

# Optimizing approach on Fibre Engineered Cementitious Materials with Self-Healing capacity (SH-FECM) by the use of slurry lime (SL) addition

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**Abstract.** SH-FECM (Fibre Engineered Cementitious Materials with Self-Healing capacity), developed at NIRD “URBAN-INCERC” Cluj-Napoca Branch in the last five years, are consistently based on Engineered Cementitious Composites (ECCs) concept, elaborated in the early nineties at Michigan University (USA). They all represent a cement-based typology of dispersed reinforcement composites able to develop high deformability by the means of multiple cracking pattern under loading, leading to a cumulative set of valuable material features: metal like behaviour when subjected to loads, brittle failure prevention, increased self-healing potential via the compositional design, etc. The process of development and constant improvement of the SH-FECMs represents a long term theoretical and experimental approach, aiming to establish the optimum raw materials (mostly locally available) compatibility within the cementitious matrix so that the composites would present superior performance under comparative evaluation. This paper presents the first results, evaluated as positive for both, fresh and hardened state materials, regarding the inclusion of Slurry Lime (SL) addition as replacement of the initial lime powder addition (L) in the material composition. The long-term effects are on ongoing investigation, but the initial results are clearly promising, starting from a better fresh state aspect and evaluating for faster setting time and improved early age mechanical behaviour. The beneficial effects are also in terms of economic and ecological aspects, considering that the used lime slurry (SL) addition represents an actual waste resulted from a local, natural stone processing factory. Its use as direct addition in the SH-FECMs mixtures could represent an efficient recycling and waste prevention action, with long term beneficial potential, in terms of Circular Economy principles.

## 1 Introduction

Fibre Engineered Cementitious Materials with Self-Healing capacity (SH-FECM) represent strain hardening, fibre reinforced composites, able to develop width & shape controlled microcracks under increasing load. This kind of cracking pattern represents the foundation of co-dependent physical and mechanical intrinsic features of the material: a) high deformability, metal like behaviour, preventing the sudden, brittle failure of classic plain concrete of cementitious materials; b) improved self-healing potential, as a direct consequence of small cracks and of dispersed reinforcement, (which not only blocks crack widening but also represents a skeleton where precipitation products, emerging from concrete structure in direct relation to the environmental conditions, can settle and facilitate the small crack closing [1, 2, 3].

The SH-FECM were developed at Incerc Cluj by initial adaptation of the Engineering Cementitious Composites (ECCs) concept of Victor Li [1] to the specificity of local conditions and raw materials [4, 5].

The ECCs and consequently, the SH-FECMs belong to the High Performance Fibre-Reinforced Cementitious Composites (HPFRCC) family, combining innovative features, theoretically integrated in the matrix functionality by the means of micromechanics tools [1, 2, 6, 7, 8, 9, 10, 11, 12, 13] in order to amplify some intrinsic material properties. Consequently, the strain hardening behaviour under loading, which ensures development of successive, opening controlled microcracks under loading, ensures by default the optimisation of material self-healing capacity: small cracks are easier to fill and seal with the healing products, generated due to specific self-sealing properties of the cement based materials [1, 2].

The Self-Healing concept in the area of cementitious materials represents a research topic that recently gained substantial attention from academia, research groups and also the construction industry, due to its consistent potential towards durable infrastructure development and maintenance. Preventive solutions and design of smart materials which can better ensure the infrastructure

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safety and durability requirements, with reduced costs in terms of rehabilitation and repair along their life span, represents new alternatives that recently delivered encouraging results [3].

The design, production and further improvement of the SH-FECMs at Incerc Cluj by using locally available raw materials represented one of the major research directions of the last years [15, 16]. The high fly ash (FA) content of the SH-FECM binding system also represents an attractive possibility of valorising the huge fly ash quantity, by-product / waste of the energy industry in Romania. Statistical data showed that in 2017 for instance, one Romanian power plant reported a production of approx. 650,000 tonnes of fly ash, 50,000 tonnes of slag and 50,000 tonnes of gypsum. Unfortunately, just a small FA amount, less than 25%, approx. 162,000 t/year is used in the construction industry (as addition for concrete and mortars, cement, soil stabilisation – mainly for the roads, infrastructure, etc.). The rest is simply converted into large quantities of industrial waste, stored in waste dumps and experiencing fast degradation of their potential as binding/filler raw material (due to complete exposure to outdoor climatic conditions) and simultaneously generating ecological discomfort [17].

Identifying new, innovative, high performance materials which could integrate large amounts of mineral additions (by-products/wastes, inert, latent or pozzolanic hydraulic potential as part of the binding system) represents one of the major goals of Incerc Cluj research in recent years [4, 5, 17]. This research topic is strongly founded and encouraged by Romania's need to develop the principles of Sustainability and Circular Economy, as part of its adherence to the European Union.

## 2 Experimental procedure – materials and methods

### 2.1 General approach

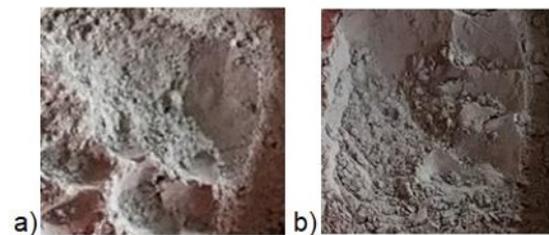
The current study evaluates the possibilities of optimising of SH-FECMs by using slurry lime addition and consequently, its influence and compatibility with the cement (C) and fly ash (FA) matrix. The mechanical and self-healing capacity evaluation of the material is also studied, with respect to the previous, stable mix containing lime powder (L), used as an inert filler for optimizing the fresh state, bleeding prevention, and matrix packing optimisation [4, 5].

### 2.2 Raw materials

The component materials for the SH-FECM mixes are mostly locally available: the binders (cement (C) and fly ash (FA)), the aggregate (fine silica sand (FSS)), and additions. The fibres used for the dispersed reinforcing of the composites are provided by the Japanese company Kuraray and they seem to generally provide excellent results in worldwide ECC development approaches [1, 2].

SH-FECM promotes the ECC *typical high fly ash (FA) content binding system*; FA even exceeds the cement (C) content and the specific ratio  $FA/C = 1.2$ . ECC typically represents high FA composites, with  $FA/C$  values ranging from 1.0 to even 1.5 or even more [1, 2, 4, 16], in order to provide proper matrix features for ensuring the strain hardening behaviour under loading and also the late self-healing potential, due to the long term FA pozzolanic activity [3].

Ordinary Portland Cement (OPC) was used (Fig. 1, a) and class F Fly-Ash (FA) (Fig. 1, b) produced by a local power station and characterised by the XRF analysis presented in Table 1.

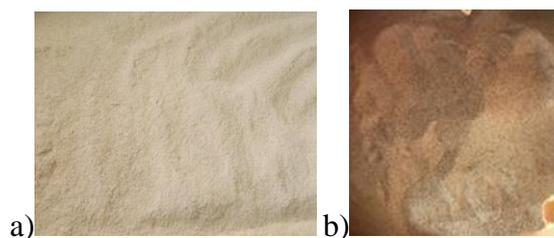


**Fig. 1.** The binding system: a) Ordinary Portland Cement, (C) b) Class F Fly Ash, (FA)

**Table 1.** XRF analysis data for the fly ash samples.

FA Oxides, %					
SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>
53.61	26.16	7.58	2.42	1.49	0.26
Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	Mn <sub>2</sub> O <sub>3</sub>	L.O.I.	-
0.59	0.12	1.04	0.08	3.57	-

The *Aggregate* represents Bega Minerale SA (Cluj, Romania) silica sand. The initial 0.5 mm maximum grain size sand (SS), (Fig. 2, a) was later replaced by a finer sand (FSS), 0.3 mm maximum grain size (Fig. 2, b), in the process of matrix packing optimisation and consequently for an increased strain hardening behaviour under loading, in accordance to previous recommendations [2].



**Fig. 2.** SH-FECM Aggregate: a) Silica Sand (SS); b) Fine Silica Sand (FSS)

The *polymeric fibres* are the Kuraray (Japan) Polyvinyl Alcohol (PVA) fibres (Fig. 3), lubricated (by 1.2% mineral oil) and generally confirming excellent performance for ECC development. The manufacturer's data sheet mentions the following Kuralon K-II fibre

RECS15x8 fibre characteristics: diameter 39µm, cut length 8.0±1.0 mm, tensile strength (tenacity) 12.0±2.5 cN/dtex (1560±325 MPa), Elongation 6.5±1.5%, Modulus - 320 cN/dtex. The high tensile strength and the elasticity properties of the fibres are proved to be crucial for the composite performance. Lower tensile strength polymeric fibres (Polypropylene fibres), used for a comparative evaluation, failed to achieve close results, especially in long term testing [4].



Fig. 3. PVA Kuralon K-II fibre (RECS15x8) (Kuraray, Japan)

The 2% (by volume) fibre content in the mix was proved to be the optimum for the dispersed matrix reinforcing [1, 2, 4]: those with a lower fibre content generally failed in ensuring the desired cracking pattern under loading; a higher fibre content does not involve additional benefits to the overall composite behaviour, but at the same time, it generates increased costs and also poor workability during mixing, with higher admixture addition.

The *secondary addition* consisting of limestone (filler or slurry) was included in the mix design for better control of the fresh state, namely preventing the bleeding effect when adding the fibre, balling of the fibre and also for ensuring the homogeneity of the mix and its creamy, desired texture [1]. The hardened state characteristics are also improved: besides better matrix packaging induced by increasing the fine particle content, the Self-Healing potential of the material is stimulated [3]. Initially, *limestone filler (L)*, available on the regular building materials market, was used (Fig 3, a). The last experimental procedures included the novelty of substituting the filler (L) by *limestone slurry (SL)* (Fig. 3, b).

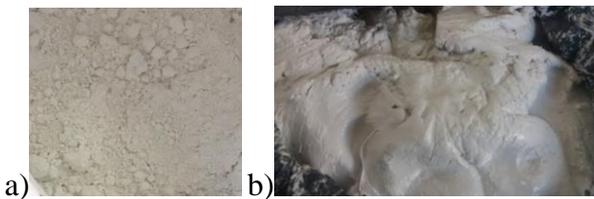


Fig. 3. SH-FECM Secondary additions: a) Limestone filler (L); b) Limestone slurry (SL), waste, with natural humidity

The limestone slurry (SL) represents a waste generated by the local producer of natural stone finishing products, which showed increased interest in finding an ecological and cheap solution for valorising it and consequently, elimination of the limestone slurry waste dumps (Fig. 4). A major direction of the research developed at Incerc Cluj-Napoca was to identify, evaluate and improve the potential of local mineral additions (by products or waste), as partial or complete raw material replacement

cement-based composites for the construction industry, and thus obtaining reliable composites in terms of applicability field, physical, mechanical and durability performance. The limestone slurry (SL) represented one of the targeted wastes of the study, considered as secondary addition, besides fly ash (FA), considered as primary addition and substantial cement replacement within the matrix. The SL initial evaluations generated very promising results, especially when combined with other additions and especially with the fly ash (FA) [18].



Fig. 4. Limestone slurry (SL) waste dumps

The low water to binder ratio (W/B), crucial for mechanical performance, was kept low by means of the *polycarboxylate superplasticizer* Glenium 51 (HRWR), (BASF), traditionally used for FECM development [2, 4, 5, 16].

### 2.3. Mix design and mixing procedure

Table 2 presents the mix proportions of the SH-FECM mixes and the specific ratios with respect to the cement (C) content (C = 1.0). Two SH-PVA-FECM mixes are considered for the current evaluation: 1) the SS-L-PVA-FA/C (1.2), developed by using the coarser silica sand (SS) and the limestone filler addition (L); 2) the FSS-SL-PVA-FA/C (1.2), developed by using the fine silica sand (FSS), with maximum grain size 0.3 mm and the Limestone slurry addition (SL).

Table 2. Mix proportions of SH-FECM.

Components	FECM Mixes	
	SS-L-PVA-FA/C (1.2)	FSS-SL-PVA-FA/C (1.2)
C*	1.0	1.0
FA	1.2	1.2
B	2.2	2.2
SS/FSS	0.8	0.8
L/SL	0.18	0.18
W	0.65	0.65
HRWR	0.021	0.021
FIBRE	2.0%	2.0%
W/C	0.67	0.67
W/B	0.30	0.30
Liq./B	0.31	0.31

\*C = 1.0;

Supplementary Symbols: B = C + FA (Binding system); SS – Silica Sand (max. grain size < 0.5 mm); FSS = Fine

*Silica Sand (max grain size <math><0.3\text{ mm}</math>); L = limestone filler; SL = Slurry Lime; W – Water; HRWR-Superplasticizer; Liq. = W+ HRWR;*

The supplementary powder addition (L) for the SS-L-PVA-FA/C (1.2) was established to 100 Kg/m<sup>3</sup> [4]. The slurry lime (Fig. 3 and 4) represents a lime paste with variable but easily determinable water content. The L replacement was performed by simply adding the equivalent of SL paste. The SL paste quantity to be added in the mix was calculated by initial evaluation of natural water content (by drying it until constant mass was reached and reporting it to the initial value) and considering as reference the L filler content (dry powder). In addition, the total water (W) in the mix was also adjusted considering the liquid input provided by the paste. The mixing sequences [5], established in accordance to the EN 196-1 [19] methodology by using the typical laboratory mixture suitable for low volume batches, are correspondingly adapted: the SL paste was added immediately after the dry mixing of the binding materials and aggregate, in order to ensure a homogeneous dispersion. The lime paste dispersed homogeneously and provided a nice, cohesive texture to the entire mix.

### 3 Experimental procedures: results and discussions

#### 3.1 Fresh state aspect

Both mixtures developed the homogeneous, creamy mix (Fig. 4), associated with the ECCs fresh state aspect [1, 2]. The SS-L-PVA-FA/C (1.2) mix already experienced an improvement regarding the fresh state workability (Fig. 4, a) and bleeding control, by using the limestone filler addition (L), when compared to the L free mixes. A clear improvement was noticed when replacing the L to SL addition (Fig. 4, b), as the material becomes more cohesive and homogeneous; these feature are preserved even after the fibre addition which happens in the last sequences of the mixture technology, usually considered the most vulnerable steps considering the risks of balling, bleeding and segregation.



a)



b)

**Fig. 4.** SH-FECM fresh state aspect: a) SS-L-PVA-FA/C (1.2) mix; b) FSS-SL-PVA-FA/C (1.2) mix

Placing into the moulds is performed easily, especially for the FSS-SL-PVA-FA/C (1.2), as the composites developed the necessary self-compacting behaviour which make vibration unnecessary.

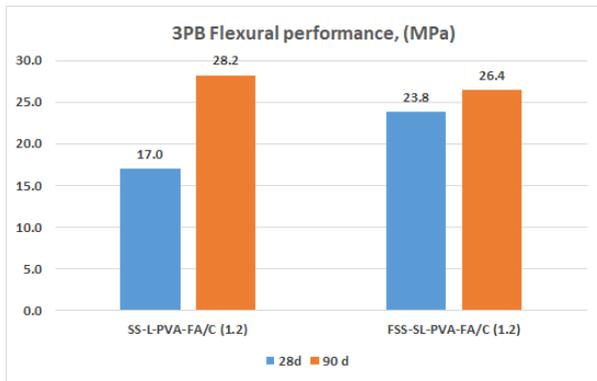
#### 3.2 Mechanical properties

The flexural and compressive strengths as relevant, basic mechanical characteristics for mix evaluation, were performed by using the EN 196-1 [19] and EN 1015-11 [20] methods. The relevant testing ages were considered at 28 days and also at 90 days: the late age testing is considered to bring important information for the high content fly ash composites, taking into account the late pozzolanic reaction of fly ash. Prismatic specimens, 40 x 40 x 160 mm<sup>3</sup>, removed from the moulds 24h after casting, were cured by water immersion at T (21 ± 2)°C until testing.

Table 3 presents the flexural strengths determined by 3PB test performed at 28 and 90 days and the strength increase recorded at the final testing, with respect to the representative 28 day values. The compression tests, performed on the half prisms resulted after 3PB tests, had a similar approach: 28 and 90 days compressive strength and the corresponding strength gain from initial testing to the final one. The obtained values are presented in Table 4. The graphical representation of the strengths are presented in Figure 5 (Flexural test) and 6 (Compression test).

**Table 3.** Mechanical strengths: 3PB Flexural Resistance.

SH-FECM Mixes	3PB Flexural resistance (MPa)		
	28 d	90 d	Strength gain (%)
SS-L-PVA-FA/C (1.2)	17.0	28.2	65.9
FSS-SL-PVA-FA/C (1.2)	23.8	26.4	10.8

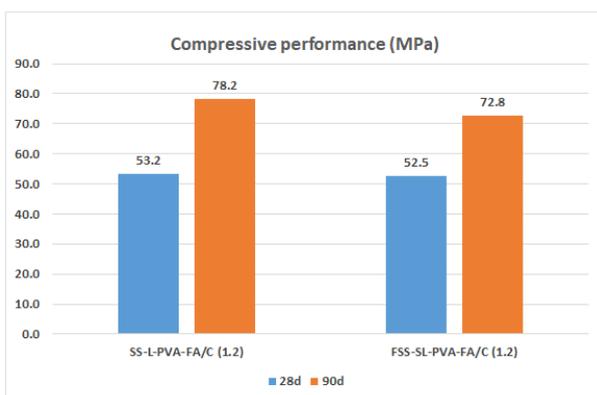


**Fig. 5.** SH-FECM, Graphical 3PB Flexural Resistance at 28 and 90 days

The analysed SH-FECM mixes show comparable results regarding the flexural performance (3PB). The slurry limestone (SL) mix seems to develop faster strengths at 28 days, but longer term evaluation (90 days) proves a clear gap recovery of the limestone filler (L) mix. The compressive results are even more balanced, proving the efficiency of replacing the limestone filler with the lime paste. Both mixes show impressive tensile performance and failure without loss of integrity.

**Table 4.** Mechanical strengths: Compression

SH-FECM Mix	Compression resistance (MPa)		
	28 d	90 d	Strength gain (%)
SS-L-PVA-FA/C (1.2)	52.5	72.8	47.0
FSS-SL-PVA-FA/C (1.2)	53.2	72.8	38.5

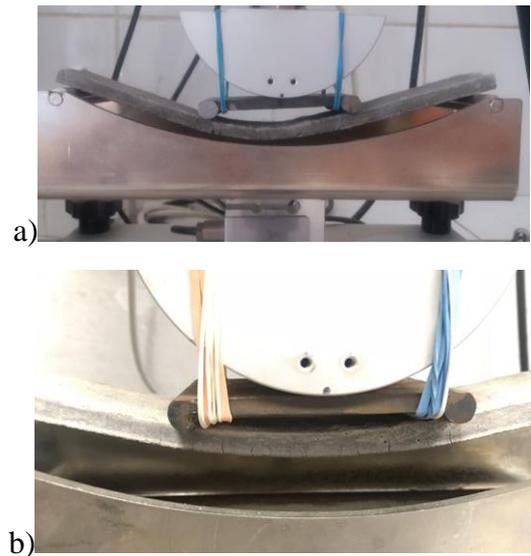


**Fig. 6.** SH-FECM Compressive Strength

### 3.3 Microcracking pattern (MC) at 28 days

The composite ability of developing the successive steady state multiplecracking network (MC) under loading generates the deformability potential and the strain hardening behaviour, characteristic for the ECC materials. The flexural 4-point bending test (4PB) at 28 days performed on coupon specimens, 200x40x8 mm provides comparative values of the two mixes. The

deflection of the materials under loading is presented in Fig. 7.



**Fig. 7** 4PB for SH – FECM: a) SS-L-PVA-FA/C (1.2) specimen; b) FSS-SL-PVA-FA/C (1.2) specimen

The testing device used for both mixes is the adapted 4PB displacement control UNIFRAME MINI. The network of the multiple cracks is characterised by the general aspect, the number of cracks developed until failure is considered and also by their openings (minimum, mean and maximum values); the crack typology (with or without branches) is also monitored.

The microscopic visual analyses were performed by using a stereomicroscope Leica S8 APO, equipped with digital camera Leica DMC2900. The LAS Analysis Bundle software was used for analysis of the data acquisition

The analytical results are summarised in Table 5.

**Table 5.** Microcracking potential (MC): 4PB testing at characteristic ages of 28 days

SH-FECM Mix	Crack number (n)	MC interval		
		Crack opening		
		$w_{max}$ (µm)	$w_{med}$ (µm)	$w_{min}$ (µm)
SS-L-PVA-FA/C (1.2)	18-22	192	29	10
FSS-SL-PVA-FA/C (1.2)	11-13	48	29	10

It was noticed that MC capacity SS-L-PVA-FA/C (1.2) mix overcomes the FSS-SL-PVA-FA/C (1.2)'s, in terms of crack numbers. Still, both mixes show nice 4PB cracking pattern and controlled microcracking developing, reaching the expected strain hardening typology.

### 3.4 The Self-Healing capacity

The Self-Healing (SH) capacity of the cementitious composites SH-FECM, is a relatively new concept associated to durability of material and also to its ability

to contribute to a sustainable development. SH means a reduced resource and energy consumption for its production and also for the preservation of its functionality, in optimum conditions and with minimum repair and maintenance input [3].

The self-healing approach implies two distinct but also interfering directions: a) Self-Closing (SC) of the cracks, meaning the material intrinsic capacity of sealing, complete or just partial, the cracks that occur within its structure due to state of stress; b) the Self-Repairing (SR) potential, namely the partial or complete, physical and mechanical performance regain of the microstructural material damage, related to the initial, virgin state performance.

The SS-L-PVA-FA/C (1.2) and FSS-SL-PVA-FA/C (1.2) mixes are analysed considering crack closing (SC) and also repairing of the microstructure (SR). The evaluation of SH performance is performed classically, by inducing a controlled state of damage, applying the healing curing and then the final, comparative analyses of the healed specimens (SH) with respect to the reference sample, (virgin state, VS).

The healing curing generally implies exposing the damaged samples to a pre-defined number of alternating cycles with variable relative humidity (RH) and temperature (T).

The experimental procedure for SH-FECM SH evaluation consisted of 28 wet and dry, alternating cycles (Wet: 12 h immersion in tap water, T (20 ± 2) °C; Dry: 12 h exposure to air, at T: (21 ± 3)°C and RH: (50 ± 5)%).

The current study is mainly focused on Self-Repairing (SR) evaluation. The Self-Repairing (SR) efficiency is performed by using the 3PB flexural test: prismatic specimens were subjected to flexural stress until the pre-cracked state is visually induced (SH samples); the Damage coefficient (D%) is quantified with respect to the flexural strength determined in identical conditions (similar specimens and similar testing, namely 3PB until failure is induced, (R samples) (Eq. 1).

After healing exposure, namely the 28 wet and dry alternating cycles, the SH samples are re-tested again to failure and the results are reported compared to the Virgin state samples (VS), representing identical prismatic specimens, cured by immersion in tap water, T (20 ± 2) °C till the testing of the healed samples. The results are expressed quantitatively, via Recovery coefficient (R%), (Eq. 2). At the same time, a qualitative evaluation is performed, via visual analysis.

The Damage coefficient (D%):

$$D\% = 100 \times (R_{ti,28d\ SH} / R_{ti,28d\ R}), (\%) \quad (\text{Eq. 1})$$

Where:

$R_{ti,28d\ R}$  - 3PB flexural strength determined on R samples, at the age of 28 days;

$R_{ti,28d\ SH}$  - pre-cracked 3PB flexural strength determined on SH samples at the age of 28 days;

The Recovery Coefficient (R%):

$$R\% = 100 \times (R_{ti,56d\ SH} / R_{ti,56d\ VS}) \quad (\text{Eq. 2})$$

Where:

$R_{ti,56d\ SH}$  - 3PB flexural strength determined on SH samples at the age of 56d (retesting after SH curing);

$R_{ti,56d\ VS}$  - 3PB flexural strength determined on VS samples at the age of 56d.

Table 5 presents quantitative evaluation of repairing capacity of SH-FECM mixes via 3PB testing, at 28 days, and 56 days respectively, after 28 wet and dry curing cycles.

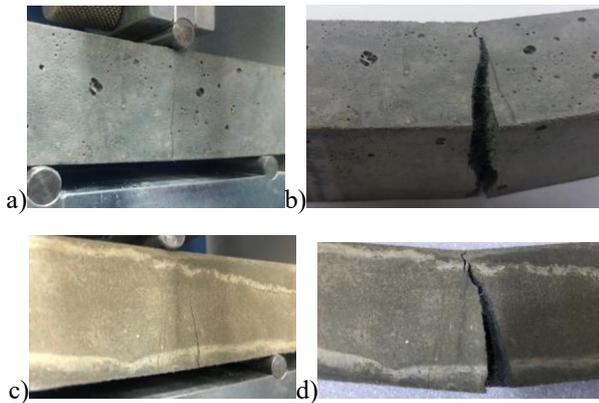
The qualitative analysis is performed by visual analysis of the cracking pattern, during the initial testing, after the healing curing and again, after re-testing the healed specimens. Visual analyses, microscopically performed, confirmed complete crack closing (100% SC efficiency) on all faces of the specimens, for both of the composites, which indicates important information regarding their durability performance. Crack sealing (SC) which is visually evaluated on the surface on the crack, does not imply crack filling with CSH or CaCO<sub>3</sub> precipitation products deep inside the crack, ensuring a regain of mechanical properties as well, but even a superficial crack closing can ensure protection against corrosion or other aggressive agents.

For both composites, SS-L-PVA-FA/C (1.2) and SL-PVA-FA/C (1.2), more than 50% of the evaluated cracking patterns produced during re-testing of the healed specimens, showed new crack(s) developing under re-loading, different from the initial one(s); the initial crack(s) remain(s) sealed during the re-testing, thus offering a reliable proof of the SH curing efficiency: the composites are able to initiate a high strength sealing products within the cracks, comparable to the virgin state material (Figure 8).

**Table 6.** Quantitative evaluation of SR capacity of SH-FECM mixes by using the Flexural 3PB testing at 28 days

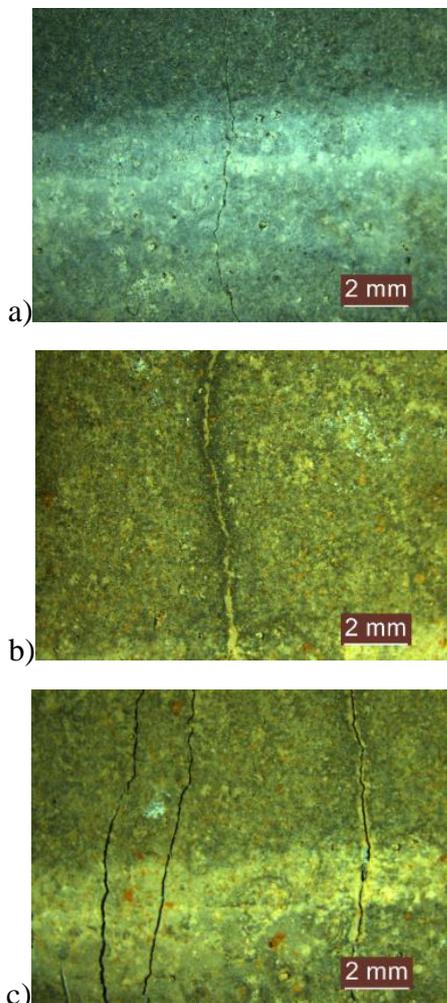
FEC M Mix	$R_{ti, 28d\ R}$ (MPa)	$R_{ti, 28d\ SH}$ (MPa)	D% (%)	$R_{ti, 56d\ SH}$ (MPa)	$R_{ti, 56d\ VS}$ (MPa)	R% (%)
SS-L-PVA-FA/C (1.2)	17.0	10.3	<b>60.6</b>	23.1	24.4	<b>94.7</b>
FSS-SL-PVA-FA/C (1.2)	23.8	9.5	<b>39,7</b>	23.4	24.2	<b>96.5</b>

Figure 9 emphasises the crack network evolution, microscopically evaluated, along the Self-Repairing experimental procedure: the initial crack initiation during the pre-cracking stage; crack sealing after curing of the samples; retesting up to failure of the SH specimens and identification of the cracking pattern, which proves to be different than initial one in more than 50% of the evaluated specimens.



**Fig. 8** Self-Repairing (cracking pattern) of SH – FECM: a) 3PB pre-cracked, healed crack; b) retesting: new failure crack for SS-L-PVA-FA/C (1.2) specimen; c) 3PB pre-cracked, healed crack; d) retesting: new failure crack for FSS-SL-PVA-FA/C (1.2) specimen

Both composites show similar, increased SH potential, both related to crack closing (Fig. 8 and Fig. 9) but also in terms of mechanical regeneration capacity of the material.



**Fig. 9** Optical microscopy analysis of the cracking network of the SH FSS-SL-PVA-FA/C (1.2) specimen - a) 3PB pre-cracked; b) 3PB pre-cracked, healed crack c) retesting: new failure cracks

## 4 Summary and conclusions

The experimental study regarding the mix design optimization approach for the SH-PVA-FECM, by replacing the limestone filler (L) with slurry limestone paste (SL), a waste generated by local industry, generally proves positive results. The fresh state aspect of the mixes shows clear improvement of the SL mixture, more cohesively than the initial L mixture. At the same time, the fresh state features of the FSS-SL-PVA-FA/C (1.2) indicate future possible reduction of liquid content. This approach is an on-going study for material optimisation.

In terms of mechanical performance, the materials indicate a similar range of values which encourage the use of the SL waste as addition to the high performance composites. The Self-healing capacity is also comparable, supporting the viability of the replacement.

Another important conclusion of the study is the good match within the SH-FECM matrix of the class F fly ash (FA) and the Limestone slurry (SL), which develops good overall performance, both regarding the fresh state and also the hardened state of the material. This could open the path of a high ecologically impacted valorisation of both additions: the fly ash and also the slurry limestone, with increased beneficial, environmental effect.

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