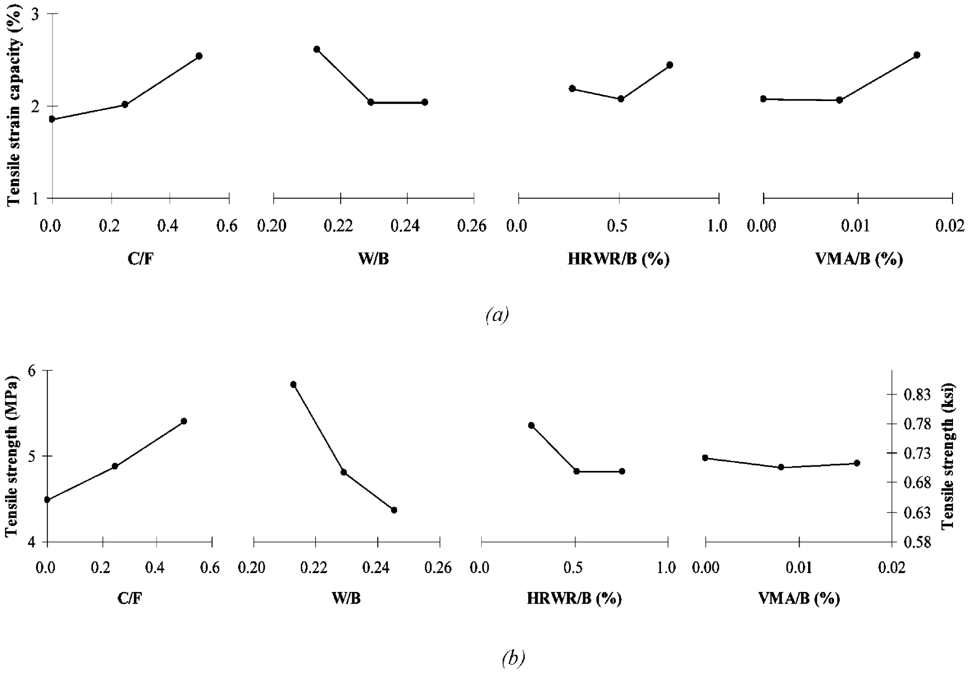
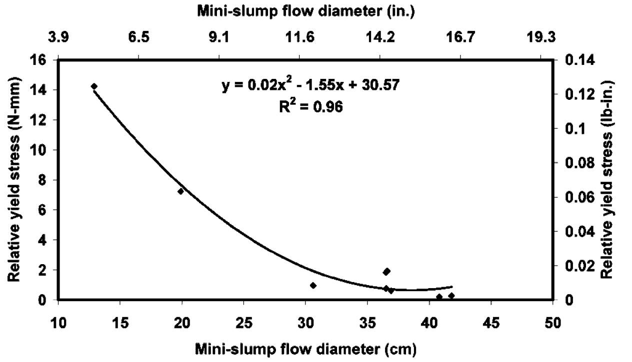


Fig. 4—The effect of each factor on: (a) tensile strain capacity; and (b) ultimate tensile strength of ECC.

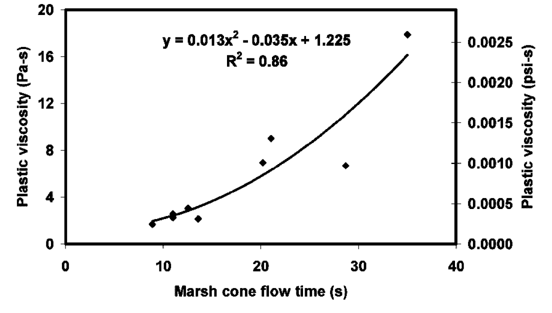
Ultimate tensile strength—Figure 4(b) shows the effect of each factor on composite ultimate tensile strength. As can be seen, a decrease of *w*/*b* and an increase of *C*/*F* tends to increase the ultimate tensile strength. Table 9 gives the ANOVA for ultimate tensile strength. Among the four factors, *w*/*b* is qualified as the very significant factor (negative) and *C*/*F* is qualified as the significant factor (positive) for composite ultimate tensile strength.

From the above ANOVA for hardened properties of ECC, *w*/*b* and *C*/*F* are the two most important factors in altering composite ultimate tensile strength and tensile strain capacity. Interestingly, high *w*/*b* tends to reduce both the ultimate tensile strength and tensile strain capacity, and high *C*/*F* is likely to increase those two ECC hardened properties.

It is known that an increase of *w*/*b* can reduce the cementitious particle concentration, resulting in relatively loose microstructure, and therefore introduce a lower interface frictional bond.[6] This weak interface bonding can potentially generate lower fiber bridging stress that can result in low ultimate tensile strength and tensile strain capacity. In addition, *w*/*b* has the highest influence on the plastic viscosity of ECC mortar. High *w*/*b* can substantially decrease the plastic viscosity of ECC mortar and may result in poor fiber distribution along with low ultimate tensile strength and tensile strain capacity. On the other hand, the effect of *C*/*F* has been recognized as insignificant on the plastic viscosity of ECC mortar. High ultimate tensile strength and tensile strain capacity at high *C*/*F* are attributed to the change of microstructure by incorporating more Class C FA. Class C FA with smaller particle size (Fig. 1) and higher reactivity when compared with Class F FA may introduce higher interfacial frictional bonding and, therefore, high ultimate tensile strength and tensile strain capacity. These observations will require verifications via single fiber pullout test in mortar matrix with different *C*/*F*.[13]



(a) Relative yield stress versus mini-slump flow



(b) Plastic viscosity versus Marsh cone flow time

Fig. 5—Rheological parameters versus workability test results of ECC mortar.

Correlation between rheological, workability, and mechanical properties

To identify any correlation between the rheological parameters (plastic viscosity and relative yield stress) and workability test results (Marsh cone flow time and mini-slump flow deformation), graphs were drawn between fresh test results of mortars and coefficients of determinations (R^2) between any of the two fresh tests were calculated.

The slump flow diameter is known to be an estimate of the deformability of concrete, which is related to the yield stress.[21,29-31] For that reason, the relative yield stress of ECC mortar is also well correlated with the mini-slump flow deformation of mortar and is illustrated in Fig. 5(a). It can be observed from Fig. 5(a) that the slump flow of ECC mortar is found to increase with the decrease of its relative yield stress. The relative yield stress showed very good polynomial correlation (R^2 = 0.96) with its slump flow (Fig. 5(a)). Therefore, a qualitative indication of the relative yield stress could be obtained from the mini-slump flow deformation test. This result was expected because the ECC mortar in a mini-slump flow test will flow if the stress due to weight of the mortar in the cone is higher than its yield stress.[32]

As seen from Fig. 5(b), a high polynomial correlation (R^2 = 0.86) is observed between plastic viscosity and Marsh cone flow time. In the Marsh cone flow test, the ECC mortar starts to flow when the yield stress is exceeded; therefore, the measured value is highly related to the viscosity. Therefore, these relatively simple test methods of mini-slump flow deformation and Marsh cone flow time can be used in field conditions instead of measuring yield stress and plastic viscosity of ECC mortar using a rheometer.

As discussed previously, the fresh properties of ECC mortar is expected to have direct impacts on hardened properties of ECC. To evaluate the effect of rheological properties on the mechanical properties of ECC, plastic

Table 8—ANOVA for tensile strain capacity of ECC

Factors	Pooling	DOF *f*	Sum of squares *SS*	Variance *V*	F-ratio *F*	Pure SS *SS'*	Percentage contribution *P*, %
C/*F*	No	2	0.779	0.389	—	0.799	40.949
w/*b*	No	2	0.551	0.275	—	0.551	28.96
HRWR/*B*	No	2	0.176	0.088	—	0.176	9.27
VMA/*B*	No	2	0.396	0.198	—	0.396	20.814
Error	—	0	—	—	—	—	—
Total	—	8	1.903	—	—	—	100

Table 9—ANOVA for ultimate tensile strength of ECC

Factors	Pooling	DOF *f*	Sum of squares *SS*	Variance *V*	F-ratio *F*	Pure *SS* *SS'*	Percentage contribution *P*, %
C/*F*	No	2	1.234	0.617	71.908	1.217	26.305
w/*b*	No	2	2.889	1.444	168.337	2.872	62.077
HRWR/*B*	No	2	0.486	0.243	28.357	0.469	10.148
VMA/*B*	Yes	(2)	(0.017)	—	—	—	—
Error	—	2	0.016	0.008	—	—	1.47
Total	—	8	4.627	—	—	—	100

Note: $F_{0.01,2,2}$ = 99; $F_{0.05,2,2}$ = 19; $F_{0.1,2,2}$ = 9.

viscosity and relative yield stress of ECC mortar are plotted against ultimate tensile strength and tensile strain capacity of ECC. As can be seen from Fig. 6(a) and (b), a strong correlation was found between the plastic viscosity and tensile properties of ECC. Due to a good correlation between plastic viscosity and Marsh cone flow time (Fig. 5(b)), a strong correlation was also found between the Marsh cone flow time and tensile properties (Fig. 6(c) and (d)). On the other hand, no correlation was found between relative yield stress and tensile properties.

Figure 6(a) shows the relation between tensile strain capacity and plastic viscosity from the nine mixtures. As described in the ANOVA for tensile strain capacity, *C/F* represents the most significant factor in determining the tensile strain capacity and ultimate tensile strength, both of which increase with *C/F*. The effect of an increase of *C/F* on the tensile properties of ECC can be clearly observed by plotting the data into three sets based on different *C/F*, as shown in Fig. 6(a) and (c). In addition, strong correlations were found between tensile strain capacity and plastic viscosity for each *C/F*. Therefore, in addition to *C/F*, plastic viscosity is another important factor governing tensile strain capacity. This observation suggests that better fiber distribution (by means of segregation prevention) and bridging efficiency can be obtained, resulting in higher tensile strain capacity in mixtures with higher plastic viscosity.

Figure 6(b) shows the general trend of increase of ultimate tensile strength with the increase of plastic viscosity among the nine mixtures. Again, better fiber distribution and bridging efficiency at higher viscosity contribute to higher ultimate tensile strength. These observations suggest that adequate viscosity, in addition to properly tailored material ingredients, is the key to obtaining high ultimate tensile strength and high tensile strain capacity in ECC.

Table 10—Effects of factors on different responses of ECC

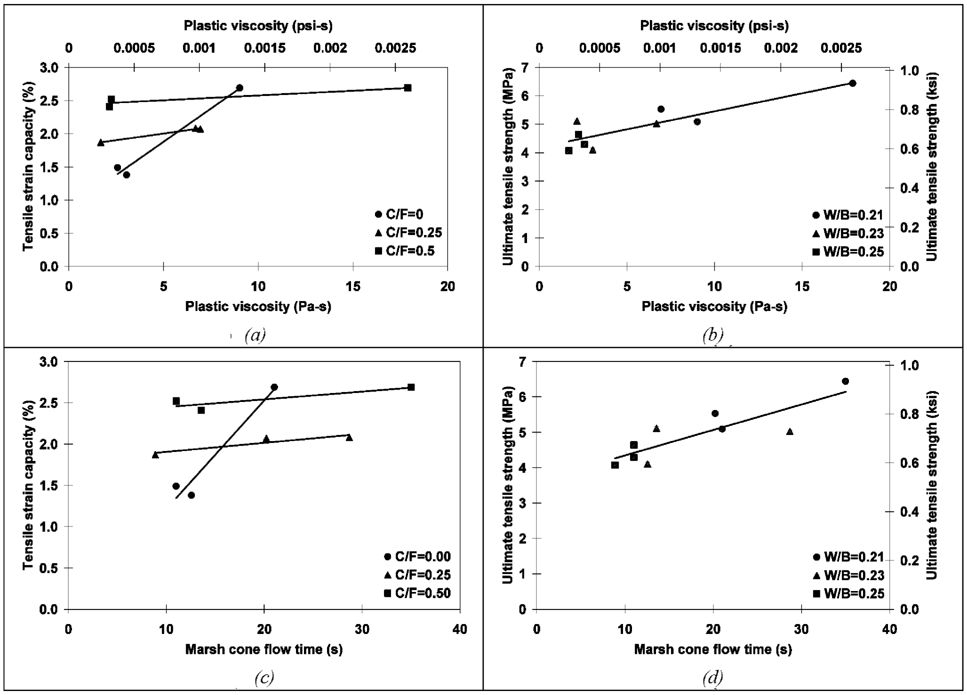
Response	Performance characteristics	C/F (↑)	*w/b* (↑)	*HRWR/B* (↑)	*VMA/B* (↑)
Plastic viscosity	Bigger is better	(+1)	–4	–3	+2
Relative yield stress	Smaller is better	–3	+1	+4	?2
Ultimate tensile strength	Bigger is better	+3	–4	–2	(–1)
Tensile strain capacity	Bigger is better	+4	–3	?1	+2

Note: + is positive impact on performance requirement; – is negative impact on performance requirement; ? is uncertain trend of impact; 4 is highest impact; 1 is lowest impact; and ( ) is insignificant/pooled factor.

Rheological control of fresh properties in producing ECC

Based on the aforementioned analyses, performance characteristics in producing ECC for different responses are summarized in Table 10. For fresh properties, high plastic viscosity is desirable for better fiber distribution, whereas low yield stress is required for better deformability of ECC. For hardened properties, high ultimate tensile strength and high tensile strain capacity are preferred.

As can be seen in Table 10, changes in the four factors can cause significant changes in the fresh and hardened properties of ECC. An increase in *C/F* has significant enhancement in ultimate tensile strength (+3) and tensile strain capacity (+4). An increase of *w/b* has very significant negative impacts (–4, –3) on almost all the responses, except for a small positive contribution on relative yield stress. Therefore, it is recommended that a smaller amount of water be used to produce ECC with high viscosity. *HRWR/B* contributes mainly to the relative yield stress of ECC as described in ANOVA. Therefore, after deciding on the *w/b*, an adequate amount of HRWR is the key to produce ECC with adequate viscosity (good fiber distribution) and low yield stress (good deformability) without segregation. In general, VMA helps to improve the fresh properties of ECC. The high cost of VMA, however,



(a) (b)

(c) (d)

Fig. 6—Relation between mechanical properties of ECC versus plastic viscosity and Marsh cone flow time of ECC mortar.

may limit its use in the field. As seen from fresh and hardened test results, ECC with adequate viscosity and good workability can easily be obtained through control of *w*/*b* and *HRWR*/*B* without depending on VMA. In some conditions, for example, where a raw material causes a negative rheological change, VMA may be used during the production of ECC to readjust the rheological properties.

A qualified fresh property (adequate plastic viscosity and low yield stress) should be obtained by adjusting *w*/*b* and *HRWR*/*B*. It should be noted, however, that a drastic change of *w*/*b* can alter material microstructure and, therefore, the hardened properties. In standard PVA-ECC mixture design, a low *w*/*b* was determined through micromechanics to satisfy proper interface properties. Therefore, it is suggested that a reasonable *w*/*b* should be in the range of 0.25 ± 0.05 based on past experience.[12] It should also be noted that fiber type and fiber length can cause significant impact on required viscosity for good fiber distribution and required yield stress for good workability performance. For example, longer fiber may require higher viscosity for better distribution and lower yield stress for good workability. In some cases, a satisfactory fresh matrix cannot be obtained by adjusting *w*/*b* and *HRWR*/*B*. In this scenario, a change of source of raw materials and type of HRWR, and adoption of additional chemical additives may be considered.

To reproduce ECC with local ingredients, it is recommended that the first attempt should be focused on optimizing fresh properties. It should be a relatively easy effort to maximize composite hardened properties for a given ECC mixture design optimized through micromechanics. If the fresh properties have been optimized and the hardened properties are still not satisfactory, material ingredient retailoring should then be performed, following guidelines from micromechanics. It is expected that robust ECC can be produced around the world by simultaneously controlling fresh properties and using appropriate tailored material ingredients such as tailored PVA fiber with special geometric and mechanical properties.[1]

CONCLUSIONS AND RECOMMENDATIONS

The influence of different combinations of factors *C*/*F*, *w*/*b*, *HRWR*/*B*, and *VMA*/*B* on the fresh properties of ECC mortars and mechanical properties of ECC mixtures was investigated. Based on the results presented in this paper, the following conclusions can be drawn:

1. Among the four factors, *w*/*b* has the greatest effect on the plastic viscosity, whereas *HRWR*/*B* has the largest impact on the relative yield stress of ECC mortar in the selected range of the mixture proportions. The increase of *w*/*b* and *HRWR*/*B* dosage leads to a reduction in plastic viscosity and relative yield stress, respectively;

2. The plastic viscosity of fresh ECC mortar has a significant impact on ECC tensile properties. The tensile strain and ultimate tensile strength of ECC were found to increase with the increase of plastic viscosity and Marsh cone flow time, which may be attributed to better fiber distribution in fresh mortar with high viscosity. A direct correlation between viscosity and fiber dispersion uniformity is being studied in continuing investigations; and

3. Good correlations between relative yield stress and mini-slump flow deformation, and between plastic viscosity and Marsh cone flow time, were established. Therefore, the rheological properties of ECC mortar can be effectively evaluated by the simpler mini-slump flow diameter and Marsh cone flow time in the field for quality control purposes.

Whereas each of these conclusions is not unexpected and largely consistent with those in the published literature, the information presented in this paper is the first systematic dataset from experiments conducted on ECC material. Based on these findings, it is recommended that a *w*/*b* in the range of 0.25 ± 0.05 be adopted in the ECC mixture, and a high plastic viscosity (high Marsh cone flow time) and a low yield stress (high mini-slump flow diameter) be achieved through adjustment of the amount of HRWR. Whereas variations in local ingredients may be unpredictable, rheological control as described in this paper provides a relatively simple means of ensuring an ECC composite with robust tensile strength and strain capacity. The recommendations for rheological control of fresh properties in producing ECC should assist in successful adaptation of ECC using local ingredients and widely available equipment.

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REFERENCES

1. Li, V. C., "Engineered Cementitious Composites—Tailored Composites through Micromechanical Modeling," *Fiber Reinforced Concrete: Present and the Future*, N. Banthia, A. Bentur, and A. Mufti, eds., Canadian Society for Civil Engineering, Montreal, QC, Canada, 1997, pp. 64-97.

2. Li, V. C.; Wang, S.; and Wu, C., "Tensile Strain-Hardening Behavior of PVA-ECC," *ACI Materials Journal*, V. 98, No. 6, Nov.-Dec. 2001, pp. 483-492.

3. Lin, Z., and Li, V. C., "Crack Bridging in Fiber Reinforced Cementitious Composites with Slip-Hardening Interfaces," *Mechanics and Physics of Solids*, V. 45, No. 5., 1997, pp. 763-787.

4. Lin, Z.; Kanda, T.; and Li, V. C., "On Interface Property Characterization and Performance of Fiber-Reinforced Cementitious Composites," *Concrete Science and Engineering*, V. 1, 1999, pp. 173-184.

5. Kong, H. J.; Bike, S.; and Li, V. C., "Development of a Self-Consolidating Engineered Cementitious Composite Employing Electrosteric Dispersion/Stabilization," *Cement and Concrete Composites*, V. 25, No. 3, 2003, pp. 301-309.

6. Kong, H. J.; Bike, S.; and Li, V. C., "Constitutive Rheological Control to Develop a Self-Consolidating Engineered Cementitious Composite Reinforced with Hydrophilic Poly(vinyl Alcohol) Fibers," *Cement and Concrete Composites*, V. 25, No. 3, 2003, pp. 333-341.

7. Kim, Y. Y.; Kong, H. J.; and Li, V. C., "Design of Engineered Cementitious Composite (ECC) Suitable for Wet-Mix Shotcreting," *ACI Materials Journal*, V. 100, No. 6, Nov.-Dec. 2003, pp. 511-518.

8. Kunieda, M., and Rokugo, K., "Recent Progress on HPFRCC in Japan," *Journal of Advanced Concrete Technology*, V. 4, No. 1, 2006, pp. 19-33.

9. Li, V. C.; Lepech, M.; and Li, M., "Field Demonstration of Durable Link Slabs for Jointless Bridge Decks Based on Strain-Hardening Cementitious Composites," *Michigan DOT Report*, 2005.

10. Taguchi, G., *System of Experimental Design*, Quality Resources, New York: International Publications, 1985.

11. Li, V. C.; Mishra, D. K.; and Wu, H. C., "Matrix Design for Pseudo Strain-Hardening Fiber Reinforced Cementitious Composites," *Materials and Structures*, V. 28, No. 183, 1995, pp. 586-595.

12. Fischer, G., and Li, V. C., "Design of Engineered Cementitious Composites (ECC) for Processing and Workability Requirement," *Proceedings of BMC 7*, Poland, 2003, pp. 29-36.

13. Wang S., and Li, V. C., "Engineered Cementitious Composites with High-Volume Fly Ash," *ACI Materials Journal*, V. 104, No. 3, May-June 2007, pp. 233-241.

14. Yang, E. H.; Yang, Y.; and Li, V. C., "Use of High Volumes of Fly Ash to Improve ECC Mechanical Properties and Material Greenness,"

ACI Materials Journal, V. 104, No. 6, Nov.-Dec. 2007, pp. 620-628.

15. Özyurt, N.; Mason, T. O.; and Shah, S. P., “Correlation of Fiber Dispersion, Rheology and Mechanical Performance of FRCs,” *Cement and Concrete Composites*, V. 29, No. 2, 2007, pp. 70-79.

16. Garcia-Diaz, A., and Phillips, T., *Principles of Experimental Design and Analysis*, Chapman & Hall, 1995, 409 pp.

17. Roy, R. K., *Design of Experiments Using the Taguchi Approach: 16 Steps to Product and Process Improvement*, John Wiley & Sons Inc., New York, 2001, 560 pp.

18. Ferraris, C. F.; Obla, K.; and Hill, R., “The Influence of Mineral Admixtures on the Rheology of Cement Paste and Concrete,” *Cement and Concrete Research*, V. 31, No. 2, 2001, pp. 245-255.

19. Ozawa, K.; Maekawa, K.; Kunishima, M.; and Okamura, H., “Development of High-Performance Concrete Based on the Durability Design of Concrete Structures,” *Proceedings of the Second East-Asia and Pacific Conference on Structural Engineering and Construction (EASEC-2)*, 1989, pp. 445-450.

20. Struble, L. J.; Puri, U.; and Ji, X., “Concrete Rheometer,” *Advances in Cement Research*, V. 13, No. 2, 2001, pp. 53-63.

21. Ferraris, C. F., and de Larrard, F., “Testing and Modeling of Fresh Concrete Rheology,” *Interagency Report* 6094, National Institute of Standards and Technology, 1998.

22. Banfill, P. F. G.; Starrs, G.; Derruau, G.; McCarter, W. J.; and Chrisp, T. M., “Rheology of Low Carbon Fibre Content Reinforced Cement Mortar,” *Cement and Concrete Composites*, V. 28, No. 9, 2006, pp. 773-780.

23. http://www.schleibinger.com. (accessed 11/24/08)

24. Flatt, R.; Larosa, D.; and Roussel, N., “Linking Yield Stress Measurements: Spread Test Versus Viskomat,” *Cement and Concrete Research*, V. 36, No. 1, 2006, pp. 99-109.

25. Banfill, P. F. G., “The Rheology of Fresh Mortar,” *Magazine of Concrete Research*, V. 43, No. 154, 1991, pp. 13-21.

26. Agullo, L.; Toralles-Carbonari, B.; Gettu, R.; and Aguado, A., “Fluidity of Cement Pastes with Mineral Admixtures and Superplasticizer—A Study Based on the Marsh Cone Test,” *Materials and Structures*, V. 32, No. 7, 1999, pp. 479-485.

27. Yahia, A., and Khayat, K. H., “Experiment Design to Evaluate Interaction of High-Range Water-Reducer and Antiwashout Admixture in High-Performance Cement Grout,” *Cement and Concrete Research*, V. 31, No. 5, 2001, pp. 749-757.

28. Grzeszczyk, S., and Lipowski, G., “Effect of Content and Particle Size Distribution of High-Calcium Fly Ash on the Rheological Properties of Cement Pastes,” *Cement and Concrete Research*, V. 27, No. 6, 1997, pp. 907-916.

29. Sahmaran, M.; Yaman, I. O.; and Tokyay, M., “Fresh Properties of High-Volume Fly Ash Self-Consolidating Concretes,” *Recent Advances in Concrete Technology*, Proceedings of the 8th CANMET/ACI International Conference, SP-235, V. M. Malhotra, ed., American Concrete Institute, Farmington Hills, MI, 2006, pp. 189-202.

30. Ferraris, C. F., “Measurement of the Rheological Properties of High Performance Concrete,” *Journal of Research of the National Institute of Standards and Technology*, V. 104, No. 4, 1999, pp. 461-477.

31. Struble, L. J., and Chen, C., “Effect of Continuous Agitation on Rheology,” *Journal of ASTM International*, V. 2, No. 9, 2005, 16 pp.

32. Lachemi, M.; Hossain, K. M. A.; Nkinamubanzi, P. C.; and Bouzoubaâ, N., “Performance of New Viscosity Modifying Admixtures in Enhancing the Rheological Properties of Cement Paste,” *Cement and Concrete Research*, V. 34, No. 2, 2004, pp. 185-193.