

KAUNAS UNIVERSITY OF TECHNOLOGY
RESEARCH CENTRE OF BUILDING
MATERIALS AND STRUCTURES
Studentų str. 48, LT - 51367 Kaunas

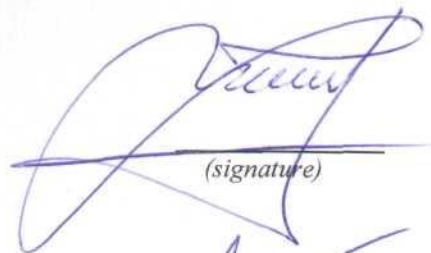
Scientific research report

**INVESTIGATION OF WATER IMPERMEABILITY CONCRETE
WITH CRYSTAL ADDITIVE**

KAUNAS, 2018

RESEARCHERS

Sen. spec. assoc. prof. dr. E. Ivanauskas



(signature)

Sen. spec. assoc. prof. dr. A. Augonis



(signature)

**Director of Building Materials and
Structures Research Centre**

Assoc. Prof. Dr. E. Ivanauskas



(signature)

The stamp is circular and contains the following text: "Kauno technologijos universitetas" (Kaunas University of Technology) at the top, "Statybos ir architektūros fakultetas" (Faculty of Building and Architecture) at the bottom, and "2018-05-31" in the center. There is also a small square stamp with the date "2018-05-31" and some illegible text.

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GOAL OF MTEP EXPERIMENTAL/SCIENTIFIC RESEARCH

The goals of the research:

1. Produce watertight self-healing concrete i.e. the concrete capable to fill up cracks occurring during exploitation by using special testing method (artificial crack till 0.3 mm formation by splitting 150×150×150 mm cube and testing self-healing effect with floating water throw crack).
2. Determine compatibility CEM I 42.5R cement with different dosages and types of additives in the designed concrete mixture compositions and evaluate the effect of crystal additives of obtained production.
3. Determine concretes, with different types and dosages of crystal additives, on fresh concrete mixture properties - air content, slump, density and temperature according EN 12350-2, 5, 6, 7.
4. Determine concretes, with different types and dosages of crystal additives, on hardened concrete properties - water penetration depth according local standard LST 1974 annex O – pass/not pass W14; water permeability test according EN 12390 T8 – when samples are kept in 1,4MPa pressure 28 days; concrete compressive strength according EN 12350 T3 after 1, 7 and 28 days
5. Determine concretes, with different types and dosages of crystal additives, on self-healing affect by using special testing method
6. Analyze quality of crack healing in damaged concrete affected by selected water column pressure (crack filling pace, maxim width of healed crack).
7. Analyze obtained results and prepare report.

The experimental researches were carried out with Sweden Cementa AB, Box 102 SE-62422 Slite, cement CEM I 42.5 R cement together with UAB “Rizgonys” quarry aggregates (sand 0/4 and gravel 4/16), Sika admixtures superplastificator Sika ViscoCrete D187 and given by Sika crystal additives: Sika WT200P and None Sika.

INTRODUCTION

In Lithuania as in the rest of the world at current time, more attention is given to possibilities to increase exploitation time of concrete construction and service life of all building. In all the world many constructions became problematic because of concrete cracks. This factor decrease concrete durability dramatically. The concrete erosion at the damaged areas in structures or cracks occurs more rapidly, because of water absorption from the environment, so the concrete and reinforcement corrosion speeds up. So, it is important for concrete to be watertight and to have no cracks, but the latter often occurs during exploitation due to the destructive environmental affect or/and mechanical loads. Through these cracks water transfers chemical elements, dissolves minerals and forms new compounds. Therefore, durability of the structures such as dams, reservoirs, parking lots or concrete floors can be reduced due to the effect of water. Diffusion of water vapour in concrete causes various problems. Kinetics of moisture penetration, for example in the ground-floor, is variable event and highly depends on environment temperature. Constructional, groundwater, hygroscopic, condensational and exploitation humidity has an effect on this sort of structures. Moisture kinetics in concrete depends on the raw material composition, newly formed structure (pore type, distribution and type of occurring cracks) and the exploitation environment.

On purpose to eliminate latter damages and improve production quality it is rational to use new type crystal additive, which reacts with minerals occurring during hydration of cement and forms stable crystals, filling resident micro and macro cracks in the entire volume of sample and, most importantly, concrete self-heals. While crystal additive is filling capillaries, it minimizes structures water absorption, increases significantly water tightness, frost resistance, and concrete structure will be possibly more resistant to chemical effect i.e. it will be more durable by all aspects. According technical literature in Fig.1 it can be seen that self-healing concrete lets to increase building service life compared to traditional concrete

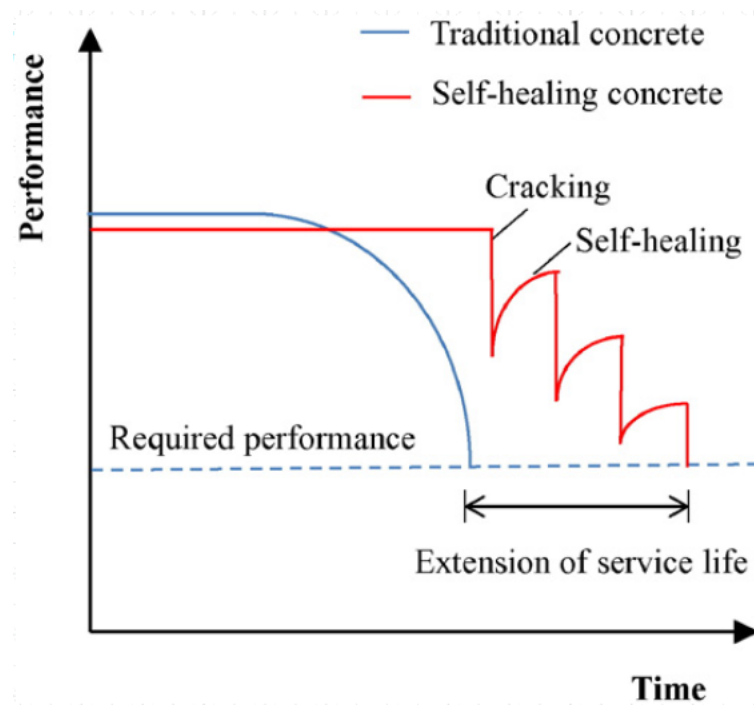


Fig. 1 Self-healing effect on exploitation time of concrete construction

The main goal of this investigation – perform scientific research properly designing and investigating compatibility of crystal additive with locally used materials and evaluating influence of fresh and hardened concrete properties. The tests were carried out in KTU SMKTC laboratory applying accredited test methods and test methods established in the world or proposed by KTU.

It is expected that the results of the investigation will help Sika Baltic company to increase competitiveness in Baltics concrete production market, because the application of the crystal additives in the concrete production will open the possibility to reduce construction costs in the long-time exploitation.

**MIXES AND MARKINGS
OF CONCRETE SAMPLES WITH CRYSTAL ADDITIVE**

To evaluate the influence of the crystal additive for fresh concrete mix and mechanical and physical properties of hardened concrete four mixture compositions were designed. Mixture design was done according SIKA recommendations to Sika Watertight concrete system (Table 1)

Table 1. SIKA recommendations for Watertight concrete

Components	Comments	Specification
Aggregate	- Balanced particle- size distribution curve required - Clean and compliant to local standard - Recycled aggregate should not be used	- Maximum size of approximately 32 mm
Cement	- Compliant with local standards	- Minimum binder content 350kg/m ³
Secondary Cement Material (SCM)	- Fly ash and ground granulated blast furnace slag only	- Maximum 40 % of total binder content
Water	- Fresh water and recycled water with requirement regarding fines. Water cement ratio according to local standards for exposure class	- Maximum 0.45
Concrete Admixtures	- Type dependent to ensure water cement ratio, initial flow workability over time	- Sika® ViscoCrete® / Sika® ViscoFlow® 0.60 – 1.50 %
	- Sika® WT to ensure watertightness	- Sika® WT 1.00 – 2.00 %

One mixture was done without crystal additives, two mixtures with crystal additive SIKA WT200P, and one mixture was prepared with crystal additive None Sika. The compositions of the concrete mixtures are given in table 2.

Table 2. The compositions and marking of the concrete mixes.

Concrete components for 1 m ³	Control	1% WT200P	2% WT200P	1% None Sika
Portland Cement CEM I 42,5R, kg	350	350	350	350
Water, l (W/C=0.45)	157.5	157.5	157.5	157.5
Sand (0/4mm), kg	861	861	861	861
Gravel (4/16mm), kg	1031	1031	1031	1031
Superplasticizer Sika ViscoCrete D187, kg	2.98 (0.85% f.c.m.)	2.98 (0.85% f.c.m.)	2.98 (0.85% f.c.m.)	2.98 (0.85% f.c.m.)
Crystal additive, kg SIKA WT200P	-	3,50 (1.00% f.c.m.)	7,00 (2.00% f.c.m.)	-
Crystal additive, kg None Sika	-	-	-	3,50 (1.00% f.c.m.)

INFLUENCE OF CRYSTAL ADDITIVE FOR PHYSICAL PROPERTIES OF FRESH CONCRETE

Slump, temperature, air content and density were measured to evaluate crystal additive influence on the concrete mixtures physical properties with different cements. The slump was measured twice – immediately after mixing and after 60 minutes (to find out if the crystal additive has any side-effect on concrete mix left for longer time without any mechanical intervention). The concrete mixtures were mixed in 50 l volume concrete mixer “Zyclos” according to standard LST EN 480-1.

Mixing and addition of concrete components sequence:

- 1) Pouring all aggregates (sand and gravel) and 1/3 of water;
- 2) Mixing for 30 s;
- 3) 1 min pause;
- 4) Pouring all the cement;
- 5) Mixing for 30 s;
- 6) Pouring rest of water;
- 7) Mixing for 1 minute while superplasticizer is added;
- 8) Pouring in crystal additive powder and mixing for 30 s.

When the concrete mixtures were ready, slump, temperature, density and air content was measured according to regulations of the standards LST EN 12350-2, LST EN 12350-5, LST EN 12350-6, LST EN 12350-7. The data is given in table 2.

Table 3. Slump, temperature, air content and density of concrete mixtures

	Control	WT200P 1%	WT200P 2%	None Sika 1%
Slump, mm	75	110	155	60
Slump after 1h, mm	70	110	175	50
Temperature, °C	18.0	17.5	17.1	17.6
Temperature after 1h, °C	18.1	17.6	17.8	18.0
Density, kg/m ³	2369	2363	2376	2382
Air content, %	2.8	3.0	2.8	2.6

As it seen in the table 3, application of the crystal additives hasn't got any practical influence on temperature of concrete mixture, air content or density, but has some influence on concrete mixture slump. The concrete without crystal additive we have got S2 slump class, while with SIKA WT200P crystal additive the slump was increased by one class – from S2 to S3 and slum increased more as SIKA WT200P additive amount was increased. Using second type crystal additive None Sika we have got no practical influence on slump and slump was even lower, compared to control mixture (75mm control and 60mm with 1% f.c.m. None Sika). After 60 min concrete slump tendencies was similar as concrete slump measured after mixing. Slump after 1h of control specimens

have showed the same value as slump measured after the mixing, while concrete specimens with crystalline additive WT200P and 2% from cement amount, slump values after 1h already fulfilled S4 concrete requirements. It can be concluded, that both crystalline additives have no influence or small influence on fresh concrete properties like temperature, density and air content, while it influence fresh concrete consistency. WT200P have positive effect and it increase concrete consistency, while adding None Sika we have got no positive effect on this parameter.

INFLUENCE OF CRYSTAL ADDITIVE ON CONCRETE COMPRESSIVE STRENGTH AND DENSITY

The concrete samples for the investigation of various physical and mechanical properties were formed according to the concrete compositions given in the table 2. For compressive strength testing 9 cubes (measurements 100×100×100 mm) for each composition were formed. The Samples were demoulded after 1 day and for the rest days were kept in water (20±2 °C). The concrete compressive strength test was carried out after curing for 1, 7, 14 and 28 days according to the regulations of the standard LST EN 12390-3. The compressive strength test results and density values are given in the table 4.

Table 4. The concrete compressive strength and density after 1, 7, 28 days of curing

Concrete composition	Compressive strength after 1 day, MPa	Density after 1 day, kg/m ³	Compressive strength after 7 days, MPa	Density after 7 days, kg/m ³	Compressive strength after 28 days, MPa	Density after 28 days, kg/m ³
Control	27.2	2377	50.0	2402	56.3 ($\sigma=1.9$)	2385
WT200P 1%	27.8	2395	53.4	2389	65.7 ($\sigma=0.5$)	2398
WT200P 2%	23.6	2386	55.9	2384	67.7 ($\sigma=0.7$)	2419
None Sika 1%	20.2	2428	51.5	2406	59.2 ($\sigma=0.8$)	2408

As seen in the table 4, the application of the None Sika crystal additive or higher dosages of WT200P has an influence on concrete's early strength i.e. after 1 day of curing it can be lower down from 25 % till 13% accordingly (WT200P 1% have no effect on early strength). Concrete manufactures should consider that application of the crystal additive reduces concrete early compressive strength, so this should be considered preparing a demoulding schedule and working in winter conditions.

After 7 and 28 days of curing, compressive strength increases comparing with control mixtures. These tendencies also can be seen in Figure 2. We can see that control specimens average compressive strength value is 56,3MPa, while using WT200P 1% and 2% dosage strength was increased 65,7MPa and 67,7MPa accordingly (9,4MPa and 11,4MPa strength increase). Using None Sika crystalline additive and 1% dosage compressive strength was increased by 2,9MPa (till 59,2MPa), compared to control specimens. We can estimate that control concrete fulfils C40/50

concrete class requirements (according LST EN 206), concrete with WT200P 1% and with WT200P 2% fulfils C50/60, concrete with None Sika 1% fulfils C45/55 concrete class requirements.

According table 4 we can see that crystal additive influence on concrete density have changed slightly. We can see higher density values with None Sika compared with control specimens.

The graphic view of concrete compressive strength test results after 1, 7, 14 and 28 days is given in the fig. 2.

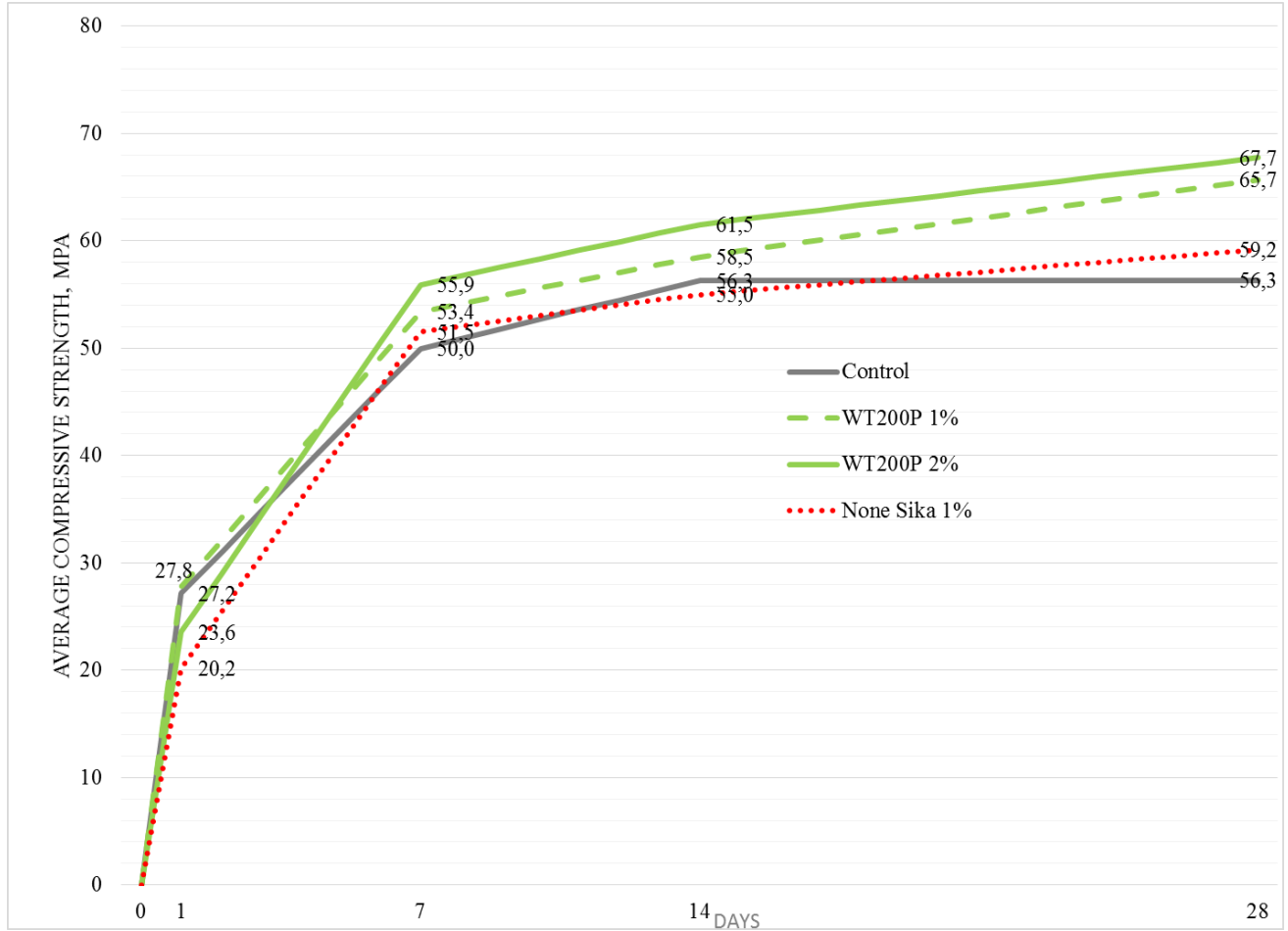


Fig. 2. The compressive strength tests results after 1, 7, 14 and 28 days of curing with crystal additive

INFLUENCE OF CRYSTAL ADDITIVE FOR CONCRETE WATER IMPERMEABILITY

3 cubes for each composition were formed (150×150×150 mm) to determine the influence of the crystal additive on concrete's water impermeability. Their water impermeability was determined after curing for 28 days in water according to the regulations of the standard LST 1974 Annex O (sample's water absorption depth was measured). During the test the concrete samples were placed in the special frame (see fig. 3), and exposed to enlarged water pressure of 1,4 MPa (on bottom samples side).



Fig. 3. Water impermeability test in special frame

The samples placed in the frame were gradually (by 0.2 MPa step) exposed to the water pressure for 7 days until it reached 1,4 MPa. After the testing it was noted that samples, made by the regulations of the standard LST 1974 Annex O, haven't let any water through the top of all samples and it fitted the requirements of W14 water impermeability class.

Because all samples passed W14 class (probably due to low W/C and high cement amount), it was made decision to keep such water pressure (1,4MPa) for 28 days. According LST EN 12390-8 standard, water pressure for the testing should be kept $500 \pm 0,50$ kPa for 72 ± 2 h, so keeping 1400kPa gives 2,8 times higher water pressure to samples.

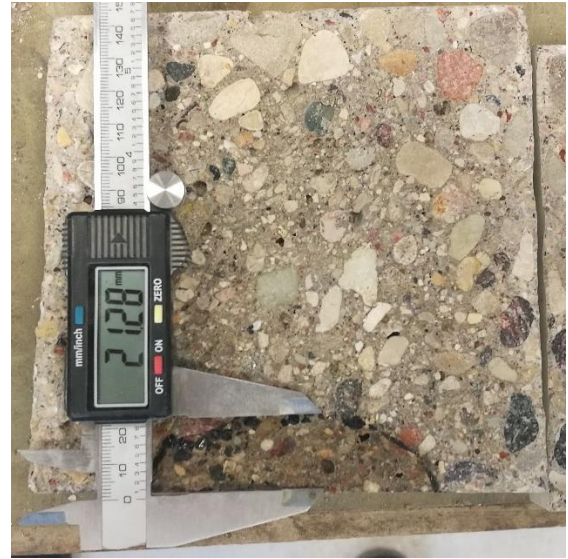
After the 28days and 1,4MPa pressure test, samples have been split in half according to the regulations of the standard LST EN 12390-8 to measure depth of water penetration. The depth of water penetration in the samples with crystal additive wasn't as deep as in the control samples. Therefore, it can be concluded that crystal additives increases concrete's water impermeability. The results are shown in the Table 5 and Fig. 4.

Table 5. Measurement of the maximum depth of penetration under the 28 days and 1400kPa pressure test.

	Control	WT200P 1%	WT200P 2%	None Sika 1%
Maximum depth of water penetration, mm	31,75	21,28	17,41	20,54
SIKA Limits, 30mm	not passed	passed <30mm	passed <20mm	passed <30mm



a) Control



b) With WT200P 1%



c) With WT200P 2%



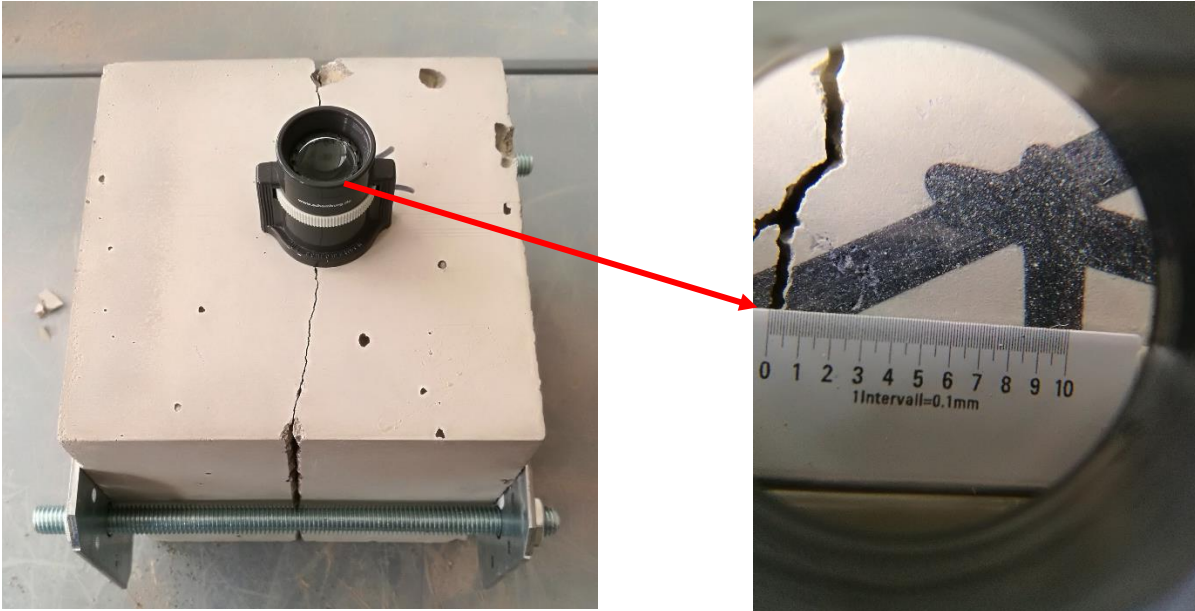
d) With None Sika 1%

Fig. 4. Maximum depth of water penetration, mm after 28days and 1400kPa pressure water impermeability testing conditions

As shown in table 5 and in Figure 4, applications of crystal additives reduces water penetration depth more than 33-45 %. According Sika given limits for Sika Watertight Concrete, water penetration depth should not exceed <30 mm when specimens are tested according EN 12390-8. In our case we can see that this value was passed in all specimens with crystalline additives, while control specimens value exceed <30 mm. Also it could be noticed that water pressure values was kept much higher as is written in EN 12390-8 standard so this conclusion is subjective.

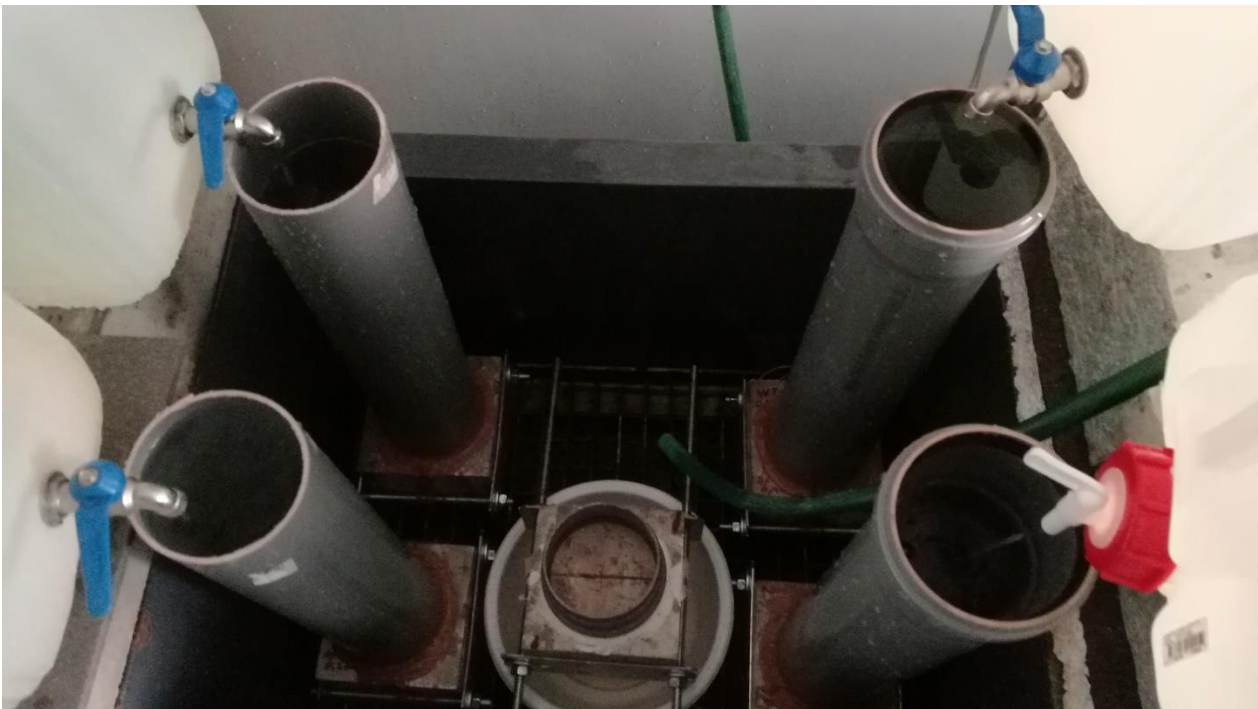
INFLUENCE OF CRYSTAL ADDITIVE ON SELF-HEALING OF CONCRETE – CRACK FILLING

The special test methodology was used to evaluate self-healing properties of concrete with the crystal additive, where an artificial crack was affected with water (0,5 m water column) for 28 days. The artificial crack was created by cutting the concrete sample and then joining both parts with the special clamps leaving the $\leq 0,4$ mm width. Later water was released to flow through the crack until crack was filled up with new crystals and water couldn't penetrate. So, after 28 days of normal curing the samples were split in half and tested as shown in the Fig. 5.



a) Tightened sample till $\leq 0,4$ mm width crack for the self-healing testing

b) Zoom view of tightened sample till $\leq 0,4$ mm width crack



c) Testing self-healing effect

Fig. 5. Testing procedure of crack healing effect

According procedure shown on Fig 5. decision was to keep maximum testing time 4 weeks. According technical literature, usually after such period cracks should be healed. Results are given in table 6. It can be seen that leakage stops in all specimens with crystalline additives. Fastest self-healing effect was obtained in specimen with WT200P 1%. Specimens with WT200P 1% after one week testing water flowability to sample decreased from small water stream till water drops and after 2 weeks was not leaking anymore. Specimens with WT200P 2% and with None Sika 1% was obtained similar tendencies only leakage stops later - after 3 weeks. While control specimens due all testing time (4 weeks) water leakage do not stops and it could be concluded that control specimen do not passed the self-healing testing.

Table 6. Self-healing effect.

	Control	WT200P 1%	WT200P 2%	None Sika 1%
Self-healing testing	Not passed	Passed (after 2 weeks)	Passed (after 3 weeks)	Passed (after 3 weeks)

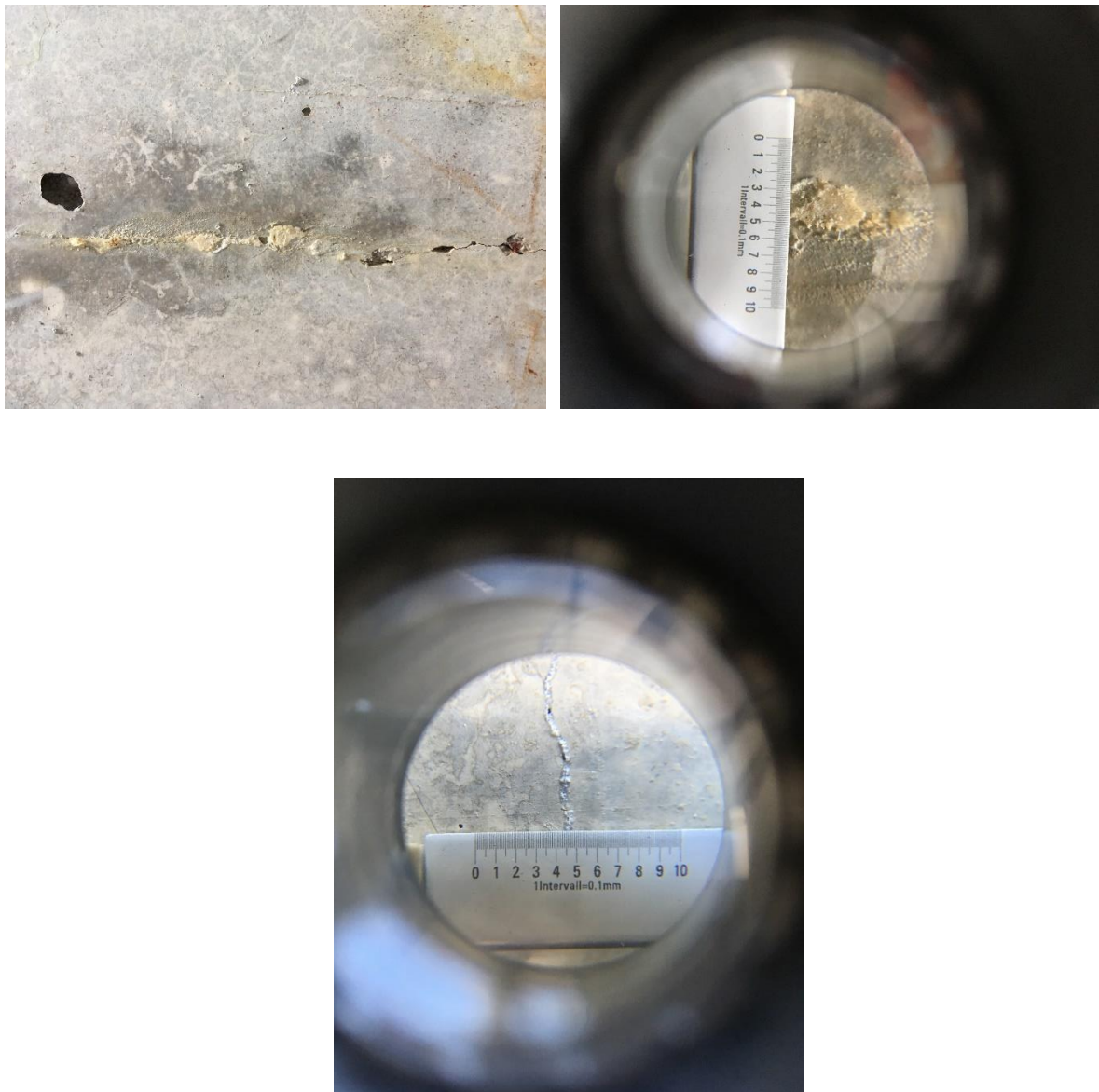


Fig. 6. Testing procedure of crack healing effect (view of crack filling by crystals in the bottom of concrete specimens after the test)

Although it is known that the crystalline additives are able to fill open concrete cracks (as demonstrated by studies of this work), it is not known what water pressure is still able to fill the crack. Also it is not known what water pressure can resist the crystal-filled crack. These issues are becoming more and more relevant in construction sites under soil water pressure.



Fig. 7. Testing procedure of crack healing effect with 5-meter high water column pressure (is about 0.5 atmospheric pressure on specimen).

So, after discussions with scientists of this self-healing methodology testing, we concluded that such testing method need to be updated because it cannot reflect real object conditions, where water column pressure is much higher. So, we have done testing with always 5meter water column pressure (see Fig 7). Results after 35 days of water filtering through the crack in the concrete with 5meter water column pressure have showed very promising results, it stops in some stage of water column pressure (filled crack hold some stage of water level in pipe), but these results will not be given so far, because needs more research and more statistical data's. This could be done in future works.

Conclusions

1. KTU SMKTC executed the concrete tests with crystal additive Sika WT200P and None Sika. The developed concrete compositions were investigated to determine the effect of concrete self-healing – natural crack filling when water filters through the crack in the concrete. The cracks of these concrete samples became water impermeable respectively after 14 and 21 days of testing, and the concrete compositions, capable healing cracks up to 0.4 mm width, were developed.
2. The experiments have shown that the crystal additive does not affect temperature of the concrete mixture, density or content of entrained air, however it changes the slump (depending on type of crystalline additive, see the table 3), so it should be considered. The compressive strength tests after 1, 7 and 28 days have shown that the crystal additive reduces the early strength of the concrete up to 25% after 1 day (depending on the type of crystalline additive, see table 4), but after 7 and 28 days the compressive strength increase 20 % when 2 % f.c.a. of Sika WT200P was used.
3. The results of the research have shown that the crystal additive didn't have any negative influence on density.
4. The water impermeability test was executed by the methods of the LST 1974 annex O (it is noted whether water penetrated through the entire sample or not) and by LST EN 12390-8 (the water penetration depth in the sample is noted) with 1.4 MPa water pressure for 28 days. The samples of the all composition classified as W14, but the concrete with the crystal additive was 30 % less permeable and water penetration depth did not exceed 30mm.
5. Overall the crystal additive WP positively modifies concrete i.e. the concrete self-heals cracks and in presence of water can fill cracks up to 0.4 mm width. So far, no negative concrete properties were noted apart from the reduced slump with None Sika additive and early compressive strength with crystalline additives.
6. KTU SMKTC advises in future to consider the effectiveness of crystal additives according to the capability to self-heal crack of the declared width, applying methods specified in this research when higher water column is used. This test can show the maximum water pressure is still be able to fill the crack.
7. Watertight concrete with crystals could be used when needs to keep water in or out or both. It could be used for: Residential and commercial basements; Public and government building basements; Underground car parks and service areas; Health, education and leisure facilities; Swimming pools, lift pits and utility/plant rooms; Utility (water and power) structures; Road, rail and mining tunnels and structures; Marine, dock, harbour and river structures.