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# Mortars with crystalline additive in aggressive environments

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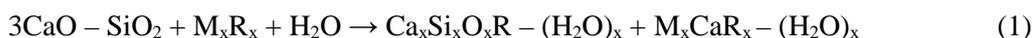
**Abstract.** The paper describes how a crystalline additive influences the properties of cement-based composites. The investigation was focused on the degradation of mortars with crystalline additive exposed to selected aggressive environments. The specimens were tested for compressive and flexural strength. The influence on the microstructure is also discussed (high pressure mercury intrusion porosimetry, SEM, DTA). The results confirmed the positive influence of a crystalline additive on the values of compressive strength after exposure to all aggressive environments.

## 1. Introduction

Nowadays, concrete is the most widely used construction material, and therefore improving its properties remains an important issue [1,2,3]. Cement composites usually consist of cement, water, aggregates, admixtures, additives and air. The binder is a continuous phase in the cement composite, thus the fact that it is porous is critical in terms of the transport of water and chemical substances in or out of the concrete [4]. One of the progressive technologies for cement based composites protection is the use of coatings, sprays or additives on so-called “secondary crystallization bases” also known as “crystalline technology” base [3]. The dry crystalline products are powder compounds that consist of Portland cement, quartz sand, and a compound of active chemicals [3,5].

In principle, crystalline materials work so that the chemical components react with the cementitious matrix during hydration, temporarily forming  $\text{Ca}(\text{OH})_2$  followed by the formation of disilicate and polysilicate anions. Available knowledge suggests that the cumulated process is accompanied by the formation of  $3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}$  together with the production of  $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 13\text{H}_2\text{O}$  [5].

Laboratory study [2] has shown, using X-ray fluorescence spectrometry (XRF) analysis, the resulting needle-like crystals likely comprise calcium or silicon [2]. The fine insoluble crystals seal the pore structure [6]. Authors [7] described a general process, where a general crystalline promoter  $\text{M}_x\text{R}_x$  reacts with tricalcium silicates and water to produce modified calcium silicate hydrates and a pore-blocking precipitate  $\text{M}_x\text{CaR}_x - (\text{H}_2\text{O})_x$  by the following equation (1):



Similar process may exist involving the calcium aluminates. The resulting crystalline deposits become integrally bound with the hydrated cement paste and become a permanent part of the cement-based composites [7].

The objectives of the work was to study the degradation of mortars with crystalline additive exposed to selected aggressive environments.



## 2. Experiment

The experiment was focused on evaluating the influence of crystalline additive on physical-mechanical properties and microstructure of mortars containing a crystalline additive and polypropylene fibres. In order to observe the degradation of the cementitious composite, specimens were exposed to an aggressive environment.

### 2.1. Materials

This study used Portland cement CEM I 42.5 R as binder, crystalline additive, quartz sand and polypropylene fibres. The chemical composition of the crystalline additive (CA) is shown in table 1. CA was added in an amount of 0.5 and 1.5 % of cement weight, see table 2.

**Table 1.** Chemical composition of crystalline additive.

Chemical composition of CA [% wt.]									
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>	K <sub>2</sub> O	Na <sub>2</sub> O	Cl <sup>-</sup>	Organic Substances
11.5	2.34	1.74	45.7	0.734	2.050	0.387	6.61	0.026	7.03

**Table 2.** Composition of the mortars per m<sup>3</sup>.

Mixture ID	Cement CEM I 42.5 R [kg]	Sand [kg]	CA [kg]	PP Fibres [kg]	Water [L]
CA0	512	1535	0	0	256
CA0.5	511	1533	3	0	256
CA1.5	510	1531	8	0	255
FCA0	511	1533	0	1	256
FCA0.5	511	1532	3	1	255
FCA1.5	510	1539	8	1	255

### 2.2. Methods

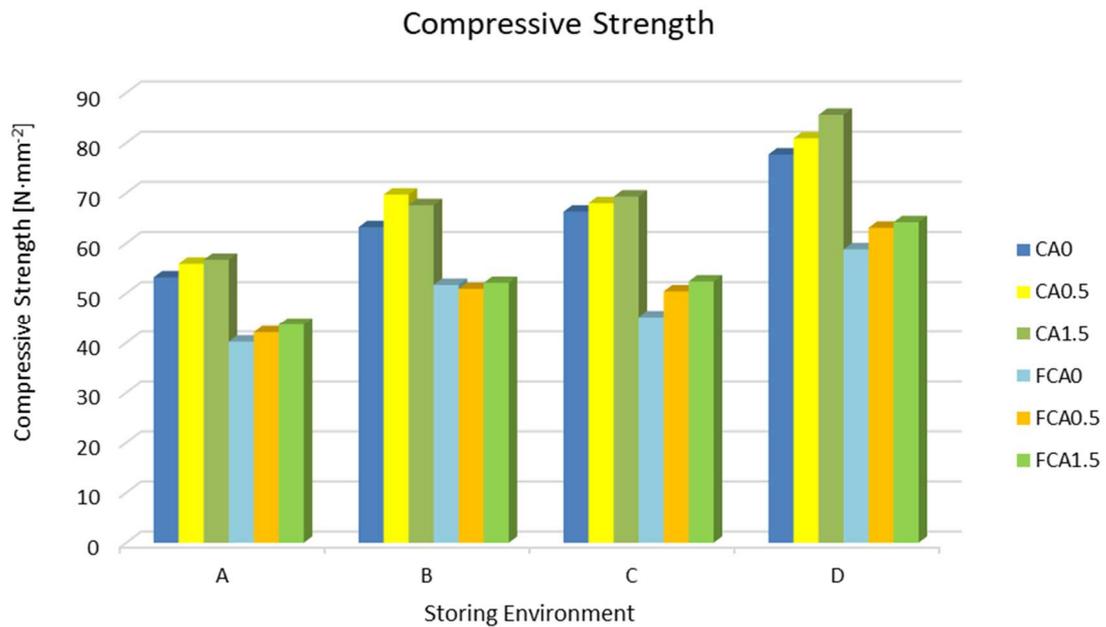
Specimens of mortars with the dimension of 40×40×160 mm were made. Porosity was determined by high pressure mercury intrusion porosimetry using a Thermo Finnigan POROTEC of the Pascal 140 – 240 type. DTA analysis was performed with a Mettler Toledo TGA/DSC 1 and SEM imaging with a TESCAN MIRA3 XMU. All specimens were stored 28 days at a temperature of 23 °C and relative humidity of 95 % and then under selected environments (A, B, C, D) which are shown in table 3.

**Table 3.** Curing environments.

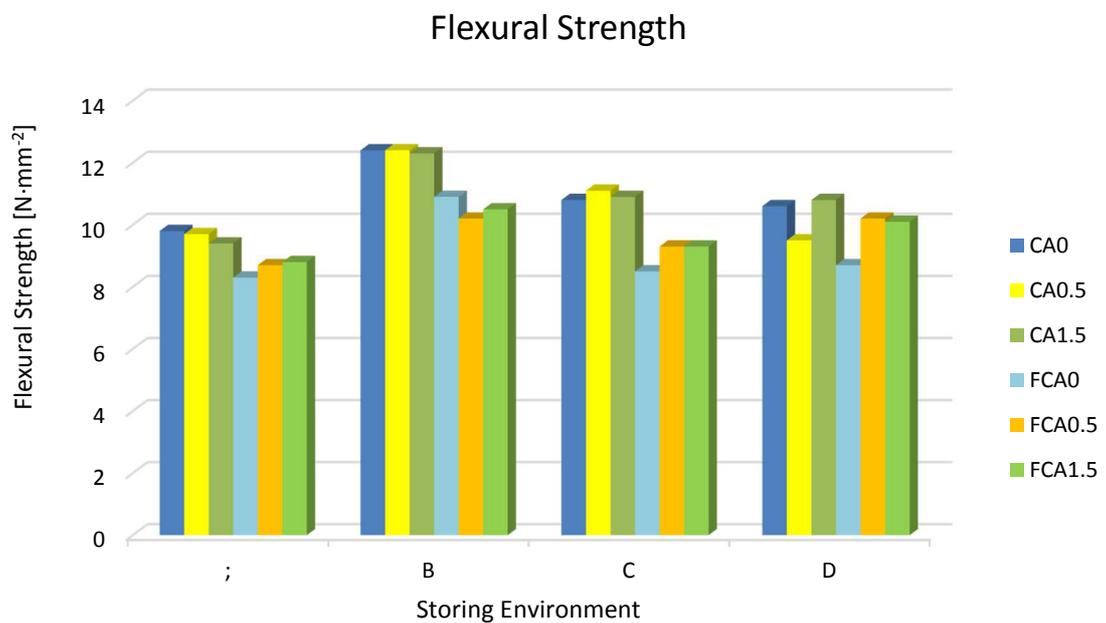
Environment ID	Curing characteristic
A (23/95)	90 days stored at a temperature of 23 °C and relative humidity of 95 % (23/95)
B (Na <sub>2</sub> SO <sub>4</sub> )	90 days in sodium sulphate solution (concentration of 36 000 mg·L <sup>-1</sup> of SO <sub>4</sub> <sup>2-</sup> )
C (NH <sub>4</sub> Cl)	90 days in ammonium chloride solution (concentration of 3 000 mg·L <sup>-1</sup> of NH <sub>4</sub> <sup>+</sup> )
D (CO <sub>2</sub> )	90 days in gaseous CO <sub>2</sub> at the concentration of φ 65 % and 75 % relative humidity

## 3. Results and Discussion

Figure 1 and 2 below show the results of compressive and flexural strength tests performed at an age of 118 days (23/95), 28 days (23/95) + 90 days in sodium sulphate environment, 28 days (23/95) + 90 days ammonium chloride environment and 28 days (23/95) + 90 days in gaseous CO<sub>2</sub>.

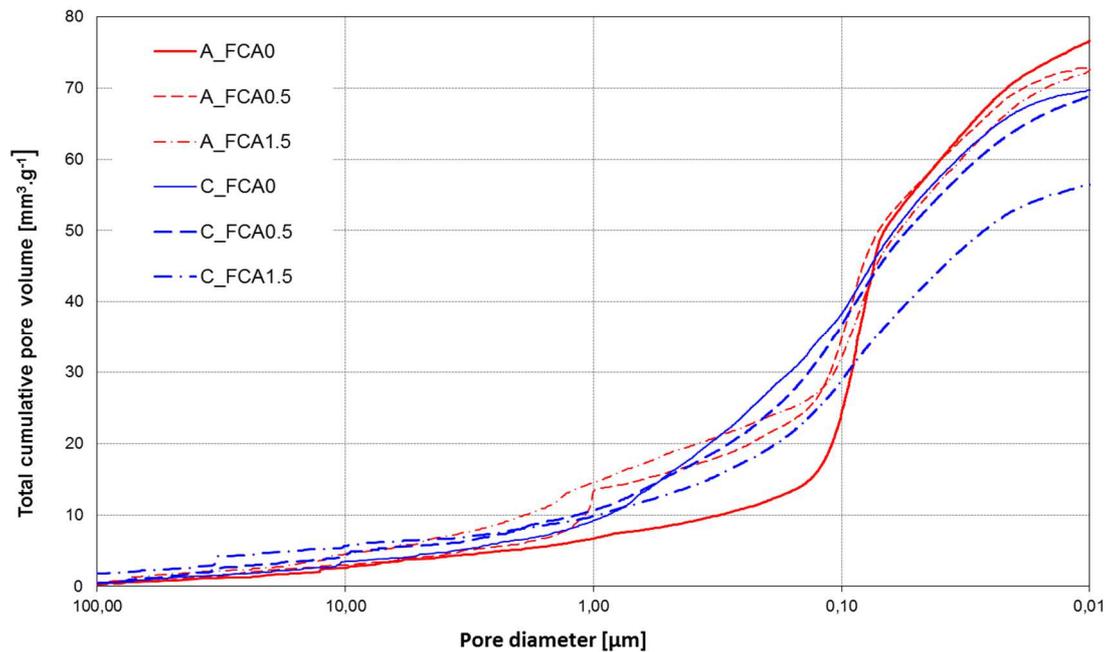


**Figure 1.** Determination of 118-day compressive strength of the mortars.



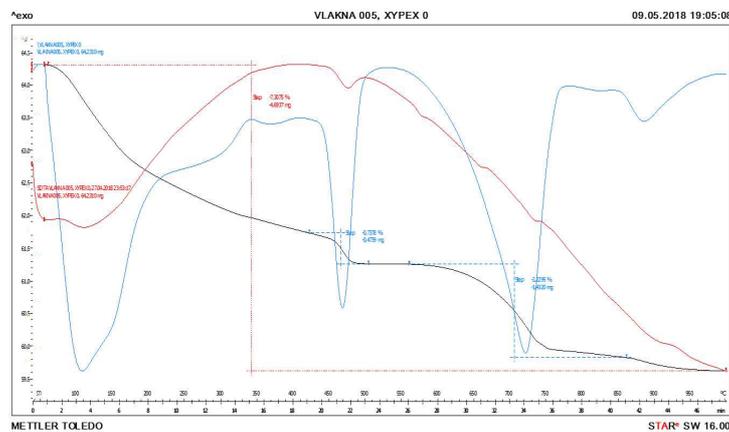
**Figure 2.** Determination of 118-day flexural strength of the mortars.

Figure 1 and 2 show that the addition of crystalline additive has a positive effect on the compressive strength values of mortars both with and without polypropylene fibres.

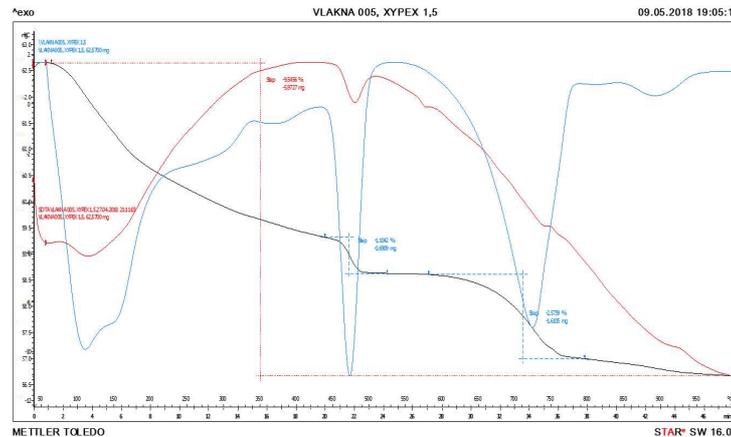


**Figure 3.** Pore size distribution of mortars stored at a temperature of 23 °C + relative humidity of 95 % (environment A) and in ammonium chloride solution (environment C).

Figure 3 shows the influence of the crystalline additive on the porosity of mortars containing polypropylene fibres stored in environment A and C. The data shows that the greatest reduction in porosity as a result of CA addition was in specimen C\_FCA1.5, which contained 1.5 % of CA and was exposed to an ammonium chloride solution.



**Figure 4.** DTA record of the mixture FCA0 stored in ammonium chloride solution.

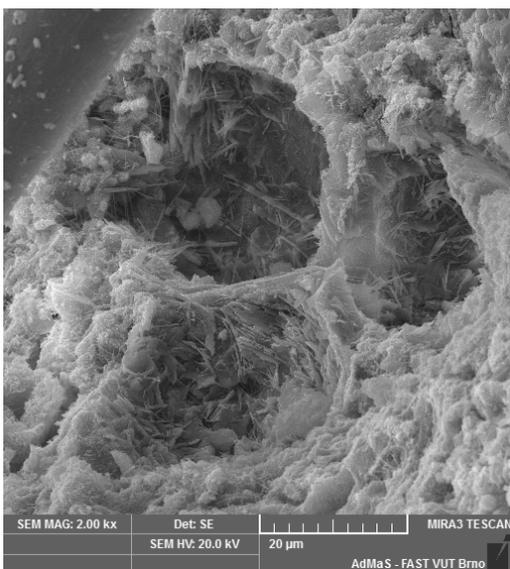


**Figure 5.** DTA record of the mixture FCA1.5 stored in ammonium chloride solution.

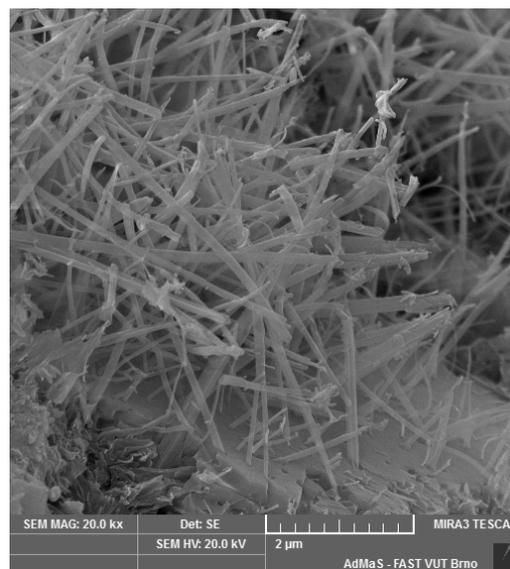
Figures 4–5 show DTA (heating rate 20 °C per minute; air atmosphere) records of mortars containing polypropylene fibres without CA (mixture FCA0) and with CA (mixture FCA1.5). Both mortars stored in ammonium chloride solution underwent three basic reactions: decomposition of C-S-H phase,  $\text{Ca(OH)}_2$  and  $\text{CaCO}_3$ . The results indicate that the crystalline additive has an influence on the increase of the amount of portlandite. Mass content of  $\text{Ca(OH)}_2$  in the mixture FCA0 was 3.03 % and in the mixture FCA1.5 was 4.54 %, see table 4.

**Table 4.** Results of DTA – mass loss and mass content.

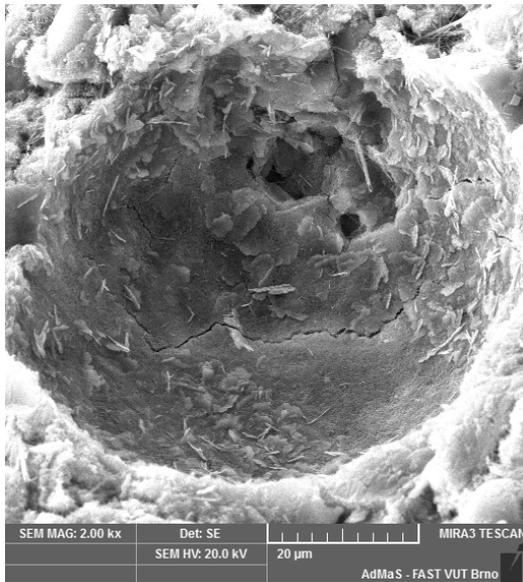
Mixture ID	Mass loss corresponding to decomposition of $\text{Ca(OH)}_2$		Mass content of $\text{Ca(OH)}_2$	
	[%]	mg	[%]	mg
FCA0	0.74	0.47	3.03	1.95
FCA1.5	1.10	0.69	4.54	2.84



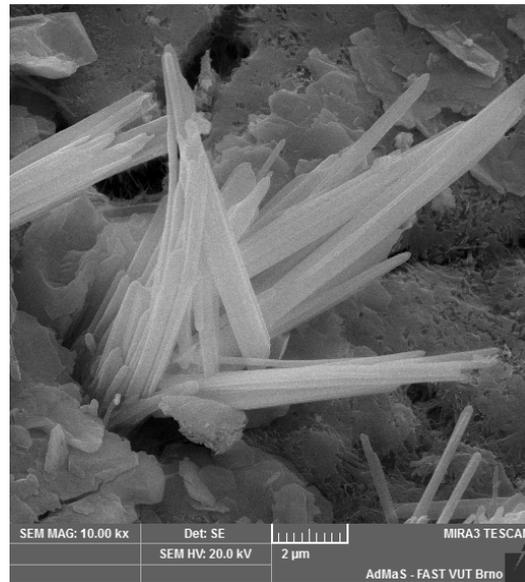
**Figure 6.** Picture of a specimen with CA (FCA1.5) stored in ammonium chloride solution.



**Figure 7.** Detail of newly formed CA (FCA1.5) in a specimen stored in ammonium chloride solution.



**Figure 8.** Picture of a specimen without CA (CA0) stored in ammonium chloride solution.



**Figure 9.** Detail of ettringite needles found in a specimen (CA0) stored in ammonium chloride solution.

Figure 6–9 show differences in the microstructure of specimens stored in an ammonium chloride solution. Mortars containing CA have clearly visible needle-like products, see figure 7.

#### 4. Conclusion

The tests confirmed the positive influence of a crystalline additive on the values of compressive strength after exposure to all aggressive environments. Mortars which contained polypropylene fibres also saw an increase in flexural strength. The addition of polypropylene fibres can negatively influence the porosity of hardened mortars, while a higher CA content, i.e. 1.5 %, can bring about a reduction in porosity in mortars exposed to aggressive environments. The research results confirm that CA has an influence on the increase of the amount of portlandite. SEM sampling revealed differences in the microstructure of mortars both with and without crystalline additives after exposure to aggressive environments.

#### Acknowledgment

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