Designing Concrete with Self-healing Properties Using Engineered Cementitious Composites as a Model

A C Mircea$^{1,2}$ and T P Toader$^{1,2}$
$^1$NIRD URBAN-INCERC Cluj-Napoca Branch, 117 Calea Florești, 400524, Cluj-Napoca, Romania.
$^2$Technical University of Cluj-Napoca, 28 Memorandumului Street, 400114, Cluj-Napoca, Romania.

*E-mail: anamaria.mircea@incerc-cluj.ro

Abstract. Engineered Cementitious Composites (ECC), also known as Strain Hardening Cement-based Composites, are an easily molded mortar-based composites reinforced with polymer fibers. ECCs are designed based on micromechanics and fracture mechanics theory, to feature large tensile ductility and a variety of unique properties, including tensile properties, superior to other fiber-reinforced composites. The properties of ECCs can be custom-tailored through micromechanics design due to the interaction between the fibers and cement matrix. A structural deterioration of ECC is avoided because the fibers do not allow cracks with large widths to form, unlike conventional concrete. ECCs have the capacity to bend, generating a flexible material. Ductile properties rather than brittle had increased, unlike ordinary concrete, leading to a wide variety of applications. Obtaining superior characteristics for ECC, both in fresh and hardened state, a transition from ECC paste to concrete with self-healing properties was made. To obtain self-healing concrete, with same ECC paste behavior and characteristics, the mix-design of the paste was optimized. The aim of this article is to present the experimental results regarding the transition from ECC paste to self-healing concrete and to analyze the results in order to establish a mix-design pattern for concrete with self-healing properties.

1. Introduction
Since, both nationally and internationally, the quantity of products considered waste is very high, their removal from the list of products that pollute the environment leads to their use/re-use by incorporating them into new, innovative materials, and tending to obtain environmentally friendly / green materials [1].

The experimental study included the identification of raw materials (cement, additions, aggregates, fibers, additives, etc.) and their compatibility in order to produce materials with special performances. Performance evaluation was carried out experimentally, using standardized but also non-standardized testing methodologies.

2. Materials and methods
Engineered cementitious composite materials are an assembly between two or more distinct materials. Their combination generates different and significantly superior properties and characteristics than those of each individual component material [2]. To the matrix or also-called basic component is added a reinforcement component that alters and improves its mechanical and self-healing properties.

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The reinforcement component is evenly distributed throughout the volume of the composite material matrix [3].

In designing the compositions of the engineered cementitious composite materials, the following constituents were used:

- Fine aggregate (Sand): Fine silica sand (FSS);
- Portland Cement: CEM 42.5 R;
- Polymer fibers: PVA and hybrid PP;
- Superplasticizer additive, water reducer, polycarboxylate type (HRWR);
- Additions (P): limestone slurry (SL);
- Water (A).

Several parameters vary in the mixtures as follows:

- The amount of water/additive, with the implicit effect on the ratios-water/cement, water/link; liquid/cement, liquid/link;
- The type of fly ash used – in terms of physical and chemical composition;
- The type of fibers used;

The mixing technology used is specific to this type of material and involves the use of a pallet mixer with a mixing vessel capacity of approximately 5 liters, in accordance with EN 196-1 specifications.

The technological sequences followed in order to produce this material are:

- conditioning the raw materials at a temperature of (20±2)°C and (50±5) RH;
- dry mixing of the aggregates (sand), fly ash and cement in the mixing container;
- the quantity of limestone slurry is added and the low-speed mix-up continues;
- the quantity of limestone slurry is added and the low-speed mix-up continues;
- approximately 1/3 of the amount of water is added to moisten the components in order to stimulate the reactivity of the additive and continue the mix-up;
- the additive and the rest of the water are added and the alternative mix-up continues;
- a break sequence is adopted to give the superplasticizer additive the ability to act fully on the mixture, improving its workability;
- continue mixing, while the fibers are gradually added, to ensure uniform distribution of them in the mass of the mixture.

The materials used in the mixtures have the following characteristics:

- Aggregates- fine aggregates of fine silica sand type, from a local source, with a granulation of not more than 0.3 mm;
- The binder material used in the compositions is Portland cement CEM I 42.5 R;
- Fly ash with the following chemical composition (Table 1);
- Superplasticizing additive, water reducer, allowed considerable reduction of specific ratios (A/C; L/C; A/B; L/B), while maintaining high workability;
- Fibers that satisfy the following conditions [4]:
  - tensile strength about 2 to 3 times higher than concrete;
  - adhesion to the concrete matrix at least equal to or even greater than the stretch resistance of the concrete;
  - Elasticity module of the fibers is significantly higher than those of concrete;
- Additions- limestone slurry (SL) considered chemically inert and without pozzolanic activity, added in order to optimize mixtures and to attenuate segregation and the tendency of bleeding of the mixture.

Polyvinyl alcohol (VPA) fibers used have high elasticity mode and high stretching resistance; size of 8mm and superior mechanical properties, non-corrosive, non-magnetic, chemically inert and with 100% resistance to alkalis [5].
Several studies were evaluated in the research for different mixtures. The hardened characteristics of the composite materials have been determined, as follows [6]:

- the density in hardened state both after taking demolding the material and at the age of 28 days;
- mechanical resistance: bending and compressive strength;
- evaluation of the repair potential of the material at different ages: 28, 56, 90 days.

Tensile strength of the material was determined on 40 x 40 x 160 mm prismatic test specimens, using the three-point bending test (3PB), in accordance with EN 196-1:2016. Compressive strength was also determined in accordance with EN 196-1:2016 *Cement test methods. Part 1: Determination of resistance* [7], using the compression test of half prismatic specimens resulting from the three-point bending test (3PB). The compression load speed used was 50 N/s (0.12 MPa/s). For each sample, all data were recorded and the results obtained represent the average of the values read/calculated on a minimum of three samples [8].

The self-healing potential of the compositions was determined on coupons as follows [9]:

- R samples, which have been tested to breakage at the specific age;
- SH samples, at the specific age of 28 days until the initial cracking in the material was recorded, they were exposed to 28 SH cycles, (self-healing cycles), after which were tested under the same conditions as the original ones, at the age of 28+28 days;
- VS samples, which were kept intact until the age of retesting the SH samples, when tested under the same conditions as the SH samples, at the specific age of 28+28 days.

Exposure to SH cycles involved an alternation in a period of 24 hours between exposure of test specimens in wet environments, or immersed in water at T of (20±2)°C, for 12 h, and exposure of test specimens in air at T: (21 ± 3)°C and (50 ± 5)% RH for 12 h.

The repair potential of microcracks is presented by microscopic visual evaluation carried out before and after exposure of test specimens (coupons) to SH cycles (28 cycles).

The network of microcracks generated is characterized by:

- Number of cracks / microcracks (n);
- Open cracks: mean value (wmed), minimum value (wmin) maximum value (wmax).

In Figure 1, the coupons for assessing the repair potential of the compositions before testing are presented.

### Table 1. Fly ash chemical composition.

| Oxides     | %   |
|------------|-----|
| SiO₂       | 53.75 |
| Al₂O₃      | 26.02 |
| Fe₂O₃      | 7.91  |
| CaO        | 2.54  |
| MgO        | 1.54  |
| SO₃        | 0.35  |
| Na₂O       | 0.59  |
| K₂O        | 2.57  |
| P₂O₅       | 0.12  |
| TiO₂       | 1.02  |
| Cr₂O₃      | 0.05  |
| MnO₂       | 0.09  |
| ZnO        | 0.04  |
| SrO        | 0.02  |
| L.O.I.     | 3.14  |
3. Results and discussions

The evolution of composite materials at different ages following conditionings at alternating wet/dry cycles is shown in Table 2.

Table 2. Microcracks evaluation.

| Samples   | n  | $w_{\text{max}}$ (µm) | $w_{\text{med}}$ (µm) | $w_{\text{min}}$ (µm) |
|-----------|----|-----------------------|------------------------|------------------------|
| R         | 18 | 186                   | 117                    | 52                     |
|           |    |                       |                        |                        |
| Self-Sealing (SS) |   |                       |                        |                        |
| SH        | 15 | 134                   | 69                     | 19                     |
|           |    |                       |                        |                        |
| Self-Sealing (SS) |   |                       |                        |                        |
| Partially closed cracks | 8 |                        |                        |                        |
| Total closed cracks        | 25 |                        |                        |                        |

The results of the mechanical tests of the analyzed composition are presented in Figure 2 and Figure 3 respectively.

Figure 2. Compressive strength of the analyzed composition.
Figure 3. Tensile Strength of the analyzed composition.

The samples were tested at ages of 28, 56 and 90 days [10] and an increase in strength can be observed with testing at a later age, both at compression and at the tensile strength.

We can observe how the strengths increase from 28 days to 56 days and from 56 days to 90 days almost constantly but with no more than 50% of their value.

Figure 4 includes the comparative evaluation (before/after) for the analyzed microcracks.

Figure 4. Microscopic analysis- specimens evaluated after microcracking and re-evaluated after supposing them at SH cycles.
4. Conclusion and perspectives

- After conditioning the samples, tested at 28 days, at 28 wet/dry cycles, 75% of the total microcracks closed completely, with only 25% partially closed;
- Following mechanical tests at different ages, a significant increase of strength from 28 to 90 days is observed, both at compression and in tensile strength;
- Since the results obtained from the testing of the compositions of cementitious composites materials with self-healing properties were very promising, the transition from small mortar-type castings to large castings – to concretes was made, trying to preserve the same compositional proportions. It has been observed that in large scale, the amount of water and additive used for small castings is too high and requires a compositional reassessment by reducing their percentages;
- The design of cementitious composite materials continues towards the creation of concrete compositions with self-healing properties, with characteristics at least the same as the cementitious composite materials studied.

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