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# The Influence of Crystallization Additives on the Porosity of Polypropylene Fibre-reinforced Cement Mortar

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**Abstract.** The phenomenon of self-healing cement materials has been described in the professional literature for decades. One self-healing mechanism is based on the presence of crystallization additives in concrete or mortar; when a defect appears in the material, the crystallization additives (CA) are activated, and new crystallization products will fill the pore or crack. The activation of the crystallization additives occurs only under certain conditions. The intensity of crystallization is affected by the nature of the environment within the crack or pores, and in order for the crystallization to occur, the cracks or pores must contain an aqueous solution with sufficient ion content to react with the crystallization additive. This paper discusses the possibility of using crystallization additives to reduce the porosity of cement mortar reinforced with polypropylene (PP) fibres, describes the effect of crystallization additives on the pore structure of cement mortar, and evaluates the influence of the medium on pore size distribution in cement mortar.

## 1. Introduction

Crystallization additives are added to concrete or mortar to extend the life of a building's structure. If a defect, crack, or tear occurs, crystallization additives present on the surface of the structure mean that if water enters the crack, the minerals react with  $\text{Ca}(\text{OH})_2$  to form new crystals, gradually filling the defect [1]. The presence of these mineral additives in the mortar ensures secondary crystallization, the products of which fill the defect, and prevents the spread of cracks and the entrance of aggressive substances to the structure, thereby helping to avoid decreases in strength during the life cycle of the structure. For the crystallization additive, it is common to use carbonates of  $\text{NaHCO}_3$  or  $\text{Na}_2\text{CO}_3$  [2], talc [3], or sodium silicate [4].

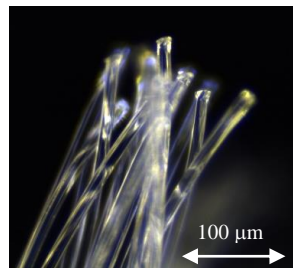
However, use of CA creates a number of problems. The mineral additive is applied directly to the concrete or cement mixture without any protection and soon as it comes into contact with water, the mixture immediately begins to react [5]. As a result, these mineral additives are used prior to the hardening of the cement material, and so their presence in the concrete loses importance, where self-healing is concerned. In order to keep the additive unreacted in hardened concrete, it is necessary to modify it before mixing so as to avoid an immediate reaction with water. Various techniques for the treatment of CA [6, 7, 8] have been described in the literature. The basic condition for secondary crystallization is the constant presence of water; the ions contained in the water exert a considerable influence on the physical and chemical processes, and affect the rate and intensity of the secondary crystallization. This issue is not yet described in the literature, but this fact should be taken into account in further research on self-healing in cement materials.



In this case of the present study, these CA were used to reduce the porosity of a PP-reinforced cement mortar in order to eliminate problems with the premature reaction of the crystallization additive. The PP fibres in the mixture reduce stress in the initial phase of the solidification, reduce plastic sedimentation, and prevent microcracks from forming in the first phase of solidification. The fibre-reinforced mortar also exhibits a higher tensile bending strength. However, the addition of fibres causes an increase in the apparent porosity of the material, a decrease in the bulk density, and thus a decrease in compressive strength [9]. The fibres have both a positive and a negative influence on the material.

## 2. Materials and methods

The experiment described here employed a secondary crystallization additive designed for cement-based mortars. The specimens were divided into two groups; the first batch of specimens was used to observe the effect of a CA, and the second to study the influence of the CA in combination with polypropylene fibres.



**Figure 1.** Fibrin polypropylene fibres, with a fibre diameter of 20 micrometres

The cement used as a binder was Portland cement CEM I 42.5 R (Českomoravský cement a.s., Mokr á factory). Sand was used according to EN 196-1, and XYPEX Admix (XYPEX CHEMICAL CORPORATION, Richmond, B.C., Canada) was used as the crystallization additive. The second batch of specimens was made with polypropylene fibres Fibrin 3/15 (KrampeHarex CZ, s.r.o.), Fig 1.

The composition of the mortars tested is shown in Table 1. PP fibres were dosed at 0.0%, 0.5%, and 1.5 % by weight of cement, and the fibre content in the formulation was 0.05%. Please follow these instructions as carefully as possible so all articles within a conference have the same style to the title page. This paragraph follows a section title so it should not be indented.

**Table 1.** The composition of the mortars

Mixture ID	Cement CEM I 42.5 R [g]	Sand [g]	CA [g]	PP Fibres [g]	Water/cement ratio
REF	450	1350	0.0	0.0	0.5
0.5			2.25		
1.5			6.75		
REF*			0.0	0.9	
0.5*			2.25		
1.5*			6.75		

The pore structure of the specimens was determined at the following ages, and after being treated by the following curing techniques:

- **H<sub>2</sub>O** 28+90days (the specimens were stored at a temperature of 23 °C and a relative humidity of 95% for 28 days, and then for another 90 days).
- **SO<sub>4</sub>** 28+90days in an aqueous solution of Na<sub>2</sub>SO<sub>4</sub> (the specimens were stored at a temperature of 23 °C and 95% relative humidity for 28 days, after which they were moved into the Na<sub>2</sub>SO<sub>4</sub> solution with a concentration of 36,000 mg/l SO<sub>4</sub> ions, where they were left for 90 days).
- **NH<sub>4</sub>** 28+90 days in an aqueous solution of NH<sub>4</sub>Cl (the specimens were stored at a temperature of 23 °C and 95% relative humidity for 28 days, after which they were moved into the NH<sub>4</sub>Cl solution with the concentration of 3,000 mg/l NH<sub>4</sub> ions, where they were left for 90 days).

- **Lab** 28+180 days (the specimens were stored at a temperature of 23 °C and 95% relative humidity 28 days, after which they were moved into laboratory conditions with a temperature of 20 °C and 55% relative humidity).

Apparent porosity, average pore diameter, bulk density, and pore size distribution were determined by high-pressure mercury intrusion porosimetry, using a ThermoFinnigan POROTEC of the Pascal 140 – 240 type. Bulk density, apparent density, and apparent porosity were determined using the hydrostatic gravity test (ČSN EN 993-1).

### 3. Selected results and discussion

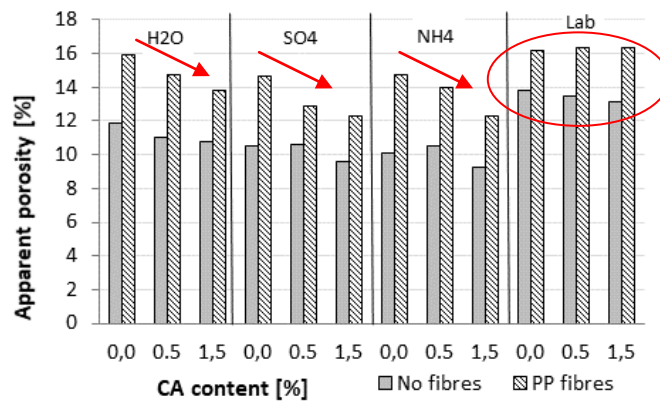
The results obtained will be discussed here, together with the results published in [10]; namely, the properties of the cement mortar prepared according to the same formulation and stored for 28+180 days in a laboratory environment. These samples were labelled Lab or Lab\*, depending on whether they contain PP fibres.

Firstly, the effect of PP fibres on the pore structure of the cement mortar is assessed. Although the fibres are only dosed at 0.05 wt. % on the cement, and their effect on the open pore content of the material is clearly visible. The apparent porosity of the mortar increased by up to 43%, as shown in Table 2. The increase in porosity is related to the deterioration of the cement mortar's workability, and is also due to the fact that air is being drawn into the mixture with the fibres, and that the air cannot escape from the mixture.

**Table 2.** Results of the mercury intrusion porosimetry test

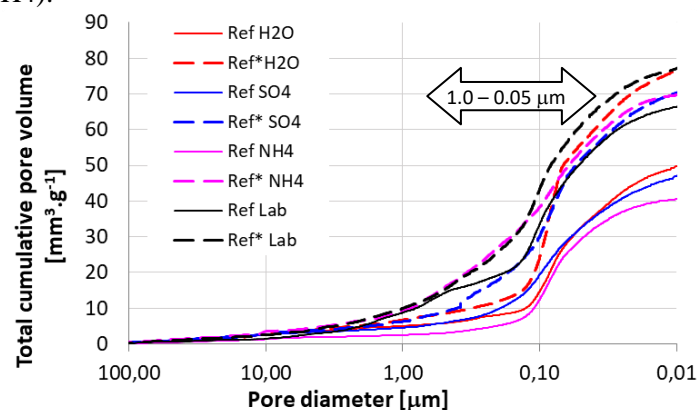
RESULTS	H <sub>2</sub> O						SO <sub>4</sub>					
	REF	0.5	1.5	REF*	0.5*	1.5*	REF	0.5	1.5	REF*	0.5*	1.5*
Average pore diameter (µm)	0.081	0.083	0.087	0.090	0.100	0.085	0.103	0.078	0.084	0.073	0.092	0.088
Apparent porosity (%)	11.9	11.1	10.8	15.9	14.7	13.8	10.5	10.6	9.6	14.7	12.9	12.3
Porosity increase (%)				<b>34.1</b>	<b>32.9</b>	<b>28.3</b>				<b>40.1</b>	<b>22.2</b>	<b>28.9</b>
Bulk density (g/cm <sup>3</sup> )	2.176	2.213	2.176	2.076	2.016	2.105	2.207	2.232	0.450	2.081	2.141	2.148
Apparent density (g/cm <sup>3</sup> )	2.442	2.488	2.499	2.469	2.364	2.486	2.466	2.495	0.460	2.439	2.458	2.450
RESULTS	NH <sub>4</sub>						Lab					
	REF	0.5	1.5	REF*	0.5*	1.5*	REF	0.5	1.5	REF*	0.5*	1.5*
Average pore diameter (µm)	0.082	0.085	0.084	0.084	0.102	0.092	0.103	0.102	0.099	0.102	0.103	0.075
Apparent porosity (%)	10.3	10.5	9.2	14.7	13.8	12.3	13.8	13.5	13.2	16.2	16.3	16.3
Porosity increase (%)				<b>42.8</b>	<b>39.5</b>	<b>33.3</b>				<b>17.4</b>	<b>21.1</b>	<b>24.0</b>
Bulk density (g/cm <sup>3</sup> )	2.187	2.223	2.228	2.108	2.131	2.177	2.151	2.172	2.091	2.092	2.112	2.080
Apparent density (g/cm <sup>3</sup> )	2.401	2.485	2.455	2.471	2.499	2.483	2.511	2.510	2.407	2.497	2.524	2.486

The difference in porosity between the PP-reinforced and non-reinforced mortar is shown in Fig. 2 below, while it is also possible to assess the influence of the environment in which the samples were stored. The apparent porosity of the samples is highest when stored in a lab environment (Lab), even though those samples are 90 days older than the others. The porosity varies between 13-14% for the fibre-free specimens and 16% for the fibre specimens. When stored at a relative humidity of 95% (H<sub>2</sub>O), the porosity is lower, about 11% in the samples without PP fibres, and 14-16% in the fibre samples. The lowest porosity was found in the samples stored in an aqueous solution (SO<sub>4</sub>, NH<sub>4</sub>). The apparent porosity is 9-10.5% for non-reinforced mortar and a maximum of 14.7% for reinforced mortar.



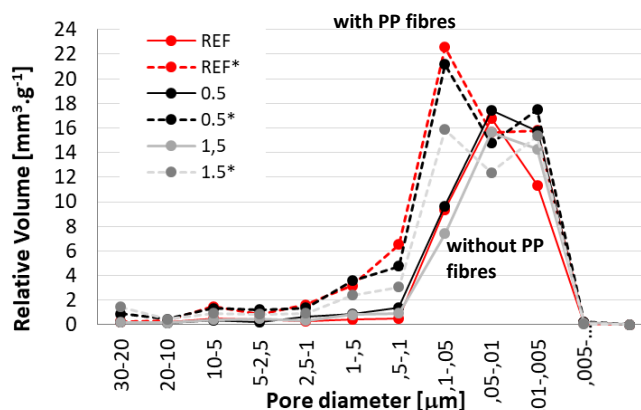
**Figure 2.** The effect of the crystallization additive on the apparent porosity of a mortar being hardened in different environments

The addition of fibre causes an increase in pore content of 1.0-0.05  $\mu\text{m}$  in all environments, as shown in Fig. 3. These pores are then filled with secondary crystallization products. In the laboratory environment, this secondary crystallization almost does not occur because there is insufficient moisture in the pores, which is necessary for the reaction of the crystallization additive, as shown in Fig. 2. The dose of the crystallization additive did not influence porosity in the laboratory environment. A different result was observed for the mortar stored at 95% relative humidity. The addition of CA reduced the apparent porosity in the samples with PP fibres to 13%, while in the case of fibre-free samples, the porosity is even lower. The same trend was also seen for the mortars stored in aqueous solutions (SO<sub>4</sub> and NH<sub>4</sub>).



**Figure 3.** The effect of crystallization additive on pore size distribution

One example of this is the decrease in apparent porosity in the reinforced and non-reinforced mortar deposited in NH<sub>4</sub>Cl solution. The apparent porosity of PP-free mortar was reduced from 10.3% to 9.2%, and with the fibre mortar from 14.7% to 12.3%. The reaction products gradually fill up the pores that have been created in the mortar through the addition of PP fibres, i.e. pores sized 1-0.05  $\mu\text{m}$ . The higher the CA dose of the additive, the higher the crystallization products and the lower the porosity, as can be seen in Fig. 4.



**Figure 4.** The pore size distribution of cement mortar being hardened in an aqueous solution of  $\text{NH}_4\text{Cl}$

#### 4. Summary

The present study examined the effect of the crystallization additive XYPEX on the pore structure of cement mortar. This is a mineral additive that is primarily used for the self-healing component of cement materials. In our case, its influence on the apparent porosity of mortar reinforced with PP fibre has been verified.

The cement mortar was prepared according to a standard procedure; the samples were stored for 28 days at a relative humidity of 95% and subsequently transferred to different environments, where they were hardened for 90 and 180 days, respectively.

The pores formed in the mortar after the addition of PP fibres can be reduced by adding the XYPEX crystallization additive. The effectiveness of the additive depends on its dosing, as well as on the setting and hardening conditions of the cement mortar. This study has also verified that the use of an additive is not suitable for laboratory samples that do not allow the crystallization reaction to start. Conversely, a high relative humidity or aqueous solution environment is suitable for use with XYPEX; the CA dose of 1.5% by weight of cement reduced the porosity of the PP fibre mortar by up to 27%. The porosity of 12.3% found in this case approximates the porosity of the unreinforced mortar.

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