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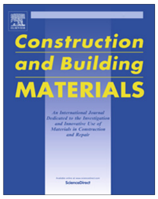

Study on fiber hybridization effect of engineered cementitious composites with low- and high-modulus polymeric fibers

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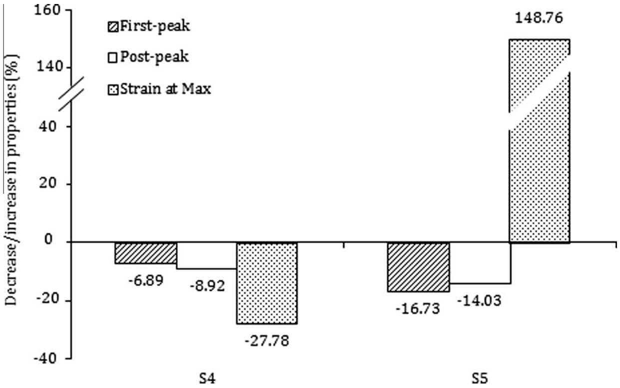
HIGHLIGHTS

- Improving the ductility of ECC composite with hybrid fibers.
- Study of low and high modulus polymeric fibers in hybrid composite.
- Study of the effect of fiber cross-sectional shape on the flexural behavior of hybrid ECC composite.
- Hybridization of PP fibers with different cross-sectional shape and PVA fiber in ECC composite.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study the flexural and compressive properties of hybrid engineered cementitious composite (ECC) was investigated. The hybridization with low- and high-modulus fibers was employed to increase deformability and flexural strain capacity of the ECC composite. It was found that the hybridization with non-round polypropylene (PP) fiber and low modulus polyvinyl alcohol (PVA) fiber have remarkable effect on the improving strain-capacity of resultant composite. The strain-capacity of hybrid ECC was increased by replacement of 20 vol.% of triangular PP fiber and low modulus PVA fiber up to 33% and 148%, respectively. This hybridization also increased the toughness ratio and meets the requirements for the strain-hardening with multiple cracking behaviors of cementitious composites.

1. Introduction

In the past two decades, engineered cementitious composites has been developed which exhibits strain-hardening behavior accompanied by multiple cracking resulting in higher strength and tensile ductility [1].

ECCs are known as cement based composites that have an ultimate strength higher than the first cracking strength. The tensile strain capacity of ECCs is 2 to 5% and shows the averaged tight crack width development about 60 μm even when strained to beyond 1% [2]. These properties can be achieved by using high modulus fibers such as PVA with moderate fiber volume fraction (e.g. typically 2%) [3]. They have industrial usage for a broad range of applications which needs load carrying capacity, deformability, and energy absorption capacity under monotonic and revers cyclic loading [4]. These high performances with a moderate fiber content combination are attained by micromechanics-based composite optimization.

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Table 1
Physical/mechanical characteristics of fibers.

Fiber		Cross-sectional shape	Diameter [μm]	Length [mm]	l/d ratio	Tensile strength [MPa]	Elongation at break [%]	Modulus of elasticity [GPa]
High Modulus	PVA-C	Circular	7.5	4	533	1600	7	35
	PVA-K	Kidney	14	6	428	1400	6–7	20
	PVA-N	Circular	38	8	210	1100	6	40
Low Modulus	PP-C	Circular	25	6	240	320	80–90	2
	PP-T	Triangular	28	6	214	300	80–90	1.5

Table 2
Chemical and physical properties of cement.

SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	Others (%)
21.6	4.61	3.49	64.07	1.94	2.59	1.7

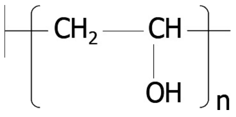
Table 3
The mix design of engineered cementitious composite (weight ratio).

Cement	Sand	Fly ash	Water [%]*	HRWR** [%]	Fiber [vol.%]
1	0.8	1.2	25.2	2.4	2

* Weight to cementitious materials ratio.
** High range water reducer, weight to cement ratio.

Table 4
The produced mixes with volume percentage of fibers.

Experimental program	Notation	PVA-N	PVA-C	PVA-K	PP-C	PP-T
First Part	S01	0	1.5%	0	0	0
	S02	0	0	1.5%	0	0
	S03	1.5%	0	0	0	0
Second Part	S1 (control)	2%	0	0	0	0
	S2	1.6%	0	0	0.4%	0
	S3	1.6%	0	0	0	0.4%
	S4	1.6%	0.4%	0	0	0
	S5	1.6%	0	0.4%	0	0



$$-\left[CH_2-\underset{\displaystyle OH}{CH}\right]_n-$$

Fig. 1. Chemical structure of PVA.

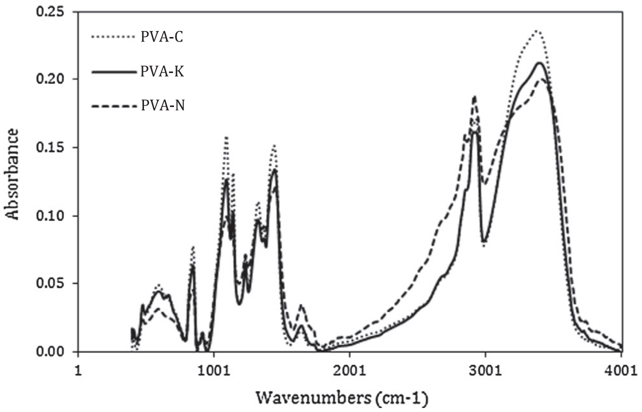


Fig. 2. FTIR spectra of different PVA fiber.

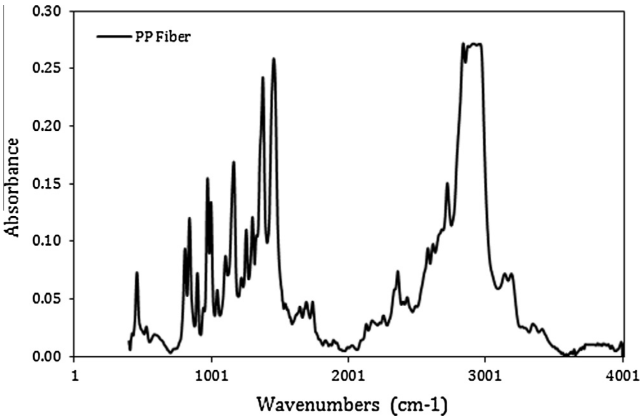


Fig. 3. FTIR spectra of PP fibers.

Table 5
Details of flexural behavior of ECC samples containing 1.5 vol.% of different PVA fiber.

	First-peak strength (MPa)	Post-peak strength (MPa)	Def. at Max load (mm)	Area under curve to def. = 1.5 mm
S01	8.59 + 1.23	11.22 + 0.38	2.85 + 0.07	1735.61 + 24.45
S02	9.22 + 0.16	9.73 + 0.55	3.41 + 0.33	2062.00 + 65.30
S03	8.06 + 0.00	9.24 + 0.48	4.11 + 0.10	2556.40 + 22.53

To achieve higher deformation and ductility performances are of great interest from the point of material design. The design criterion for ECC that relates fiber, matrix and interface parameters has first proposed by Li and Leung [5]. The strain-hardening behavior can be attained by tailoring the synergistic interaction between fiber, matrix and interface.

A major challenge of using PVA fibers in ECCs is the fact that these fibers have high hydrophilic nature which capable them to develop strong chemical bonding to cementitious matrix. The development of chemical bonding between the PVA fibers and cementitious materials is due to the presence of hydroxyl groups in their chemical structure. This strong chemical bonding can cause PVA fiber rupture instead of fiber pull-out during load bearing that tends to limit the multiple cracking effect and strain capacity of ECC in the post-cracking zone [6]. Many attempts have been made to develop a ductile fine aggregate concrete using tailoring fiber, matrix, and interface. It has been reported that the deformability of ECC can be modified by matrix tailoring using fine sand [7]. It was also reported that applying oil coating reduced chemical bonding strength of the PVA fiber to the cementitious matrix and significantly increased the ductility of PVA-ECC composite [8]. The chemical bonding of PVA fiber to ECC matrix drops with an increase in fly ash content due to the result of lower hydration degree in fiber/matrix interface [9]. Increase in fly ash content is also appropriate for reduction in crack width and increase in the frictional bond. It was exhibited that the ECC mix designed with slag particles shows higher toughness ratio which is helpful for

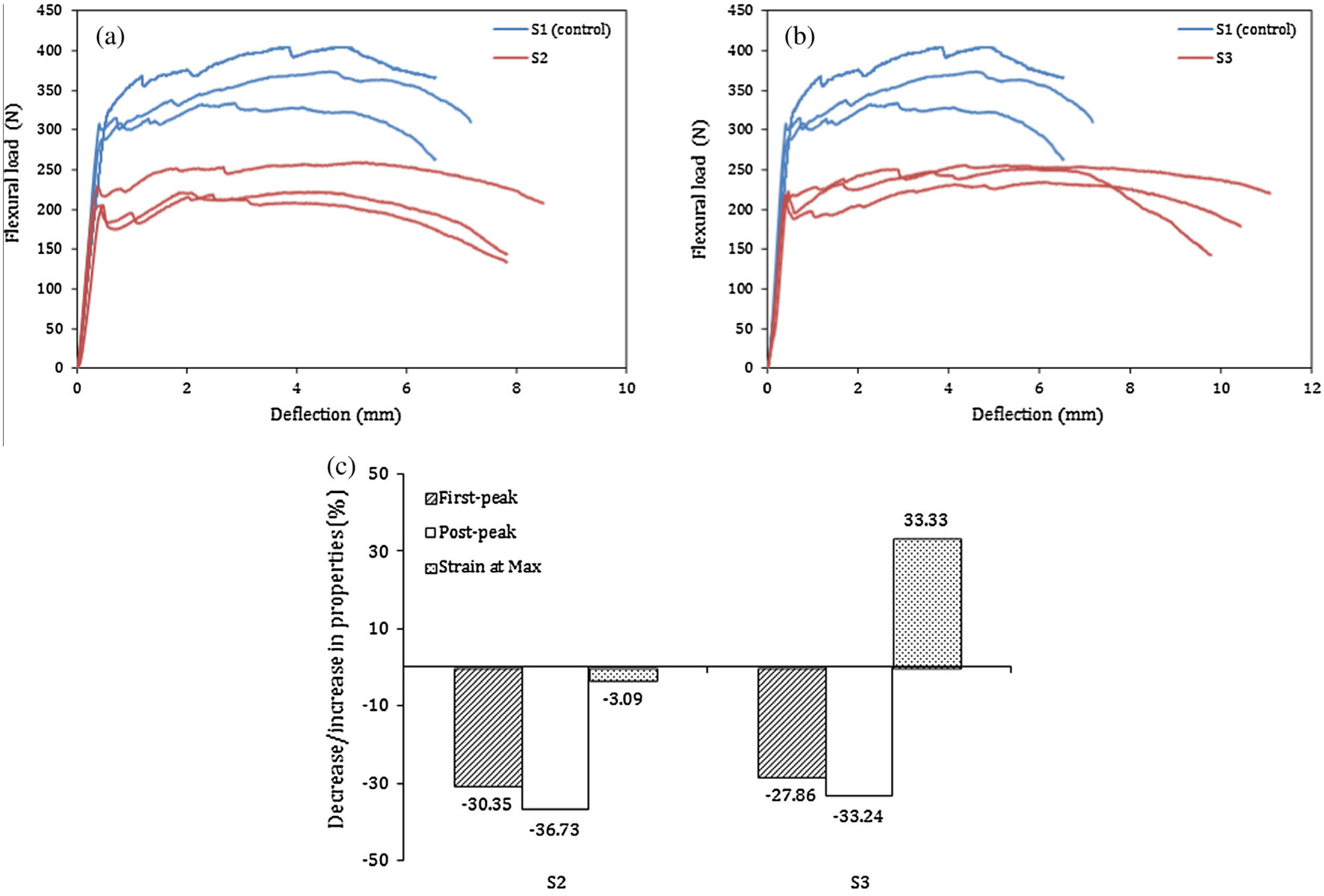


Fig. 4. Flexural load-deflection curve of composite containing; a) S1 vs S2, b) S1 vs S3, and c) Decrease/increase in the properties respect to the S1.

achieving strain hardening behavior associated with an increase up to 50% in the tensile strain capacity [10].

Both chemical bonding and slippage of fibers across the generated cracks in the post-cracking zone are necessary to achieve strain-hardening behavior. The amount of these mechanisms of bonding (chemical and mechanical) in the interface of fiber/matrix should be controlled.

It has been demonstrated that hybridization of two or more different types of fibers in lengths, diameters, elastic modulus or tensile strengths can produce composites with better strain hardening behavior, improved ultimate strength and strain capacity than mono fiber containing composite [11–15]. The hybrid-fiber composite derives benefits from each of the individual fibers and exhibits a synergetic response under mechanical loadings [14,16]. The impact resistance of hybrid-fiber ECC containing steel and polyethylene (PE) fibers have been studied and showed that the ECC panels exhibit lesser damage, improved impact resistance and energy absorption capacity [17]. Malej et al. [18] reported high energy absorption capacity and resistant to multiple impacts of hybrid-fiber ECC against the high velocity impact test. The mechanical properties of hybrid-fiber ECC reinforced with PVA fiber and steel fiber has been investigated using impact test [19]. Z. Pan et al. [20] investigated the feasibility of producing cost effective ECC using unoiled PVA fibers and hybrid fibers in ECC. They found that unoiled PVA fibers which have lower cost can be used in hybrid with oiled PVA fiber to produce moderate cost ECC. This cost effective has shown relatively high tensile strength and tensile ductility in comparison to higher cost mono fiber ECC.

In this paper especially focused is placed on improving the ductility of PVA-ECC using hybridization with fibers which having different cross sectional shape and modulus of elasticity. As mentioned before, most of the studies have tried to enhance strain hardening performance of ECCs using different treatments either for fiber, matrix and interface. The hybridization methods used in this experimental investigation are proven that ECCs does not require any extra treatment on PVA fiber to attain higher ductility.

2. Experimental

The experimental program is divided into two parts. In the first part, the flexural behavior of ECC containing three different types of PVA fibers in diameter and length is studied. In the second part the hybridization effect of ECC samples containing different fibers in modulus and shape with selected PVA fiber from first part of this study were investigated using flexural test. The total fiber volume fraction in hybrid composite was 2% and 20% of selected PVA fiber was replaced with other fiber types.

2.1. Materials

Three different types of PVA fibers in length and diameter were selected. The PP fibers with two different cross-sectional shapes including circular and triangular was used. The physical/mechanical characterizations of the fibers used in this study is presented in Table 1. Fibers added to the matrix were 2% of the total composite volume.

An ordinary Portland cement type I was used in this research (Table 2). The aggregate type was ordinary silica sand with the maximum aggregate sizes of 200 micron. The mix proportions of the cementitious matrix used in this study are given in Table 3.

The design of ECC compositions is given in Table 4. Eight mixes were prepared with different fiber types and hybrid ratios.

2.1.1. Production of test specimens

At first solid ingredients including cement, fly-ash, and aggregate were poured into a mixer and mixed. Then water and superplasticizer were added into the dry mixture and mixed to produce consistent and uniform slurry. The fibers were slowly added to the cement paste mix by hand. The composite were casted in

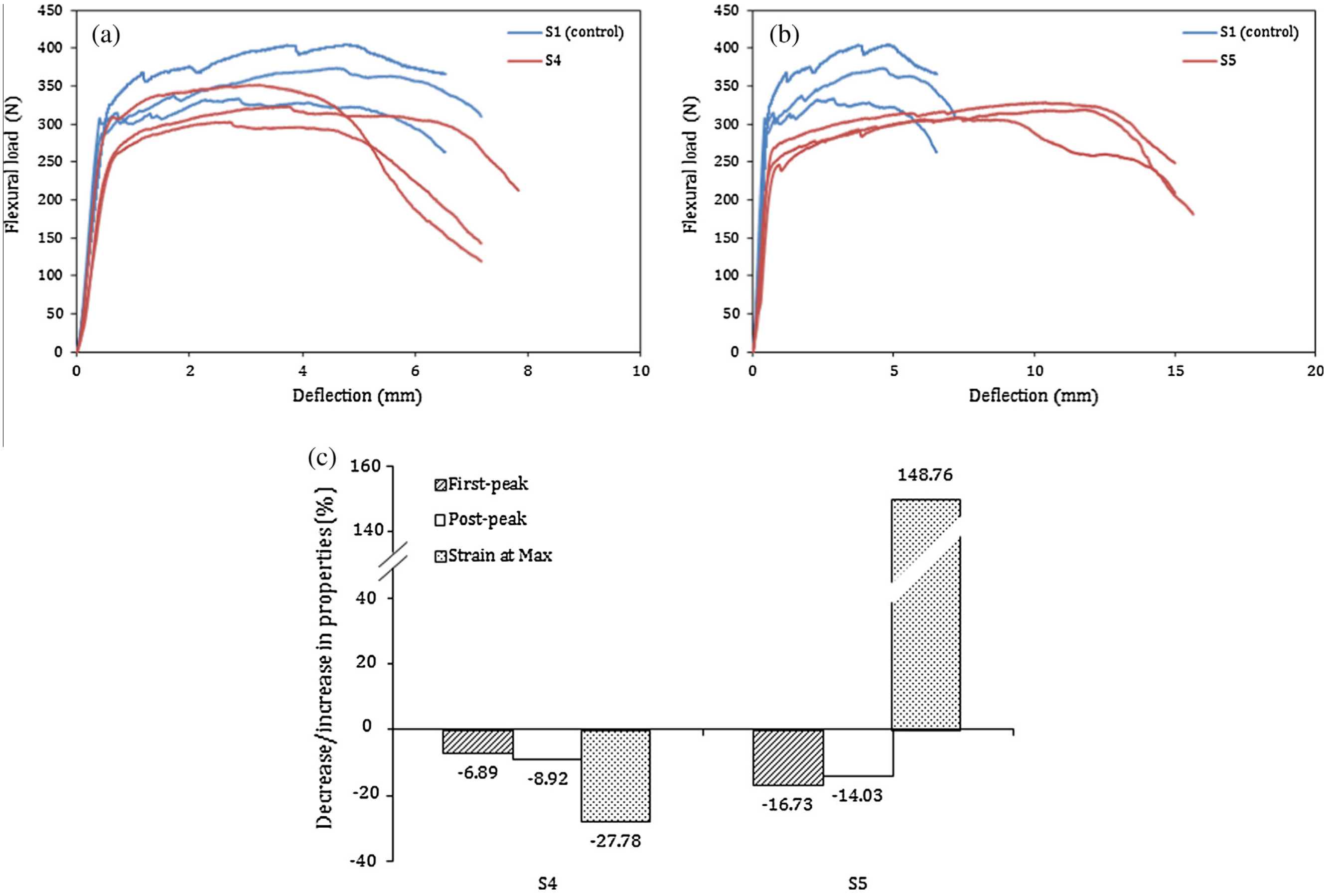


Fig. 5. Flexural load-deflection curve of composite containing; a) S1 vs S4, b) S1 vs S5, and c) Decrease/increase in the properties respect to the S1.

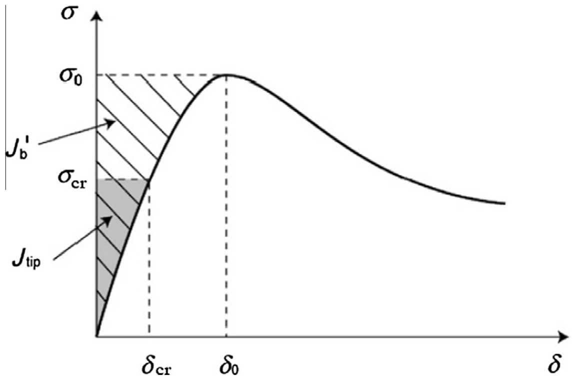


Fig. 6. Complementary energy J'_b (The hatched area) and crack tip fracture toughness J_{tip} (shaded area) of fiber reinforced composites.[10]

plastic molds and vibration was employed to remove trapped air bubbles. Specimens were demolded after 2 days and were immersed in water until testing at 28 days of curing.

2.2. Methods

2.2.1. FTIR-ATR analysis

Fourier Transform Infra-Red-Attenuated Total Reflection (FTIR-ATR) analysis has been used to analyze the surface chemical composition of the PVA fibers. The analysis was performed using the Nexus 670 FTIR spectrometer (from Nicolet, USA).

2.2.2. Flexural strength test

Flexural behavior of ECC samples was carried out using a three-point bending test by a SANTAM Universal testing Instrument according to the EN 12467 standard. The dimension of specimens was 230 mm × 100 mm× 9 mm. The test was performed under displacement control at a loading rate of 0.083 mm/s. The span length of the flexural loading was 200 mm. During the flexural tests, load and mid-span deflection were recorded.

Table 6
Toughness ratio of ECC samples.

Sample type	J'_b/J_{tip} ratio
S1 (control)	2.83
S2	2.61
S3	3.64
S4	1.91
S5	3.95

2.2.3. Compressive strength

Three 5 × 5 × 5 cm^3 cubic specimens were produced to determine compressive strength of ECC mixtures at the age of 28 days according to the ASTM C39-94.

3. Results and discussion

3.1. IR spectra analysis

The used PVA fibers were characterized for the chemical structure by IR spectroscopy. The PVA has a relatively simple chemical structure as shown in Fig. 1. PVA polymer is produced by the polymerization of vinyl acetate to poly (vinyl acetate) (PVAc), followed by hydrolysis of PVAc to PVA [21].

According to the Fig. 2, in the IR spectrum, the large bands observed between 3500 and 3200 cm^{-1} are related to PVA's hydroxyl group absorption peak. The vibration band between 3000 and 2800 cm^{-1} is link to the alkyl groups, and the peaks between

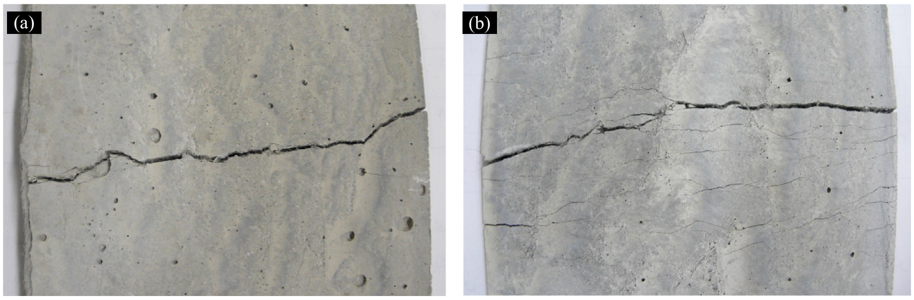


Fig. 7. Micro-cracking pattern of hybrid samples with, a) lower ductility (S4), and b) higher ductility (S5).

1750 and 1690 cm^{-1} are attributed to the acetate group remaining from the cross-linking process. An IR spectrum of a conventional PP fiber is shown in Fig. 3 for comparison. The main chemical group in polymer structure of PP fibers is C–H groups (vibration bond between 2850–3000 cm^{-1} and 1380–1460 cm^{-1}).

The size of –OH stretching vibration peak in PVA's IR spectrum is directly related to the degree of cross-linking. The crystallinity of PVA fiber which is affected by degree of crystallinity is an important structural factor that determines many macroscopic properties of polymer causing an improvement in mechanical properties and increase in the water resistance of PVA [22].

The polarity and hydrophilicity of PVA fiber is strongly depended on the amount of hydroxyl groups in the polymer chain. Fiber with higher contents of —OH group has better hydrophilic properties which can cause a high adhesion with the cementitious materials. In addition, fiber with the higher hydrophilic property and l/d ratio can cause fiber balling and inhomogeneous dispersion during the mixing with cementitious matrix.

It was assumed that higher hydrophilicity has negative effect on the rheology of ECC mixture which limits the amount of incorporating fiber to the mix. In practice, the rheology of cementitious mixture was considerably decreased with incorporation of fiber with greater hydrophilic properties, even at lower volume content.

3.2. Flexural properties

3.2.1. Comparison between different PVA fibers

Flexural behavior of ECC samples containing 1.5 vol.% of different PVA fibers are evaluated which results are given in Table 5.

There is no significant difference in first-cracking strength between ECC samples, while the post-cracking strength of S01 sample containing PVA-C is higher than two other samples. In general, for the studied fibers, the flexural strength of cementitious composite is inconsistent with PVA fiber length. The result of deflection at maximum load shows that the sample S03 (containing PVA-N) has better ductility in comparison to the S01 and S02 samples. This is due to the greater length of PVA-N in comparison with other studied PVA fibers. The PVA fiber with higher fiber length exhibited lower flexural strength but better deformability.

The energy absorption of composites is determined from calculation of area under the flexural load-deflection curves up to deflection of 1.5 mm. The result shows that composite containing long PVA fiber (sample S03) has better energy absorption in comparison to other composites. Due to higher hydrophobicity of PVA-C and fine fiber diameter which produces higher bonding with cementitious matrix, S01 sample exhibited lowest energy absorption and ductility in comparison to S02 and S03 samples.

The flexural strength is directly depended on the hydrophilic nature of PVA fibers as determined using FTIR spectra and l/d ratio. The PVA fiber with higher hydrophilic nature (PVA-C) was produced composite (S01) with higher flexural strength. Since the flexural strength of composite increased by increase in the amount of hydroxyl groups in chemical structure of fiber, a reduction on the strain capacity was obtained.

For the second part of experimental program, PVA-N fiber was selected for main fiber component in hybrid ECC samples due to its better stiffness and greater fiber length. In the hybrid fiber reinforced concrete, the stiffer fiber is responsible for the increase in the bulk strength and toughness, while the flexible fibers are responsible for improvement in the ductility of the composite [19]. The hybrid ECC samples with 2 vol.% of fiber were prepared in PVA-N/(PVA or PP) ratio of 100/0% and 80/20% and investigated under three-point bending test.

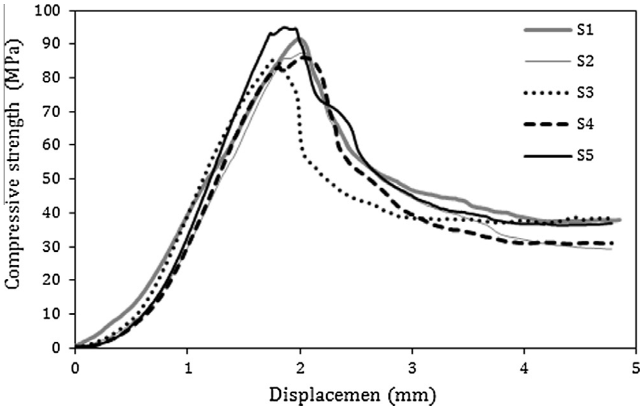


Fig. 8. Compressive behavior results of hybrid composite.

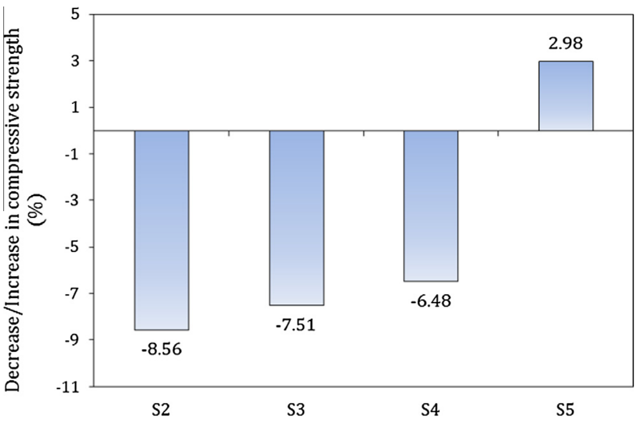


Fig. 9. Decrease/increase in the compressive strength of hybrid composite in respect to the control sample.

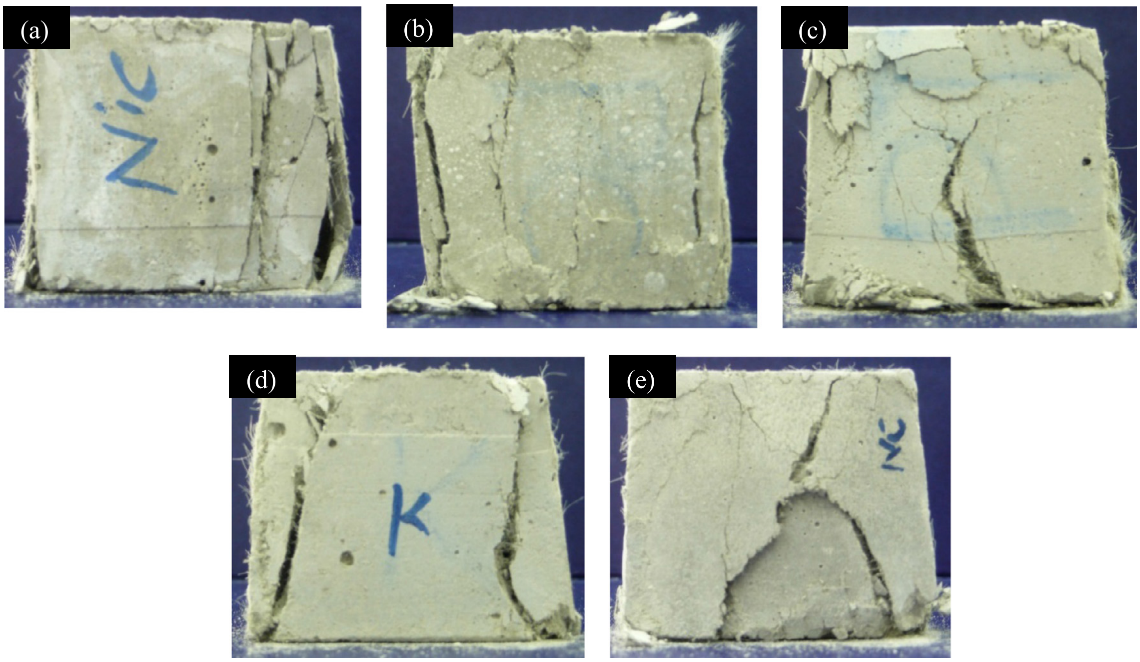


Fig. 10. Pattern of composite failure under pressure; a) S1 (control), b) S2, c) S3, d) S4, e) S5.

3.2.2. Effect of hybridization

3.2.2.1. Hybridization of PVA-N with Low modulus fibers. In order to understand the effectiveness of fiber hybridization, the flexural load-deflection curves of composites containing hybrid versus mono fibers produced at 2 vol.% fiber are plotted. Low modulus fibers in two different cross-sectional shapes including circular and triangular are studied. The effect of this hybridization is presented in Fig. 4. All samples exhibited strain-hardening behavior under three-point bending load. Fig. 4a shows the effect of PVA fiber replacement with a portion of circular PP on the flexural behavior of composite. The hybrid S2 sample shows lower flexural strength which is attributed to the lower PVA fiber content in composite. The flexural load-deflection curve in Fig. 4a indicated that there is no improvement on the deformability of the hybrid sample with circular PP fibers.

The flexural load-deflection behavior of hybrid composite containing triangular PP fiber is shown in Fig. 4b The flexural strength of composite was decreased by replacement with low modulus triangular PP fiber. The flexural load-deflection curve exhibited an improvement on the deformability of hybrid composite compared to the mono fiber reinforced composite sample.

The decrease or increase in the flexural test results of S2 and S3 hybrid samples in respect to the S1 control sample is shown in Fig. 4c. In the flexural stress-strain curve, the maximum flexural stress is defined as the flexural strength (Post-peak strength), and the corresponding strain is defined as the flexural strain capacity of composite [23]. As can be seen, the reduction in the strength of the hybrid composites containing PP-C and PP-T is 36% and 33% in average, respectively. The post-peak strength is more sensitive to the amount of PVA fibers in hybrid composite in comparison with first-peak strength.

On the basis of the results, hybridization with PP fiber that has circular cross section (PP-C) did not improve the strain capacity of composites, whereas it is significantly improved by incorporation of non-round PP fiber (PP-T) up to 33%. It could be concluded that the deformability of hybrid composite is depended on the cross-sectional shape of replacing flexible fibers regardless of their tensile strength and modulus of elasticity. Since, PVA-N fiber which is stiffer may provide reasonable strength of composite, while flexible fibers leading to improved toughness in the post-cracking area [24]. It should be noted that the increase in the PP-T fiber length up to 12 mm may further increase the strain capacity of hybrid composite [25].

3.2.2.2. Hybridization with High modulus fibers. The flexural load-deflection behavior of hybrid composites with high modulus fiber is demonstrated in Fig. 5. The PVA-N fiber is replaced by 20% of PVA-C and PVA-K in volume of composites. The results of flexural test in Fig. 5a and b indicated that the flexural strength of hybrid samples (S4 and S5) is decreased in comparison with the mono fiber composite sample. The flexural load-deflection curve shown in Fig. 5a indicated that the hybridization with PVA-C has not improved the strain capacity of ECC sample. It can be seen that the strain capacity of hybrid S5 sample (1.6% PVA-N + 0.4% PVA-K) remarkably is increased compared with the mono fiber composite sample as shown in Fig. 5b.

The decrease or increase in the flexural test results of S4 and S5 hybrid samples in respect to the S1 control sample is shown in Fig. 5c. According to the results first-cracking and post-cracking strength of hybrid samples are decreased in comparison to the control sample. This reduction in flexural strength is greater for S5 sample than S4 sample.

The strain capacity of S4 sample decreased up to 27% compared to the S1 sample, while S5 sample shown an increase up to 148%. Decrease in the strain capacity of S4 should be attributed to the higher chemical bonding of PVA-C to the cementitious matrix and its fineness leading non-uniform distribution of fibers in mixture.

Remarkably increased strain capacity of S5 may be related to the synergetic effect induced from difference in elastic modulus of hybrid fibers and chemical bonding of PVA-K with matrix. PVA-N fibers which have higher modulus of elasticity provides crack bridging in early stages of loading and strength of composite, while PVA-K due to the lower modulus of elasticity compared to the PVA-N can resist to further crack propagation and enhances deformability of composite in post-cracking zone. The PVA-K which has moderate chemical bonding with cementitious matrix can effectively improve the resistance against crack opening in post-peak area.

From the above results it can be concluded that both mechanical interaction and modulus of elasticity have positive effect on the ductility of hybrid composites related to the used fiber type. Although, hybridization with PVA-K shown better performance in deformability of composite, but rheology of mixture is still a main concern. Using greater length of flexible fiber (i.e. up to 12 mm) can be further improved the strain capacity of hybrid composite.

On the basis of micromechanical principles, the satisfaction of two conditions is necessary to achieve strain-hardening and multiple cracking behaviors of composites [26]. These conditions are expressed as strength and energy terms. The strength criterion states that the tensile first-crack strength σ_{cr} must be below the maximum bridging stress of the fibers σ_0 crossing initiated crack [2], as expressed in Eq. (1)

$$\sigma_0 \geqslant \sigma_{cr} \tag{1}$$

The energy criterion states that the crack tip toughness J_{tip} (km/Em) should be less than the complementary energy J'_b, calculated from the bridging stress vs. crack opening curve [26], as illustrated in Fig. 6. To ensure flat crack propagation mode, J'_b must exceed J_{tip}:

$$J'_b > J_{tip} \tag{2}$$

A high ratio of $J'_b > J_{tip}$ is necessary for robust multiple cracking in the fiber reinforced cementitious composites [27]. On the basis of experiences it was indicated that to achieve saturated multiple cracking a ratio of J'_b/J_{tip} (toughness ratio) equal to or greater than 3 is necessary [11].

The research done by Y. Ding [28], indicated that there is a linear relation between the mid-span deflection and the crack opening for FRC. Therefore, in this study the load-deflection curve was employed to calculate the toughness ratio for better understanding of hybrid fibers effects on strain hardening and multiple cracking behaviors of composites, by considering the same test conditions for all samples. Complementary energy (J'_b) and crack tip toughness (J_{tip}) are determined from the hatched area and shaded area represented in Fig. 6.

The calculated J'_b/J_{tip} ratios for all samples are given in Table 6. The toughness ratio for control sample (S1) is below 3 and this composite can hardly show multiple cracking behavior. Application of PP-C fibers as replacement a portion of PVA-N fibers did not improve the toughness ratio of composite.

It can be seen that the hybridization with PVA-C fiber (S4) which have a high adhesion with cementitious matrix decreased the J'_b/J_{tip} ratio of composite. A high chemical bond limits debonding of fiber from matrix and suppresses the J'_b value [2]. Both S3 and S5 hybrid samples have a toughness ratio greater than 3, which indicated their multiple cracking potential.

The PP-T fibers with non-round cross-sectional shape may enhance the mechanical bonding in fiber/matrix interface by increase in the specific lateral surface. This type of fibers provides the frictional bond during the fiber slippage that increases the toughness ratio. Mechanical bonding can maintain load carrying capacity across the multiple cracks and satisfy multiple-cracking behavior.

The cracking pattern of composite with low (S4) and high (S5) toughness ratio is shown in Fig. 7. The number of micro-cracks on composite surface with toughness ratio below 3 is very less than another sample. This behavior reveals better energy absorption ability and deformability of S5 sample in comparison with the S1 and S4 samples.

3.3. Compressive strength

The compressive behavior curves of ECC mixtures are shown in Fig. 8. The average compressive strength of all samples lies between 85 MPa and 95 MPa. It is evident that the compressive strength of hybrid ECC with low modulus PP fibers (S2 and S3) decreases slightly because of the low bonding strength of these fibers to cement matrix. Hybridization with PVAK fibers (sample S4) also decreased the compressive strength in comparison to the control sample (S1).

The percentage decrease or increase in the compressive strength of all mixtures is plotted in Fig. 9 for comparison. Hybridization with PP-T (S3) and PP-C (S2) resulted in a decrease in compressive strength up to 7.5% and 8.5%, respectively. The decrease in compressive strength of S4 could be attributed to the non uniform distribution of PVA-C fibers in the matrix.

The failure pattern of the samples after compressive test is shown in Fig. 10. It can be seen that the failure mode of hybrid specimens (S2, S3 and S5) is more ductile compared to control sample (S1). The fine and distributed cracks are formed on the surface of these hybrid samples. In contrast, S4 specimens failed with the separation of larger parts of lateral surface as shown in Fig. 10d. However, for all ECC samples the fragments are still attached using fiber bridging mechanism.

4. Conclusion

In this study hybrid fiber with high and low modulus polymeric fibers are used to investigate the importance of fiber cross-sectional shape and modulus of elasticity. On the basis of results, it was concluded that PP fiber with non-round cross-sectional shape increased ductility of ECC up to 33%, regardless of its low strength and modulus of elasticity. However, PP-T fibers due to the lack of chemical adhesion and lower modulus of elasticity reduced flexural strength of the hybrid composite. It was found that hybridization with fibers which have high chemical bonding to the cement matrix have shown an adverse effect on the mechanical behavior of resultant ECC and significantly reduced both workability of mixture and ductility. Furthermore, the synergetic effect of hybridization with PVA-K fibers with lower modulus of elasticity caused a slight decrease in strength, but further improvement in ductility of hybrid ECC up to 148%. All hybrid samples had compressive strength of 85 MPa–95 MPa. Cracking pattern of hybrid samples showed that S2, S3 and S5 are more ductile in comparison to control sample.

Finally, this research demonstrated that hybridization with fibers which have different physical/mechanical properties can be very effective in significantly modifying ductility of ECC materials.

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