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**Mixture Optimization on internally Cured High Strength Engineered Cementitious Composite with pre-wetted Sand-like Zeolite**

Jun Zhang¹, Xuan Zheng² and Qing Wang²

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Abstract

In this paper, mixture optimization on internally cured high strength engineered cementitious composite (HSECC) with pre-wetted sand-like zeolite is conducted based on shrinkage and mechanical performance tests. Experimental study on the effect of the replacing ratio of the cement with the calcined zeolite on matrix compressive and bending strength was carried out first. Based on above test results, four optimized mixtures with the zeolite addition of 144, 194, 244 and 294 kg/m³ were obtained. Shrinkage and uniaxial tensile tests on the four high strength matrix reinforced with 2% PVA and 1% steel fibers were conducted. Through mixture optimization, strength of cement matrix with the calcined zeolite addition is further improved. The optimized replacing ratio of cement with the zeolite in the matrix is existed to obtain the highest compressive and bending strength. Shrinkage the HSECC with optimized addition of the calcined zeolite is significantly reduced. The highest reduction rate on shrinkage of the composite comparing to the reference achieves 78%. Tensile performance with optimized addition of the calcined zeolite is improved comparing with the reference mixture as well. Four optimized mixtures of HSECC are obtained with cracking and tensile strength, ultimate tensile strain and average crack width at tensile strength achieving of 4.31 - 5.48 MPa and 6.36 - 7.27 MPa, 1.01 - 1.27% and 18 - 24 μm.

1. Introduction

Engineered Cementitious Composite (ECC) is a class of fiber reinforced cement-based composite that has a unique property of strain-hardening accompanied by multiple cracking (Li 2002). In order to obtain this strain-hardening and multiple cracking performance, only a small amount of fine sand is allowed to be used in the matrix to control the fracture toughness of matrix (Li 1993; Li et al. 1995; Zhang et al. 2002; Zhang et al. 2009). Therefore, the shrinkage, including autogenous and drying shrinkage, is much greater than that of normal concrete due to its high cementitious materials content. Meanwhile, to maintain the multiple cracking behaviors, relatively low strength of the cement matrix was used in ECC generally (Li et al. 1995; Zhang et al. 2002; Zhang et al. 2009). However, some applications, such as permanent formwork for concrete structures, steel-concrete composite bridge decks and joint-less concrete pavements, still wish the material has a relatively higher strength, meanwhile remaining adequate ductility as long as satisfying the deformation requirements resulting from temperature change, shrinkage and mechanical loading (Shao et al. 2013; Zhang et al. 2013; Zhang et al. 2017). Recently, ECC with compressive strength around 80 - 100 MPa (HSECC) under normal curing condition had been developed by Zhang et al. (Zhang et al. 2017a). However, the shrinkage of HSECC may be greater than normal strength ECC due to its very low water to binder ratio. The high shrinkage strain may result in cracking at an early age in structures that should shorten the service period without cracking. Most recently, Zhang et al. investigated the shrinkage of HSECC by use of internal curing with sand-like calcined zeolite (Zhang et al. 2017b). Shrinkage of HSECC, including autogenous shrinkage and drying shrinkage was significantly reduced and the cracking load of designed steel-HSECC composite bridge deck was increased. In this study, a single amount of internal curing agent was used in the experimental program. In a recent study regarding the use of calcined zeolite in normal strength concrete for shrinkage reduction (Zhang et al. 2018), it was found that the pre-wetted calcined zeolite in concrete may be used to replace cement without loss of strength. It indicates that it is necessary to optimize the mixture proportion of high strength ECC to obtain better performance in the view of both strength and shrinkage.

The purpose of this paper is to explore the mixture proportion of HSECC with addition of calcined zeolite for shrinkage reduction. In this study, calcined zeolite particles are used as internal curing agent of the matrix. Shrinkage and internal relative humidity of HSECC with different amount of pre-wetted sand-like zeolite addition are measured. Meanwhile, uniaxial tensile tests were conducted as well to make sure the strain-hardening behavior was remained.
2. Experimental program

In the present work, a kind of sand-like zeolite was used as internal water reservoir in the matrix to reduce shrinkage of HSECC. The average particle size of the zeolite is 0.18 mm that was produced in China. Zeolite is aluminosilicate mineral containing relatively large amount of micropores and physically bonded water in it. The zeolite particles undergo 500°C calcination in a muffle furnace for 30 minutes, named Zeolite-500 before used. The calcined zeolite was pre-wetted in water for 3 days before mixing with other components. Figure 1a displays the particle distribution of the calcined zeolite. For comparison, the size distribution of the quartz sand used in the matrix as well is displayed also. The particle size of the zeolite is similar with the quartz sand. The specific surface area of the calcined zeolite and quartz sand is 49 m²/g and 1.2 m²/g respectively. The pore size distribution of the calcined zeolite and quartz sand is displayed in Fig. 1b based on nitrogen adsorption test. As expected, high pore volume is obtained with calcined zeolite than that of the quartz sand although the particle size of the two materials is similar. The pore content of the calcined zeolite and the quartz sand is 0.26 ml/g and 0.002 ml/g respectively according the nitrogen adsorption test.

In the test program, the effect of calcined zeolite addition on compressive and bending strength of high strength cement matrix was investigated first. Then, the optimized composition of the matrix with the calcined zeolite addition was used as a high strength ECC matrix to make HSECC with needed fiber reinforcements. The shrinkage and tensile performance of the composite with the optimized matrix should be evaluated by shrinkage and uniaxial tensile tests. To obtain the optimized proportions of the matrix with pre-wetted calcined zeolite addition, four additive amounts of the zeolite, 144, 194, 244 and 294 kg/m³, were used. An optimized cementitious binder composition of 70% cement, 10% silica fume (SF), 10% grounded granulated blast furnace slag (SG), 10% fly ash (FA) in weight, which achieving the highest compressive and bending strength of matrix under given water to binder ratio and normal curing condition (Zhang et al., 2016), was used in the tests as basic mixture proportion. Calcium sulfoaluminate cement (SAC), that possesses extra high early-age strength (Zhang et al., 2015), was used as cementing material. Silica sand with particle size of 0.075 to 0.150 mm was used to form the mortar matrix. Constant water to binder ratio (w/b) of 0.2 and constant sand to binder ratio of 0.833 were used in the basic matrix mixture proportion. For each additive of the zeolite, different replacing amount of cement and silica sand with the zeolite was used to study the effect of the replacement on strength of the matrix. In the tests, the replacement of cement with the zeolite in weight of the cement of 0%, 5%, 10%, 15%, 20%, 25% and 30%, were used. The rest of the zeolite, i.e. 100%, 95%, 90%, 85%, 80%, 75% and 70% in weight, was used to replace the sand in the basic mixture. Thus, for each addition of the zeolite, seven mixtures of the cementing binders were formed. Through compressive and bending tests on seven cement mortar with constant sand to binder ratio of 0.833 and water to binder ratio of around 0.2, the optimized replacing strategy for each addition of the zeolite can then be obtained. After that, shrinkage and tensile tests on HSECC using the developed high strength matrix were conducted. A polycarboxylate-based super plasticizer (SP) and citric acid based retarding admixture (RT) were used in the mixtures to guarantee all fresh mortars having similar flow ability, as well as setting performance after mixing. Flow ability of fresh mortar or HSECC was assessed by the spreading test with small slump cone showed in Fig. 2.

2.1 Materials

Chemical compositions of calcium sulfoaluminate cement and the three supplementary cementitious materials (SF, SG, FA) used in tests are listed in Table 1. As state previously, four series tests focus on the effect of cal-
Fig. 2 Equipment of spreading test on fresh matrix or HSECC, unit: mm.

Table 1 Chemical composition of the cementitious materials used in tests. (wt\%)

| No. | SiO$_2$ | CaO | Al$_2$O$_3$ | Fe$_2$O$_3$ | MgO | K$_2$O | Na$_2$O | SO$_3$ | LOI |
|-----|---------|-----|-------------|-------------|-----|-------|--------|------|-----|
| SAC | 6.19    | 42.9| 24.16       | 1.04        | 1.96| 0.48  | 0.08   | 12.91| 8.91|
| Silica fume (SF) | 90.56 | 0.81 | 0.41 | 0.52 | 0.95 | 1.59 | 0.63 | - | 3.72 |
| Fly ash (FA) | 47.02 | 5.08 | 35.06 | 3.88 | 1.36 | 1.30 | 1.18 | 0.89 | 1.85 |
| Slag (SG) | 38.83 | 38.70 | 12.92 | 1.46 | 4.63 | 0.37 | 0.28 | 0.60 | 0.06 |

Table 2 Properties of PVA and steel fibers.

|                | Density (g/cm$^3$) | Tensile strength (MPa) | E (GPa) | Diameter (mm) | Length (mm) |
|----------------|--------------------|------------------------|---------|---------------|-------------|
| PVA fiber      | 1.2                | 1620                   | 42.8    | 0.039         | 12          |
| Steel fiber    | 7.8                | 2850                   | 210.0   | 0.12          | 10          |

cined zeolite to strength of the matrix were first carried out. The shrinkage, uniaxial tensile and steel ring tests should be conducted based on above test results. Therefore, detailed mix proportions of each kind of tests used will be given in the section of results and discussion in order. The fibers used to check the shrinkage and tensile performance of HSECC with the developed high strength matrix include Polyvinyl alcohol fiber (PVA) supplied by Kuraray company in Japan and steel fiber supplied by Changhong company in China. The mechanical properties of the fibers are listed in Table 2. In the mix design of ECC, 2% volume fraction of the PVA fibers and 1% volume fractions of the steel fibers were applied.

2.2 Specimens, casting, curing and testing procedures

For mixture optimizing tests, rectangular specimen with size of 40 × 40 × 160 mm was used to conduct the compressive and bending tests for each mixture at age of 1, 3, 7 and 28 days after casting. The molds used to cast the specimens were made of steel. After removing from their molds, the specimens were stored in water at 20 ± 2°C for curing until compressive and bending tests were conducted. For shrinkage tests, mold made by plexiglass with inner dimensions of 100 × 100 × 400 mm was used. Two plexiglass plates with dimensions of 20 × 100 × 400 mm were used as recoverable filler respectively along the two long inside sides of the mode. Thus, the actual specimen size is 60 × 100 × 400 mm. After the initial set of the composite, the removable plastic sheets were lifted to create the “free restraint” conditions and the space for specimen drying. In this study, the humidity at the center of the test specimen were measured. Resistance based digital sensor that can measure humidity was used. The measuring accuracy of relative humidity and temperature is 2%. The measuring method of internal relative humidity is the same as used in (Zhang et al. 2010). The deformation was measured by two linear variable displacement transducers (LVDT) mounted on the two long ends of the specimen as used in (Zhang et al. 2010). The measuring range of the LVDT is 2 mm and the measuring accuracy is 1 μm. Figure 3a shows the overall schematic diagram of the humidity and deformation measurement set-up. Figure 3b is a photo showing the two parallel specimens under testing. For the uniaxial tensile test, rectangular coupon specimens with size of 100 × 200 × 20 mm were used. The same curing procedures were used for tensile specimens. The tensile specimens were tested in uniaxial tension with displacement control in a 250 kN capacity MTS 810 material testing system with hydraulic wedge grips. Aluminum plates were epoxy glued onto the ends of the specimens prior to loading at least 6 hours to enhance the ends for gripping. The actuator displacement rate used for controlling the test was 0.0025 mm per second. The strain was measured by two extensometers mounted on the surface of the specimen. The measured gage length of extensometer was 50 mm. The tensile test set-up and specimen with aluminum plates glued and extensometers mounted is shown in Fig. 4.

The mixer used was horizontal shaft concrete mixer with capacity of 60 liters. The mixing procedure of the composite material consists of the following steps: (1) First, the cementitious material and silica sand were mixed together for 2 minutes at low speed. Then water with superplasticizer and pre-mixed zeolite-water slurry (additional water calculated by the pore content of the zeolite was added) was gradually added, and mixing was continued for 2 minutes which result in a uniform fluid matrix. Within this period, the bottom of the mixing bowl should be scraped manually to ensure that no solid materials stick to the bottom. After such scraping, the matrix was mixed at a higher speed for 1 minute. If fibers were added, all fibers should be added after the above preparation of matrix. The fibers were gradually spread into the mixer by hand as the matrix was mixed at a slow speed. The fibers must be added slowly to ensure uniform distribution with no fibers bundled together. Before
casting, workability of fresh material was assessed by the spreading test with small slump cone. In the present study, the spreading diameter of fresh mortar is about 220 - 230 mm in the case without fiber addition and with fiber is about 190 mm. (2) Casting and curing: For strength test specimens, the fresh mortar without or with fibers was carefully cast into the mold in two layers. First about half of the material were placed. Then the mix was vibrated for about 1 - 2 minutes to ensure that the material was well compacted. Next, the second half of the mold was filled by the composite in the same manner. After smoothing the surface, the specimens were covered with a polyethylene sheet to prevent loss of moisture and stored for 24 hours at room temperature prior to de-molding. For shrinkage test specimens, before casting, plastic sheets used to seal the specimen were put into the mold first. In the mean time, the two copper measuring probes were fixed at the designated locations.

Fig. 3 Experimental set-up for humidity and deformation measurement of HSECC under plastic film sealed and drying conditions, (a) schematic illustration, (b) photograph during test.

Fig. 4 Tensile test set-up (a) and specimen with aluminum plates glued and extensometers mounted (b).
Then, the fresh composite was cast into the mold in two layers and was consolidated by a vibrating table. After compacting, the PVC tube, which one end was covered with a plastic sheet glued to the end and two rectangular holes were made at the surface of the tube (Zhang et al., 2010), with the steel bar inserted was put into the concrete at the appointed depth from the casting surface. In order to investigate the effect of calcined zeolite on compressive and bending strength of high strength ECC matrix, four series tests with four additive amounts of the zeolite, 144, 194, 244 and 294 kg/m³ were conducted. All the six series tests with four additive amounts of the zeolite to the cement in weight are displayed in Table 3. The test results of compressive and bending strength of each mixture proportion were cast for each mixture. After 3 days of concrete casting, the plastic film covering the five surfaces of the one of the two specimens was removed. While the other specimen maintains the sealing state to compare with the drying specimen. The tests were continued until 28 days since material casting. All tests were performed in a laboratory under room temperature.

3. Results and discussion

3.1 Effect of calcined zeolite on compressive and bending strength of high strength ECC matrix

To investigate the effect of calcined zeolite on compressive and bending strength of high strength ECC matrix, four series tests with four additive amounts of the zeolite, 144, 194, 244 and 294 kg/m³ were conducted. All the six mixture proportions of each additive amount according to the replacing strategy of the zeolite described in the section of the experimental program are listed in Table 3. The test results of compressive and bending strength of the mortar in terms of strength versus the replacing ratio of the zeolite to the cement in weight are displayed in Figs. 5 to 8 for the four amount of the zeolite addition respectively.

| Mixture No. | Cementitious Materials Total (Cement:SF:SG:FA) | Sand Total (Rep:Cement+Sand) | Water | RT (%) | SP (%) |
|-------------|-----------------------------------------------|-------------------------------|-------|--------|-------|
| SAC+144-0%  | 1 (0.700:0.100:0.100:0.100)                    | 0.708                         | 0.125 | 0.200  | 0.19  |
| SAC+144-5%  | 0.994 (0.694:0.100:0.100:0.100)                 | 0.714                         | 0.125 | 0.200  | 0.19  |
| SAC+144-10% | 0.988 (0.688:0.100:0.100:0.100)                 | 0.721                         | 0.125 | 0.200  | 0.19  |
| SAC+144-15% | 0.981 (0.681:0.100:0.100:0.100)                 | 0.727                         | 0.125 | 0.200  | 0.19  |
| SAC+144-20% | 0.975 (0.675:0.100:0.100:0.100)                 | 0.733                         | 0.125 | 0.200  | 0.19  |
| SAC+144-25% | 0.969 (0.669:0.100:0.100:0.100)                 | 0.739                         | 0.125 | 0.200  | 0.19  |
| SAC+144-30% | 0.963 (0.663:0.100:0.100:0.100)                 | 0.746                         | 0.125 | 0.200  | 0.19  |
| SAC+194-0%  | 1 (0.700:0.100:0.100:0.100)                    | 0.665                         | 0.168 | 0.200  | 0.19  |
| SAC+194-5%  | 0.992 (0.692:0.100:0.100:0.100)                 | 0.673                         | 0.168 | 0.200  | 0.19  |
| SAC+194-10% | 0.985 (0.685:0.100:0.100:0.100)                 | 0.682                         | 0.168 | 0.200  | 0.19  |
| SAC+194-15% | 0.975 (0.675:0.100:0.100:0.100)                 | 0.690                         | 0.168 | 0.200  | 0.19  |
| SAC+194-20% | 0.966 (0.666:0.100:0.100:0.100)                 | 0.699                         | 0.168 | 0.200  | 0.19  |
| SAC+194-25% | 0.958 (0.658:0.100:0.100:0.100)                 | 0.707                         | 0.168 | 0.200  | 0.19  |
| SAC+194-30% | 0.950 (0.650:0.100:0.100:0.100)                 | 0.716                         | 0.168 | 0.200  | 0.19  |
| SAC+244-0%  | 1 (0.700:0.100:0.100:0.100)                    | 0.622                         | 0.212 | 0.200  | 0.19  |
| SAC+244-5%  | 0.989 (0.689:0.100:0.100:0.100)                 | 0.633                         | 0.212 | 0.200  | 0.19  |
| SAC+244-10% | 0.979 (0.679:0.100:0.100:0.100)                 | 0.643                         | 0.212 | 0.200  | 0.19  |
| SAC+244-15% | 0.968 (0.668:0.100:0.100:0.100)                 | 0.654                         | 0.212 | 0.200  | 0.19  |
| SAC+244-20% | 0.958 (0.658:0.100:0.100:0.100)                 | 0.664                         | 0.212 | 0.200  | 0.19  |
| SAC+244-25% | 0.947 (0.647:0.100:0.100:0.100)                 | 0.675                         | 0.212 | 0.200  | 0.19  |
| SAC+244-30% | 0.936 (0.636:0.100:0.100:0.100)                 | 0.686                         | 0.212 | 0.200  | 0.19  |
| SAC+294-0%  | 1 (0.700:0.100:0.100:0.100)                    | 0.578                         | 0.255 | 0.200  | 0.19  |
| SAC+294-5%  | 0.987 (0.687:0.100:0.100:0.100)                 | 0.591                         | 0.255 | 0.200  | 0.19  |
| SAC+294-10% | 0.975 (0.675:0.100:0.100:0.100)                 | 0.604                         | 0.255 | 0.200  | 0.19  |
| SAC+294-15% | 0.962 (0.662:0.100:0.100:0.100)                 | 0.617                         | 0.255 | 0.200  | 0.19  |
| SAC+294-20% | 0.949 (0.649:0.100:0.100:0.100)                 | 0.629                         | 0.255 | 0.200  | 0.19  |
| SAC+294-25% | 0.936 (0.636:0.100:0.100:0.100)                 | 0.642                         | 0.255 | 0.200  | 0.19  |
| SAC+294-30% | 0.924 (0.624:0.100:0.100:0.100)                 | 0.655                         | 0.255 | 0.200  | 0.19  |

1 RT: Retarding admixture in the percentage of cement weight; 2 SP: Plasticizer in the percentage of cement weight
Fig. 5 Effect of replacing ratio on compressive and bending strength of high strength cement matrix with the zeolite addition of 144 kg/m³.

Fig. 6 Effect of replacing ratio on compressive and bending strength of high strength cement matrix with the zeolite addition of 194 kg/m³.

Fig. 7 Effect of replacing ratio on compressive and bending strength of high strength cement matrix with the zeolite addition of 244 kg/m³.
From Table 3, we may first observe that with increase of the replacing ratio of the cement with the zeolite, the amount of superplasticizer used in the mortar is slightly decreased as similar fluidity is maintained. This is understandable because the particle size of the zeolite is larger than cement particles. Second, from the test results shown in Figs. 5 to 8, it is found that the overall trend of compressive and bending strength of the mortar within the range of the replacing ratio used in the tests is first increasing then decreasing with increase of the replacing ratio. Such tendency appears to be enhanced with the increase of age. However, it is noted also that the optimized replacing ratio depends obviously on the amount of the zeolite used in the mortar. According to the present tests, the optimized replacing ratio of cement with the zeolite in the mortar is about 20%, 15%, 10% and 5% of the total zeolite weight to obtain the highest compressive and bending strength for the zeolite addition of 144, 194, 244 and 294 kg/m³ respectively. For example, the compressive strength at 28 days of the matrix changes from 80.4, 79.0, 76.5 and 73.5 MPa (zero replacement) to 88.8, 84.2, 80.8 and 75.9 MPa (optimized replacement) respectively for the zeolite addition of 144, 194, 244 and 294 kg/m³. Obviously, the effect of the replacing ratio of the cement with the zeolite on strength of the high strength matrix is significant. Meanwhile, as expected, the strength is decreased with increase of the total amount of the zeolite. According to above test results, the optimized mixtures named SAC+Z144-20%, SAC+Z194-15%, SAC+Z244-10% and SAC+Z294-5% respectively in Table 3 for the zeolite addition of 144, 194, 244 and 294 kg/m³ were used as high strength ECC matrix in the following shrinkage and uniaxial tensile tests.

### 3.2 Effect of calcined zeolite on shrinkage of HSECC

Mixture proportions of HSECCs used in shrinkage tests are listed in Table 4. To compare with the case without zeolite addition, the referencing mixture (Zhang et al. 2017b) was listed in the table as well. Previously study shows that the optimal additional steel fiber content for the composite with constant PVA fiber content of 2% is about 1% considering its improvement on tensile strength and strain, as well as the ability of crack opening control (Zhang et al. 2017a). Therefore, 2% PVA fiber and 1% steel fiber were used in the composites. From Table 4, it can be seen that as PVA and steel fiber were...
added, the amount of superplasticizer used is little more than that used in the pure matrix without fibers, to obtain a reasonable workability for fiber distribution. The compressive strength of the composite measured by cube specimens with the dimension of 100 × 100 × 100 mm of the six mixtures of HSECC at 28 days are listed in Table 5. From compressive test results, it can be observed that the compressive strength at 28 days of SAC+Z144 and SAC+Z194 are comparable to that of reference mixture and the rest two mixtures, SAC+Z244 and SAC+Z294, present a lower compressive strength at 28 days than that of reference. A similar trend is observed in the pure matrix tests.

From shrinkage test, the progress of deformation of the specimen along the long direction and internal relative humidity at the center of the sample can be recorded. Afterward, the complete shrinkage strain versus age diagram since set of the composite can be obtained. In the present study, the setting point of the composite is determined according to the deformation characteristic before and after setting, i.e. the end of the initial swelling of the specimen is defined as the setting point of the composite (Zhang et al. 2010). Figures 9 to 13 present the shrinkage-age and internal relative humidity-age diagrams since specimen casting of all the four mixtures with the zeolite addition and the reference mixture under sealing and drying conditions respectively. From the test results, we can observe that the progress of internal relative humidity of HSECC since specimen cast follows two-stage mode that had been found in concrete already (Zhang et al. 2010; 2013): a water-vapor saturated stage with 100% relative humidity (stage I) followed by a stage the relative humidity is gradually decreasing (stage II). The shrinkage-age diagram follows a two-stage mode also: First develops at a fast speed within the first day after casting and then goes into a slow growing stage afterwards. The development of shrinkage and internal relative humidity of HSECC under both sealing and drying conditions are greatly influenced by the addition of the zeolite particles. The shrinkage strain and relative humidity at 28 days after casting under sealing and drying conditions of all the five mixtures are summarized in Table 5.

First, look at the test results of the reference mixture, SAC-OC, displayed in Fig. 9. The reduction of internal relative humidity in early-age HSECC due to cement hydration is extremely obvious. Surface drying further decreases the internal relative humidity. However, the difference on relative humidity between sealing and drying samples is relatively small due to the very low water to binder ratio and relatively higher water consumption of cement hydration. The amount of water left for diffusion is less. The relative humidity at the center of the test sample of HSECC at 28 days after casting is 59.7% and 52.9% respectively under sealing and drying condition, which is even lower than that of 100 MPa concrete (Zhang et al. 2016). Meanwhile, large shrinkage strain is developed within the first day after casting even that the internal relative humidity still remains at 100% in this period. After this period, the shrinkage is further growing at a relatively slow speed. At 28 days after casting, the shrinkage strain is 1370 μm/m and 1481 μm/m respectively under sealing and drying status. Clearly, more than 90% of the shrinkage strain at 28 days

| Mixture no. | Compressive strength at 28 days (MPa) | Shrinkage at 28 days, Seal (μm/m) | Shrinkage at 28 days, Dry (μm/m) | RH at 28 days, Seal (%) | RH at 28 days, Dry (%) |
|------------|--------------------------------------|----------------------------------|-------------------------------|-----------------------|-----------------------|
| SAC-OC     | 88.1                                 | 1370                             | 1481                          | 59.7                  | 52.9                  |
| SAC+Z144   | 87.9                                 | 670                              | 715                           | 86.9                  | 73.5                  |
| SAC+Z194   | 83.4                                 | 458                              | 509                           | 88.4                  | 76.7                  |
| SAC+Z244   | 79.5                                 | 357                              | 456                           | 90.6                  | 79.1                  |
| SAC+Z294   | 73.2                                 | 292                              | 368                           | 92.0                  | 84.6                  |

Fig. 9 Development of shrinkage and internal relative humidity of SAC-OC.

Fig. 10 Development of shrinkage and internal relative humidity of SAC-Z144.
are developed within the first few days after casting. This may be considered as an important shrinkage characteristic of the present HSECC with calcium sulfoaluminate cement.

As pre-wetted calcined zeolite particles (Zeolite-500) were used in the matrix, the moisture status inside of the composite was improved compared to the reference mixture (SAC-OC). The increment on internal relative humidity is dependent on the amount of internal water induced in the matrix, which is reflected by the amount of the zeolite used. For a given age, the higher the zeolite used, the higher the relative humidity. The internal relative humidity at 28 days under sealing and drying conditions of SAC-OC, SAC+Z144, SAC+Z194, SAC-244, SAC-294 is 59.7% and 52.9%, 86.9% and 73.5%, 88.4% and 76.7%, 90.6% and 79.1%, 92.0% and 84.6% respectively. Accordingly, shrinkage strain at 28 days is 1370 and 1481 μm/m, 670 and 715 μm/m, 458 and 509 μm/m, 357 and 456 μm/m, 292 and 368 μm/m respectively for SAC-OC, SAC+Z144, SAC+Z194, SAC-244, SAC-294 under sealing and drying condition. The shrinkage reduction rate comparing to the reference mixture (OC) of the internally cured HSECC achieves 51% to 78%. Based on the present study, the shrinkage strain of HSECC is comparable or even lower than that of the concrete with similar compressive strength (Zhang et al. 2016). Obviously, efficiency on shrinkage reduction of Zeolite-500 is significant. Certainly, as the addition of the zeolite particles up to 294 kg/m³, the negative effect of porous particles on strength of the composite becomes relatively obvious, for instance the compressive strength at 28 days of SAC-OC and SAC+Z294 is 85.1 MPa and 73.2 MPa. The strength reduction due to the use of the zeolite particles may be overcome by adding some additional fine particles, such as fine glass powder (Soliman and Taginit 2017; Wang et al. 2018). However, by contrast, a few techniques are available to reduce shrinkage of high strength cementitious matrix at present.

3.3 Tensile performances of HSECC with the calcined zeolite addition

To study the tensile performance of the HSECCs with the calcined zeolite addition, uniaxial tensile tests on the composites at 7 and 28 days after specimen casting were conducted. Figure 14 displays a typical tensile stress-strain curve of the composite, in which Fig. 14a shows the overall curve and Fig. 14b shows the initial portion of the curve. Three sections followed with different tensile parameters in each section can be defined: First, stress and strain linearly goes up to first cracking occurring. The point of first cracking is corresponding to the end of the linear section of the stress-strain curve. The corresponded stress at first cracking is defined as cracking strength \( \sigma_{fc} \), and the corresponding strain is defined as the cracking strain \( \varepsilon_{fc} \). Second, strain hardening stage: tensile stress continues to increase with strain, resulting in strain-hardening accompanied by multiple cracking. The maximum stress is the tensile strength \( \sigma_t \) and the corresponded strain is ultimate tensile strain \( \varepsilon_t \). Third, with increase of tensile strain, a single crack localizes and the load slowly drops with increase of deformation. Thus, the overall tensile performance of HSECC can be characterized by two pair parameters of \( \sigma_{fc} \) and \( \varepsilon_{fc} \), \( \sigma_t \) and \( \varepsilon_t \). In addition, to analyze the impact of the zeolite addition on individual crack width of the composite in the strain-hardening and multiple cracking stage, the average crack opening at the point of tensile
strength achieving can is calculated according to tensile test results. In tensile tests, if the number of cracks \( n \) within the range of measuring length \( L \) at any given tensile strain \( \varepsilon \) and stress capacity \( \sigma \) is know, the average crack width \( w_a \) of individual crack can be expressed as (Zhang et al. 2017a):

\[
   w_a = \frac{L(\varepsilon - \sigma / E)}{n}
\]

The number of cracks within the length \( L \) can be counted after the test. Therefore, the average crack width at the point of tensile strength achieved \( w_{am} \), which is the maximum crack width theoretically before crack localization in the composite, can be estimated with the ultimate tensile strain \( \varepsilon_t \) and strength \( \sigma_t \) through:

\[
   w_{am} = \frac{L(\varepsilon_t - \sigma_t / E)}{n}
\]

Based on tensile test results, the corresponding first cracking strength and strain, the ultimate tensile strength and strain, elastic modulus and maximum crack width at tensile strength achieving of each mixture are listed in Table 6. Figures 15 to 19 display the tensile stress-strain diagrams of SAC-OC, SAC+Z144, SAC+Z194, SAC+Z244 and SAC+Z294 respectively at the age of 7 and 28 days after specimen casting. From the test results, first it can be found as high matrix is used, ultimate tensile strain is obviously decreased comparing with that as low strength matrix is used in the composite (Li 2002; Li 1993; Li et al. 1995; Zhang et al. 2002; Zhang and Li 2002; Zhang et al. 2009). For example, for the same PVA fiber content, 3% tensile strain may be achieved for ECC with matrix strength lower than 50 MPa. In present tests, only about 1% ultimate tensile strain is obtained for HSECCs. This is understandable because an inverse relationship between strain-hardening degree and matrix strength is exist according the micro-mechanics principles of ECC (Li 2002). High strength matrix will significantly increase the fracture toughness and first cracking strength of the composite, while less improvement on fiber bridging stress. Therefore, simply increasing matrix strength will decrease the potential for strain-hardening of the composite. Second, as pre-wetted calcined zeolite particles were used, for the same fiber content, the tensile performance is improved comparing with the reference mixture of SAC-OC. Cracking and tensile strength at 28 days is changed from 4.72 and 5.57 MPa to 5.48 and 7.27 MPa. Ultimate tensile strain at 28 days is changed from 0.99% to 1.27%. The zeolite particles may act as artificial flaws to improve tensile cracking behavior of the composites, which was described by Wang and Li in design of lightweight ECC (Wang and Li 2004). In addition, tensile stress-strain profiles obtained from tensile tests are different between four optimized mixes. For example, L-SAC-OC,

| Age(days) | Mixture no. | \( \sigma_t (\text{M} \text{Pa}) \) | \( \varepsilon_t (\%) \) | \( E (\text{T} \text{Pa}) \) | \( \sigma_t (\text{M} \text{Pa}) \) | \( \varepsilon_t (\%) \) | \( w_{am} (\text{mm}) \) |
|-----------|-------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 7 days    | SAC-OC      | 3.00            | 0.011           | 27.99           | 4.74            | 1.04            | 0.032           |
|           | SAC+Z144    | 4.19            | 0.013           | 29.96           | 5.87            | 1.10            | 0.030           |
|           | SAC+Z194    | 4.01            | 0.013           | 29.46           | 5.24            | 1.19            | 0.029           |
|           | SAC+Z244    | 3.80            | 0.013           | 28.93           | 5.09            | 1.28            | 0.026           |
|           | SAC-Z294    | 3.41            | 0.012           | 28.09           | 4.88            | 1.39            | 0.024           |
| 28 days   | SAC-OC      | 4.72            | 0.016           | 29.87           | 5.57            | 0.99            | 0.025           |
|           | SAC+Z144    | 5.48            | 0.017           | 31.37           | 7.27            | 1.01            | 0.024           |
|           | SAC+Z194    | 5.20            | 0.016           | 30.81           | 6.99            | 1.09            | 0.022           |
|           | SAC+Z244    | 4.89            | 0.016           | 30.05           | 6.71            | 1.18            | 0.020           |
|           | SAC-Z294    | 4.31            | 0.015           | 29.59           | 6.36            | 1.27            | 0.018           |

**Table 6** Tensile properties of internally cured HSECC.
SAC+Z194 and SAC+Z244 at 28 days show smooth profile without stress fluctuation at cracking. However, some others have significant fluctuation. The stress fluctuation at cracking should be due to hysteresis of fiber bridging at cracking. Initial elastic bonding strength between fiber and matrix may be probably responsible for this phenomenon. Therefore, a reasonable bond between fiber and matrix may be achieved for L-SAC-OC, SAC+Z194 and SAC+Z244 at 28 may be achieved. Further investigated on the optimized elastic bond between fiber and matrix, especially the bond of steel fiber and matrix may be needed. Furthermore, first cracking strength of SAC+Z144 at 28 days has sharp stress drop after first cracking. This should be explained by the higher cracking strength of SAC+Z144 at 28 days. Higher elastic bond may lead higher initial cracking strength and a sharp drop after cracking.

Using equation (3), the average crack width at the point of tensile strength achieved of the composites at 7 and 28 days is calculated and the results are listed in Table 6. By comparing the cracking opening at tensile strength achieving between the reference and mixtures with the calcined zeolite addition, it can be clearly shown the improvement is significant. Although the crack width as tensile strength achieved of hybrid PVA and steel fiber reinforced HSECC already reduced from 60 μm to 25 μm (Zhang et al. 2017a). In the present study, the crack width is further reduced from 25 μm to 18 μm. This improve-
ment will greatly enhance the long-term durability and self-healing characteristic of the composite. This outstanding performance on crack width controlling of hybrid PVA and steel fiber reinforced HSECC should help to improve the concern regarding the negative effect of cracks inside of ECC on permeability of the material (Zhang et al. 2017a). In summary, through mixture optimization on internally cured HSECC with calcined zeolite particles, improved mechanical performances in tension are observed apart from shrinkage reduction. Four optimized mixtures of HSECC are obtained with compressive strength, cracking and tensile strength, ultimate tensile strain and average crack width at tensile strength of 73.2 - 87.9 MPa, 4.31 - 5.48 MPa and 6.36 - 7.27 MPa, 1.01 - 1.27% and 18 - 24 μm.

4. Conclusions

Mixture optimization on internally cured high strength engineered cementitious composite (HSECC) with pre-wetted calcined zeolite is conducted in this paper based on shrinkage and mechanical performance tests. The following conclusions can be drawn from the present study:

(1) Through mixture optimization, strength of cement matrix with the calcined zeolite addition is further improved. The optimized replacing ratio of cement with the zeolite in the matrix is existed to obtain the highest compressive and bending strength.

(2) Shrinkage the HSECC with optimized addition of the calcined zeolite is significantly reduced. The highest reduction rate on shrinkage of the composite comparing to the reference achieves 78%.

(3) Tensile performance with optimized addition of the calcined zeolite is improved comparing with the reference mixture as well. Four optimized mixtures of HSECC are obtained with cracking and tensile strength, ultimate tensile strain and average crack width at tensile strength achieved of 4.31 - 5.48 MPa and 6.36 - 7.27 MPa, 1.01 - 1.27% and 18 - 24 μm.

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