Efficiency of the crystallizing waterproofing admixture in lower-quality concrete

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Abstract. The article deals with the topic of a crystallizing waterproof admixture using in concrete. This type of admixture is designated for making a hardened concrete more durable by minimizing its permeability. Considering that concretes of higher strength classes meet the requirements for durability parameters in most cases, in this experiment 3 low-durability (in terms of high w/c ratio) concrete (Dmax8) batches were mixed. From each of mixture, 2 sets of samples (with and without the admixture) were prepared for depth of penetration of water under pressure test, strength test and water absorption test after 28 days of curing. Likewise, samples for microscopic visual assessment of crack-filling process were produced. Cracks onto these samples were intentionally introduced after their curing in time of 7 days and subsequently were further cured under water and regularly observed. According to the results, tested admixture acted more counter-productive than helpfully.

1. Introduction
It is well-known and widely accepted that concrete is, due to its technical parameters as well as for price, one of the most, if not the most, used building material in structure assembling. On the other hand, there are some aspects that must be managed to ensure durability and long-term resistance of concrete structure [1, 2] in accordance with requirements of sustainability in civil engineering [3]. The cracking-problem is inherent of concrete, that mainly resides in the low ability of fresh concrete to resist of tensile stress as well as hardened concrete [4, 5, 6]. Also, permeability and porosity are inseparably connected with concrete microstructure and consequently with durability of concrete [7]. All these parameters influence concrete resistance against deterioration. One of the current trends of concrete technology is designing concrete not only with high mechanical properties, but also with environmental added value [8, 9, 10]. For this purpose, research and development is focused on preparing modern additives and admixtures for cement and concrete with the aim to make the concrete microstructure denser with low and very low permeability and porosity or intentionally porous by entraining microparticles (mainly for freeze-thaw resistance) [11, 12]. The process of crack forming and developing is very complex and has several reasons such as drying up including plastic shrinkage, heat development and autogenous shrinkage, plastic settling or overloading [13]. This problem can be handled by technological-operational curing processes against interface temperature volatility, windy environment or fast water evaporation from fresh concrete. The actions for decreasing (capillary) porosity and permeability reside in using pozzolanic or latent-hydraulic mineral admixtures [14]. Another opinion is based on using air-entraining admixture as well as waterproofing admixture based on crystalline technology [15, 16, 17, 18, 19, 20].

In this paper, one type of crystallizing waterproof admixture is the object of experimental investigation. Nowadays, it is widely used admixture in concrete for slab foundation and in underground structure, where pressure water occurs. In technical sheets of this kind of admixture can be found, that
it has positive effects on lowering the depth of penetration of water under pressure, but also about their ability to make self-healing product by reacting with cement paste under the wet condition. The chemical composition of this admixture is designed for creating the insoluble crystal forms in capillary system of concrete. For this purpose, moisture in concrete is necessary. Considering that most of the concretes of higher strength class and with low cement/binder ratio are able to meet the requirements of durability parameters on itself without using extra additive/admixture, experimental investigation is focused on the concretes with higher w/c ratios (considering local relations).

2. Materials and methods

For the experiment, 3 various batches with two sets of samples (with and without crystalline admixture) were mixed. Consistency by cone-slump and density of fresh concrete were tested as well as crack-filling, density, compressive strength, splitting tensile strength, depth of penetration of water under pressure test and water absorption test were performed on hardened concrete.

2.1. Concrete mix designs

For preparing fresh concretes, cement CEM II/A-S 42.5 R (“CEM”), tap water (“WAT”), natural aggregate fraction (“AGG”) 0/2, 0/4 and 4/8, superplasticizer based on PCE (“PCE”) and crystalline admixture (“CA”) were used. Overall 3 batches were mixed. Within every batch, reference samples were made and then CA in amount approximately 7.35 kg.m\(^{-3}\) were added (“+”), batch remixed and samples with CA produced. Labelling with mix compositions are given in table 1.

Concrete mix compositions were designed with aim to prepare 3 various strength classes by using higher content of cement together with decreasing w/c ratio.

**Table 1. Concrete mix designs.**

| Material (wt %)                  | M1  | M1+ | M2  | M2+ | M3  | M3+ |
|---------------------------------|-----|-----|-----|-----|-----|-----|
| CEM                             | 10.00 | 10.85 | 11.65 |
| WAT (total)                     | 8.35 | 8.00 | 7.65 |
| W/C total ratio (excl. PCE and CA) | 0.835 | 0.740 | 0.655 |
| AGG 0/2                         | 2.45 | 2.45 | 2.40 |
| AGG 0/4                         | 42.40 | 42.15 | 41.90 |
| AGG 4/8                         | 36.70 | 36.50 | 36.30 |
| PCE (wt % of CEM)               | 0.78 | 0.91 | 0.96 |
| CA (wt % of mix)                | 0.000 | 0.175 | 0.000 | 0.175 | 0.000 | 0.175 |

2.2. Fresh concrete properties

Fresh concrete consistency (“S”) was measured after 5 minutes from first contact of cement and water (for M1, M2 and M3), respectively immediately after CA addition and concrete remix (for M1+, M2+ and M3+). Consistency was performed as cone slump test in 3 layers, each compacted by 25 rod strikes. Fresh concrete density (“\(D_{\text{mix}}\)”) was performed in cylindric mould (slimness 1:2) with volume of 5.3 litres. \(D_{\text{mix}}\) was also controlled on hardened concrete after demoulding and taking into account evaporated water.

2.3. Hardened concrete curing and properties

Produced samples were cured in laboratory conditions for 1 day protected against water loss. After demoulding, samples were cured in water bath till the test execution.

On 7\textsuperscript{th} day, crack with width <0.4mm was intentionally introduced into the samples, for later observation of the self-healing effect. These samples were consequently cured in water again till 28\textsuperscript{th} day.
Density in saturated state of hardened concrete ("D_{sat}\) was calculated as ratio of weight and measured dimensions of saturated samples, density in dried state were determined on samples that underwent water absorption ("WA") test.

Compressive strength ("f_{c}\) in age of 28 days was performed on water saturated surface dry (SSD) cube (a = 100 mm).

WA test were performed on cubes (a = 100 mm), that were dried in the laboratory oven at 105±5 °C for 7 days, until constant mass was reached. Water absorption was calculated as ratio of absorbed water and dried mass. Samples after WA test also underwent compressive strength test ("f_{c,WA}\).

Maximum depth of water under pressure were determined on cubes (a = 150 mm), when pressure of water 500±50 kPa (5 bars) were applied for 72 hours and then measured the height ("h_{max}\) from the bottom, where pressure water reach the maximum. At the end, splitting tensile test ("f_{ct,sp}\) were performed.

3. Results and discussion

3.1. Fresh concrete properties

Results of consistency and fresh concrete density are given in table 2. Comparing batches with and without CA, it is obvious that consistency decrease within each batch (for M1 – 50 mm, for M2 – 60 mm and for M3 – 80 mm), where CA is applied despite the fact, that CA was applied in liquid form. The density of fresh concrete also decreased when CA was added (for M1 – 15 kg.m\(^{-3}\), for M2 – 20 kg.m\(^{-3}\) and for M3 – 30 kg.m\(^{-3}\)), what could be caused by adding extra water from CA. Workability behaviour of fresh concrete with CA has character like slightly aerated concrete.

For adjustment consistency it would be necessary to add mix water, what should decrease the durability of concrete or add PCE what would make the 1 m\(^3\) more expansive.

| Parameter | M1 | M1+ | M2 | M2+ | M3 | M3+ |
|-----------|----|-----|----|-----|----|-----|
| S [mm]    | 80 | 30  | 100| 40  | 140| 60  |
| D_{mix} [kg.m\(^{-3}\)] | 2345 | 2330 | 2340 | 2320 | 2395 | 2365 |

3.2. Hardened concrete properties

Results of measured parameters are given in table 3. The trend of density decrease with CA addition is obvious also for D_{sat} (for M1 – 20 kg.m\(^{-3}\), for M2 – 20 kg.m\(^{-3}\) and for M3 – 30 kg.m\(^{-3}\)) and D_{dry} (for M1 – 15 kg.m\(^{-3}\), for M2 – 20 kg.m\(^{-3}\) and for M3 – 35 kg.m\(^{-3}\)).

In each pair of samples, increase of WA by 0.1% (abs) can be seen, what could be caused by CA addition due to its liquid form and slightly w/c ratio increase. WA varied as supposed, the lowest values reached the concretes (M1 and M1+: 6.4 and 6.5 wt %) with the lowest w/c ratio, the highest the concretes (M3 and M3+: 7.6 and 7.7 wt %) with the highest w/c ratio. Values of penetration height of pressure water were observed in the same way: 9 and 15 mm for M3 and M3+; 20 and 24 mm for M1 and M1+ as well as splitting strengths 3.35 and 3.10 MPa for M3 and M3+; 2.75 and 2.60 MPa for M1 and M1+.

| Parameter | M1 | M1+ | M2 | M2+ | M3 | M3+ |
|-----------|----|-----|----|-----|----|-----|
| D_{sat} [kg.m\(^{-3}\)] | 2370 | 2350 | 2360 | 2340 | 2415 | 2385 |
| D_{dry} [kg.m\(^{-3}\)] | 2200 | 2185 | 2205 | 2185 | 2275 | 2240 |
| WA [wt %] | 7.6 | 7.7 | 7.1 | 7.2 | 6.4 | 6.5 |
| h_{max} [mm] | 20 | 24 | 19 | 20 | 9 | 15 |
| f_{ct,sp} [N.mm\(^{-2}\)] | 2.75 | 2.60 | 3.00 | 2.55 | 3.35 | 3.10 |
The decrease of compressive strength when CA was added can be also observed. Relative decrease of compressive strength for M1 was -13.3%, for M2 -12.1%, and for M3 -12.2%, what could be assumed to be similar decrease. This trend was also approved on the dried sample. Significant increase in compressive strength after drying the samples was also achieved. Despite the fact, that this issue is not the object of this experimental investigation, it can be noted, that $f_c$ increase in this way: +53.0% for M1, +60.7% for M1+, +49.2% for M2, +47.3% for M2+, +47.5% for M3 and +50.0% for M3+.

In the figure 1 and 2 digital microscopy pictures on 7th day (as reference time) and on 28th day of samples M1 and M1+ are illustrated, where no any insoluble product (excluding soluble efflorescence limestone leaking out through cracks) in cracks was observed (the same was observed for all other samples). Thus, in this experimental investigation with pre-set conditions of testing, the self-healing effect of CA was not confirmed.

| $f_c$ [N.mm$^{-2}$] | 28.5 | 24.7 | 35.4 | 31.1 | 44.4 | 39.0 |
|---------------------|------|------|------|------|------|------|
| $f_{c,WA}$ [N.mm$^{-2}$] | 43.6 | 39.7 | 52.8 | 45.8 | 65.5 | 58.5 |

Figure 1. Sample M1 (crack width 0.2 mm) in age of 7 days (up) and 28 days (down). Magnification: 50x (left) and 150x (right).
4. Conclusion
In this experimental study, efficiency of the crystallizing waterproof admixture in lower-quality concrete was investigated. Based on results of mechanical properties, durability parameters as well as from optical and microscopical observation can be concluding, that in this test with described materials and methods was not confirmed positive effect using this type of admixture. Contrarily, quality decrease in every testing property was achieved. However, that could be caused by increase of w/c ration due to using liquid form of CA. Before using (not only) this type admixture, it should be performed the exhaustive pre-testing, considering the balance between high economical expense of CA on the one hand and technical and environmental contribution for concrete on the other hand.

5. References
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