

**FIBRE ENGINEERED CEMENTITIOUS MATERIALS (FECM)  
OPTIMISATION BY THE USE OF HYBRID POLYPROPELENE FIBRES AND  
SUPPLEMENTARY POWDER ADDITION**

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**ABSTRACT**

Development of Fibre Engineered Cementitious Materials with Self-Healing Capacity (SH-FECM) was an important goal of the recent research programmes conducted in NIRD “URBAN-INCERC” Cluj-Napoca Branch. Several theoretical and experimental studies were performed in order to improve the composites, considering both, fresh and hardened state properties and also the performance as filling material of the narrow spaces at the beam to column interface within the Hybrid spatial joint model [1]. Present studies are focused in optimising the already developed mixtures regarding several aspects: the self-compacting behaviour of the fresh composites, considering the increased risk of “balling” effect when using 2% (of volume) of polymeric fibres as disperse reinforcement of the cementitious matrix, bleeding control during and after the mixes sequences by using supplementary powder addition, evaluation of the mixes when using a combination of two distinct types of polypropylene fibres, and development of controlled multiple cracks under loading, etc. The present paper offers the first results regarding the performance of the cement based composites, namely the fresh state self-compacting effect and also the flexural and compressive strengths at 7 and 28 days of age. Initial conclusions when replacing the traditional silica sand as aggregate of initial mixes with regular sand (0/1 mm fraction) are also considered. The Self-Healing potential of the mixes will be evaluated further on, ensuring the complete range of characteristics related to durability improving, repair and maintenance cost reduction and superior structural performance, in the context of the expected features of the sustainable development.

**Keywords:** microcracks, fibre engineered cementitious materials (FECM), crack control, self-healing capacity; durability.

## INTRODUCTION

In the last years NIRD “URBAN-INCERC” Cluj-Napoca Branch hosted a complex research programme regarding the design and experimental evaluation of seismic resistant joints that can improve the overall stability of precast structures subjected to dynamic actions (strong winds and mostly earthquake incidence). Two main models were effectively built up and subjected to repeated testing procedures for a complex evaluation of their performance: planar and spatial models [1], [2], [3].

In both models, the vulnerable aspects were the beam-column contact interface surface, a narrow space subjected to dynamic, high rate loading, tensile – compressive alternating stresses, demanding for a high performance filling composite that could behave in a satisfying way under these conditions.

Starting from the design principles and mixing technology provided by V. Li [4], regarding the classical ECC M45, several composites were developed using locally available raw materials [2], [3]. The further improvement of the mix designs considered the provisions developed by D. Snoeck [5]. The evaluation of the materials (SH-FECM) was promising with respect to: dynamic performance, namely load rate sensitivity, mechanical characteristics, namely compressive and tensile strengths (3PB and 4PB tests), and also Self-Healing Capacity (Self-Closing – crack sealing parameter and Self-Repairing – mechanical recovery) [2], [3].

## POLYMERIC FIBRE CEMENTITIOUS COMPOSITES

### 1. Design principles

As mentioned, the cement-based mixtures were realized taking into consideration the previously achieved results. The mix proportion is quite close to the previous one, namely to PP – M1.5, that also relies on Snoeck’s specifications [5], but maintaining a higher amount of fly ash with respect to the cement. Supplementary, powder additions are introduced for increasing the fine particle content, assumed to be insufficient when the SH – FECMs were initially produced [2]. Finally, the sequence of mixing relied on Snoeck’s specifications [5], but some small adjustments were introduced.

### 2. Raw materials

The raw materials, specific for ECC’s, are locally available: the binding material (B) is a combination of CEM I 52.5 N (C) and class F Fly-Ash (FA), FA/C = 1.2 as V. Li recommends [4], [6]. The fly ash used for the present mixtures is delivered by the power station Mintia (Hunedoara County, Romania), slightly different in terms of chemical composition to the Govora FA, initially used. Samples from the two types of FA were subjected the X-ray fluorescence analysis (XRF analysis), in accordance to the ASTM norms, for determining the chemical composition, presented in Table 1.

Two types of sand were used: silica sand (SS), 500  $\mu\text{m}$  maximum grain size and regular sand (NS) (0/1 mm fraction), for initial evaluation of aggregate influence, both in fresh and hardened state of the composites. The aggregates are also local, delivered by a nearby aggregate quarry. The specific granulometric curves for the silica sand (SS) and regular sand (0/1 NS) are presented in Fig. 1, a and b.

The Polymeric fibres used as dispersed reinforcement in the mixtures 2% (by volume) are a combination of two types of Polypropylene (PP) fibres provided by the Romanian producer Romfracht: RoWhite PP (PP1), approx. 1/3 from the total amount and the rest of 2/3, ECONO NET (PP2), structural PP fibres (Fig. 2, a and b).

Table 1 Chemical composition of fly ash batches

Chemical Composition	Govora [%]	Mintia [%]
SiO <sub>2</sub>	51.76	53.61
Al <sub>2</sub> O <sub>3</sub>	21.86	26.16
Fe <sub>2</sub> O <sub>3</sub>	9.40	7.58
CaO	6.56	2.42
MgO	2.43	1.49
TiO <sub>2</sub>	0.84	1.04
K <sub>2</sub> O	2.16	2.60
P <sub>2</sub> O <sub>5</sub>	0.14	0.12
SO <sub>3</sub>	0.38	0.26
L.O.I.*	3.69	3.57

\*L.O.I. - loss on ignition

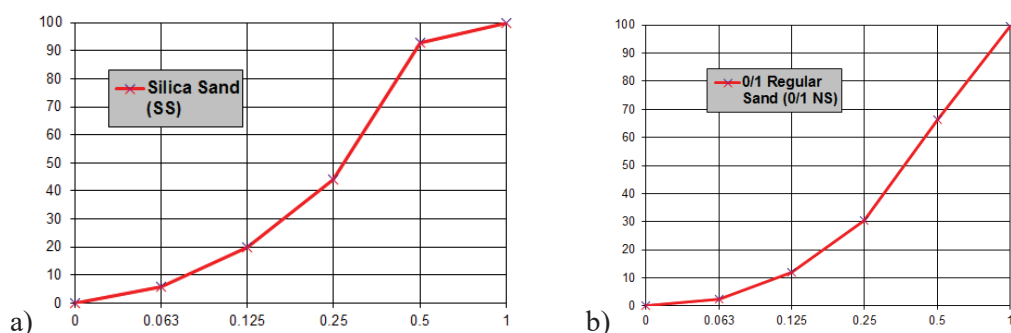


Figure 1 Granulometric curve of: a) Silica sand (SS); b) Regular Sand (0/1 NS)



Figure 2 Polypropylene (PP) fibres: a): RoWhite (PP1); b) al ECONO NET (PP2)

Two reference mixtures were initially produced: T SS, by using the SS sand and T NS0/1, by using the developed NS0/1, regular sand. Further on, new four mixtures were developed starting from the basic ones, by adding supplementary admixtures, namely Silica Fume (SF) or limestone filler (L), in order to increase the powder content of the matrix. The admixture amount in all mixtures is constant, namely 80 kg/m<sup>3</sup>.

The High-Range Water Reducer (HRWR) polycarboxylate superplasticizer Glenium 51, provided by BASF Company, was used in varying amounts, 10 to 12 kg/m<sup>3</sup>, depending on the fresh state characteristics of the mixture.

### 3. Mix proportions and sequence of mixing

The mix proportions are presented in Table 2.

Table 2 Mix proportions

C	FA	B	S	W	HRWR	Powder	PP	W/C	W/B	L/B
<b>T SS – Silica sand, no addition</b>										
1.0	1.2	2.2	0.8	0.65	0.021	-	2%	0.65	0.30	0.31
<b>T NS0/1 – Regular sand, no addition</b>										
1.0	1.2	2.2	0.8	0.65	0.018	-	2%	0.65	0.30	0.30
<b>SS-SF – Silica sand, Silica Fume addition</b>										
1.0	1.2	2.2	0.8	0.65	0.021	0.18	2%	0.65	0.30	0.31
<b>NS 0/1-SF – Regular sand, Silica Fume addition</b>										
1.0	1.2	2.2	0.8	0.65	0.018	0.18	2%	0.65	0.30	0.30
<b>SS-L – Silica sand, Limestone filler addition</b>										
1.0	1.2	2.2	0.8	0.65	0.021	0.18	2%	0.65	0.30	0.31
<b>NS 0/1-L – Regular sand, Limestone filler addition</b>										
1.0	1.2	2.2	0.8	0.65	0.018	0.18	2%	0.65	0.30	0.30

Regular 0.8 l batches were obtained by using an adapted mixing procedure based on the EN 196-1 Standard. Two speeds, Low speed (LS) and High Speed (HS), at 140/280 rot/min, were used. The first step included 60 s of LS dry mixing of binding agents, C+FA; if the mixture included admixture, this material, SF or L, was added to the binding system. Addition of sand, silica sand or regular implies another 30 s of LS dry mixing, followed by addition of approximately one third of the water amount, in order to accelerate the activation of superplasticizer, recommended to be placed on wet aggregates. Afterwards, the HRWR was added together with the rest of water and 60 s of LS mixing is completed by 30 s of HS mixing, followed by a 90 s waiting period, for the composition to rest and the HRWR could be fully active. The further 3 min of LS mix included the step-by-step addition of the hybrid PP fibres, in order to ensure a homogeneous dispersion and avoid the balling effect. The procedure is completed by extra 30 s of HS mixing.

## RESULTS AND DISCUSSIONS

### 1. Fresh State Characteristics

The two initial mixtures, T SS and T NS 0/1 had the typical ECC fresh state aspect (see Fig. 3): creamy, with proper fibre dispersion, good workability that allowed placing in the mould without jolting or vibrating procedures. Considering the increased percentage of fibres, the Self-Compacting (SC) characteristics are not achieved with respect to traditionally required parameters. Sensitive bleeding of the mixtures was also noticed, but the hardened state specimens were not affected by this phenomenon.

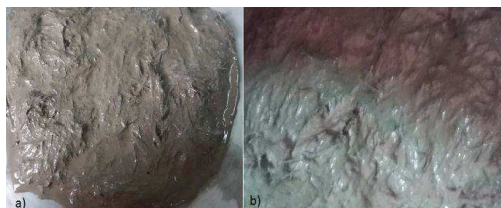


Figure 3 Fresh state of initial mixes: a) T SS and b) T NS 0/1

The mixtures containing additions (SF or L) showed an improved fresh state aspect, more homogenous and increased cohesive behaviour: reduced bleeding and proper workability when casting in the mould, without the need for jolts applying (see Fig. 4). It was noticed that the supplementary additions did not require increase of liquid quantity, nor water (W) or superplasticizer (HRWR) for similar and improved fresh state characteristics.



Figure 4 Fresh state of: a) SS-SF b) NS 0/1-SF c) SS-L and d) NS 0/1-SF

## 2. Physical and Mechanical Properties

After removing from the mould at the age of 24 h, the specific 40 x 40 x 160 mm<sup>3</sup> prismatic specimens are cured by immersion in water, at the temperature T (21 ± 2) °C.

The specific tensile and compressive tests are carried out on considering the EN 196 (Cement) and EN 1015 (mortar) specific testing methods.

### 2.1 Specific weight (SW)

The specific weight (kg/m<sup>3</sup>) was determined by mass to volume ratio, by weighting the prismatic specimens immediately after removal from the mould, at the age of 24 h and the results are included in Table 2.

### 2.2 Mechanical properties

The tests are performed at the early age of 7 days, 28 days and at a later age of 56 days, considered relevant due to substantial amount of FA in the mixtures, known for late age pozzolanic potential.

Table 2 includes, besides the specific weights of the materials, the mechanical properties at the relevant ages of 7, 28 and 56 days.

Table 2 Physical and mechanical characteristics of developed mixtures

Mix	SW (kg/m <sup>3</sup> )	3PB Tensile resistance (MPa)			Compressive resistance (MPa)		
		7 d	28 d	56 d	7 d	28 d	56 d
T SS	1870	13.2	14.0	17.2	26.5	40.5	50.8
T NS 0/1	1870	14.3	14.8	16.8	27.6	42.3	51.8
SS-SF	1870	13.2	14.8	17.4	40.4	67.4	77.7
NS 0/1-SF	1870	14.3	16.4	16.8	27.6	63.4	71.9
SS - L	1900	14.6	17.6	19.3	40.4	50.1	55.1
NS 0/1-L	1910	14.3	14.8	17.5	37.3	51.6	56.7

The two initial mixtures, T SS and T NS 0/1 showed similar results, both in tension and compression and at all testing ages. Silica fume (SF) addition proves to be helpful in raising the compressive strength, as expected, and especially in the silica sand (SS) mix: early age high compressive strength and consistent increase, both at 28 and 56 days. The limestone filler addition is beneficial in tension, where superior performance is achieved especially in the case of silica sand (SS) mix, SS – L, that proves to offer a balanced, lubricated matrix, with proper compatibility to the fibres, encouraging for further evaluation and possible optimisation (Fig. 5). The regular sand, lime addition mix, NS 0/1-L offered close results, but a less promising cracking pattern under loading.



Figure 5 Mixture SS-L: Failure and cracking pattern under tension and compression

The graphical representation under 3PB bending of the four composites with supplementary powder additions are presented in Figure 5 and their compressive evaluation in Figure 6.

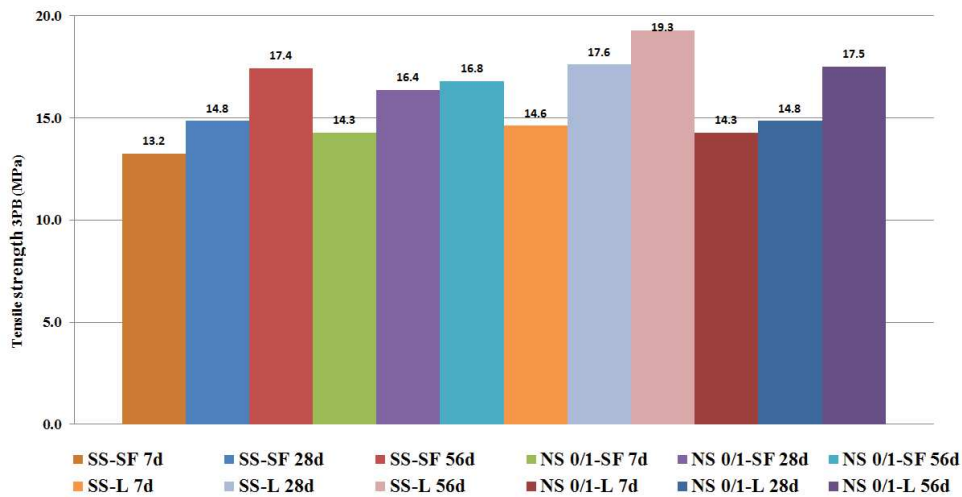


Figure 6 Tensile performance of the composites with supplementary powder addition

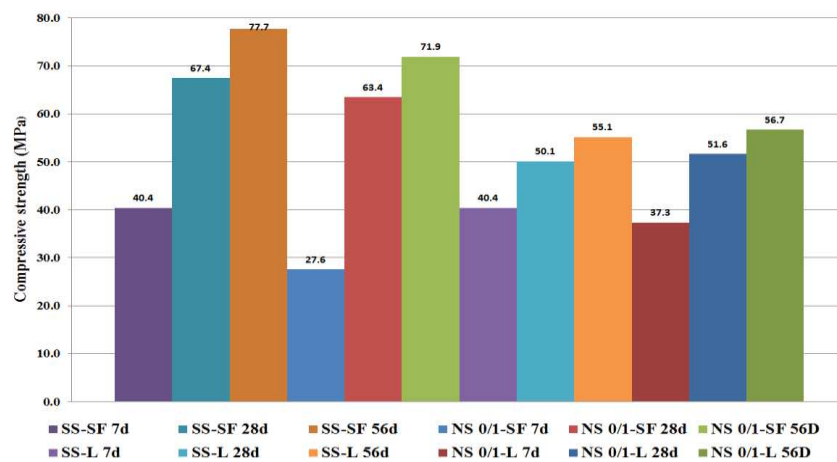


Figure 7 Compressive performance of the composites with supplementary powder addition

### 2.3 Multiple cracking under 4PB

The 4 point bending (4PB) is considered the typical, characteristic flexural test for ECCs, emphasising in the most conclusive manner the multiple cracking potential of the mix and, as consequence, the crack control potential, which is a particular feature of ECCs. In order to evaluate the mixtures performance under 4PB, 220 x 40 x 8 mm<sup>3</sup> coupon specimens were produced. The casting and curing conditions were identical to the previous test specimens. UNIFRAME MINI, a displacement control testing device, specific to EN 12002 adhesive mortar transversal deformation testing, was adapted and used to perform the 4PB required loading. The testing procedure, composites evaluation and method improvement is on-going project.

Initial evaluation, at the age of 7 days, identified the SS-L mix as the most suitable one for the multiple cracking development under loading, showing ductile behaviour, consequently. Figure 8 shows relevant aspects regarding the testing device and the used procedure, also emphasising the SS-L mixture potential to develop the desired multiple cracking (Fig. 9), increased mid-span displacement (exceeding 20 mm) and the typical ECC ductility. The other mixtures did not show such promising results, usually developing one crack that enlarges continuously until failure is considered.

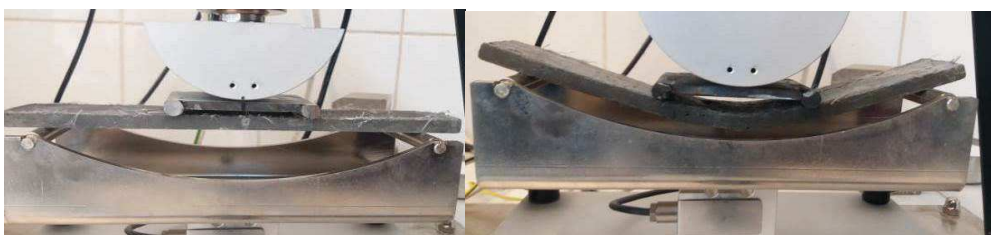


Figure 8 SS-L mix: 4PB testing (support condition; mid-span maximum deflection)

### 2.4 Self-Healing Capacity (Self-Closing of Cracks)

The Self-Healing capacity of the materials is evaluated by the means of Self-Closing of the developed cracks under loading. The visual evaluation is performed on several tested specimens, after exposure to 20 wet and dry curing cycles, for healing inducing: WET: 8 h immersion into tap water, at the temperature of  $(20 \pm 2)$  °C; DRY: 16 h exposure to air, at T:  $(21 \pm 3)$  °C and RH:  $(50 \pm 5)$  %. Partial and even complete sectors of cracks are remarked, the sealing efficiency depending on initial crack opening. Precipitation products and also further hydration gels are expected to develop further on, due to late pozzolanic reactivity of Fly Ash. The testing, curing and healing efficiency evaluations are an on-going project.



Figure 9 SS-L mix, 4PB multiple cracking: a) initial; b), c) crack closing after curing exposure

## CONCLUSIONS

The preliminary procedures for SH –FECM optimisation by the means of powder addition and using locally available raw materials shows encouraging results. The limestone filler, a cheap available compound, proves to bring beneficial effect, mainly when used together with the silica sand (SS). The PP hybrid hydrophobic fibres, provided by the Romanian producer Romfracht: RoWhite PP fibres (lower tensile strength, early cracking prevention) and ECONO NET PP fibres (structural, increased tensile strength), developed good behaviour and compatibility in the cement-based matrices, the multiple cracking pattern being achieved for the SS-L mixture. The regular sand mixes achieved comparable performances in terms of flexural and compressive strengths, but failing in multiple cracking development under 4PB loading. Nevertheless, the optimisation of the mix designs, both in fresh and hardened state, followed by specific testing represents the future approach in this direction, The Self-Compacting (SC) behaviour of the fresh mixes and the ductile behaviour under loading of the hardened state materials are the general, near future goals of the research programme.

## ACKNOWLEDGEMENTS

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