The investigations on properties of self-healing concrete with crystalline admixture and recycled concrete waste

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Abstract. The concept of self-healing concrete is becoming more necessary as sustainability in construction is more desirable. Amongst the current solutions in this technology are autogenous, chemical, and bacterial self-healing. It is paramount that secondary raw materials be used in the production of self-healing concrete as a form of a sustainable solution. Therefore, in this paper, the admixture "Betocrete-CP-360-WP", which is a crystallizing waterproofing admixture with hydrophobic effect and is 100% recyclable, has been used and its effect on the physical, chemical, and mechanical properties of concrete, as well as self-healing capabilities of concrete, have been determined. According to the obtained results, the crystalline additive "Betocrete-CP-360-WP" has no effect on density and slightly increases the amount of entrained air in the concrete mix. However, it does decrease the workability of the concrete mixture which could prove problematic in transportation to the construction site or in concreting in general. Also, with the crystalline admixture in the concrete mix, a 60% reduction in concrete compressive strength after one day of hardening has been estimated, but after 7 and 28 days, the strength attained is within the ranges of the control samples. In addition, concrete containing Betocrete-CP360-WP was 30% less water permeable as compared to control samples. The self-healing efficiency of the concrete was determined by a water flow test through a formed crack (approximately 0.35 mm wide). This was done by gluing a plastic pipe to the top of the cracked concrete specimens and maintaining a constant pressure of the water in the pipe. The experiment was continued for 28 days, and the crack self-healing efficiency of the concrete was calculated from the differences in the amount of water passed through the crack before healing and after 7, 14, 21 and 28 days of the healing process. After 28 days of the water flow test, the cracks in the concrete with the crystalline admixture and recycled concrete dust were completely healed, while the control specimens were not.

Keywords: concrete, self-healing, crystalline, admixture, crack

1. Introduction

Nowadays, the recycling of building materials is a topic of interest for most engineers. The world as a whole is more attentive to the effects of CO_2 emissions and is quick to identify in which areas this problem can be reduced. Construction is one of the leading polluting industries; therefore, there should be sought ways to reduce its effects in new structures. One of these ways is through utilising concrete from demolished buildings as aggregates in new mixes. Another way is through the creation of technologies that promote a longer life cycle for structures, such as self-healing concrete to reduce or slow the effects of cracking, which in turn result in a reduction of use of materials that are major pollutants of the environment. Concrete cracking is a common problem which, if not considered in design or taken care of if it appears in early stages, can cause structural failure, which in turn could cause harm to people and the environment. The rate of deterioration of concrete that has cracks is sped up in the presence of moisture because water containing chemical elements that cause dissolution of minerals and corrosion of reinforcement is absorbed into them. [1, 2, 3] Therefore, it is important that the concrete is watertight and does not have cracks, but often the latter occurs during operation due to the disruptive effects of the environment and/or mechanical loads. The moisture fluctuates more in buildings rather than structures like dams or roads; therefore, we see more of a problem with cracking. [1, 2]

One of the current technologies to resolve the issue of concrete cracking is the use of self-healing concrete in construction. There are two main mechanisms of self-healing: autogenous and autonomic self-healing. [4, 5] In autogenous self-healing, the process occurs internally with usage of minerals and chemical compounds within the concrete. With autonomous self-healing, incorporation of materials not traditionally used in concrete is needed, example being bacteria. [6] These healing agents are often encapsulated in damage-resistant capsules and then released in the microstructure of the concrete when a crack appears, for example. [4, 5] Much ground has been covered in research of using these autonomic methods as bacteria in self-healing, but the biggest concern is the cost incurred. Aside from that, there is also the issue of knowledge of use as it's not something construction workers commonly work with. More

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measures would need to be taken into account when performing concreting works.

Relying on autogenous self-healing without the inclusion of special chemical admixtures can prove a lengthy process with no effect, immediate or over time. Another method of self-healing that has been discussed is the use of different types of admixtures, including mineral or crystalline in creating self-healing concrete. [4, 5, 7, 8, 9, 10, 11-20] With crystalline admixtures, the process requires moisture because the CO_2 present in it reacts with calcium ions (Ca^{2+}) in the concrete to form calcium carbonate ($CaCO_3$) which starts to fill the cracks and heal the concrete. The healing capability this promotes is for cracks up to 300 µm in width. [8, 9,11] However, when such admixtures are used in combination with expansive agents, which expand in the presence of water to fill voids, larger crack widths (up to 400µm) can be healed. In order to eliminate the damages and improve the properties of the products, it is possible to use a new generation crystalline additive, which in response to products released during cement hardening forms stable crystals (through crystallization) that eliminate micro and macro cracks in the structure in the entire volume of the product; therefore, promoting the concept of self-healing concrete. By filling the capillaries, the crystalline additive Betocrete-CP-360-WP can minimize the water absorption of the structure, significantly decrease water permeability, increase frost resistance, and the concrete construction is likely to become more resistant to chemical environmental influences, i.e., it will be more durable [10]

In this paper, the crystalline additive Betocrete-CP-360-WP and crushed concrete waste at 3% by cement mass content has been used along with different cement types to determine the characteristics of the product obtained and its applicability as a self-healing concrete. The tests conducted include X-ray Diffraction Analysis (XRD), Scanning Electron Microscopy (SEM) that analyses the structure of the resulting newer (crystal) and X-ray Energy Dispersion Spectrum (EDS) for cell analysis. In addition, other properties of concrete have been tested: flowability, compressive strength, frost resistance and more.

2. Materials & Methods

The crystalline additive Betocrete-CP-360-WP is a product manufactured by the German company SCHOMBURG for use as a waterproofing agent in concrete structures. It has a healing penetration capability for cracks up to 0.4mm in depth as well as tapering applications for cracks up to 0.5mm. It has also been shown to reduce chloride migration.

To assess the influence of the crystalline additive on the physical and mechanical properties of a fresh concrete mix and hardened concrete, compositions were designed. Two different types of cement were used: Lithuanian cement AB " Naujoji Akmenė" CEM I 42.5 N and Latvian cement SIA "CEMEX" CEM I 42.5 N. Additionally, the crystalline additive Betocrete-CP360-WP of the German company SCHOMBURG was added to some of the samples. In all but one sample, 3% of cement mass was replaced by crushed concrete dust as shown in Table 1.

The influence of the admixture Betocrete-CP-360-WP on the physical properties of a fresh concrete mix was assessed. In addition, its influence on the following properties of hardened concrete was analysed: strength and density, shrinkage, chemical resistance, water permeability, water absorption, frost resistance and self-healing ability were assessed.

Table 1. Composition of concrete samples.

Material	LT	LT-P	LV	LV-P
Portland cement*, kg	350	350	350	350
Water, l (W/C=0.45)	157.5	157.5	157.5	157.5
Sand (0/4mm), kg	885	885	885	885
Gravel (4/16mm), kg	965	965	965	965
Superplasticizer Remicrete SP 56, kg	6.0	6.0	6.0	6.0
Crystalline additive Betocrete-CP-360-WP, kg (0.8%)	-	2.8	-	2.8
Crushed concrete dust, kg (3%)	10.5	10.5	10.5	10.5

*In concrete compositions with marking LT, CEM I 42.5 N AB “Naujoji Akmenė” was used, and in the compositions marked LV, SIA “CEMEX” CEM I 42.5 N was used, and samples with “P” meaning with crystalline additive Betorcrete-CP-360-WP.

2.1. Flowability

A fixed procedure was followed in the preparation of the concrete mixes for a better comparison. The aggregates (sand and pebbles) were poured first along with a third of the water and stirred for 30 seconds. Following this was a minute wait then the addition of cement, a 20-second stir then the rest of the water added. This was then stirred for a minute and the superplasticizer added after. Finally, the crystalline additive was included, and the mix stirred for 30 seconds before conducting tests for flowability, temperature, air content and density of the mixture according to the procedure described in EN 12350-2, EN 12350-5, EN 12350-6 and EN 12350-7 standards. Properties of hardened concrete.

2.2. Strength and density

The mixture was poured into cubes of 100 x 100 x 100mm and was removed from the forms after 1 day. They cubes were then immersed in water until the 28-day mark and tests were conducted for compressive strength and density according to the requirements of EN 12390-3 standard.

2.3. Shrinkage

For the determination of shrinkage deformations with each concrete composition, 3 prisms were formed with dimensions 75×75×250 mm. The samples were removed after 1 day of hardening and the measurements were carried out using a special deformation bench with an electronic indicator. In all cases, the ratio of the length of the samples to the reference steel rod was measured. After 7 days of air hardening, the measurements were repeated, and relative deformations of the concrete were calculated from the difference between the results and the initial length of the samples. After 7 days of solidification in the air, concrete samples were immersed in water at 20°C and maintained for 21 days. The deformations of the samples were then measured again. The relative deformations of concrete samples were calculated according to the formula in EN 12617-4.

2.4. Chemical resistance

Concrete usually has a low resistance to chemically aggressive environments. $Ca(OH)_2$ is one of the most water-soluble compounds in concrete and the calcium ions easily react with other alkalis or salts and this may lead to sulphate corrosion if the water exposed to the concrete contains a sufficient concentration of dissolved sodium or magnesium sulphate, as an example. The resistance of concrete to different types of corrosion is increased by a lower W/C ratio and a higher density since lower water absorption slows down the transfer of ions in concrete.

As such, to determine the effects of Betocrete-CP-360-WP, tests were conducted according to the standard DIN 4030-2, whereby concrete was stored in a solution containing ammonium sulphate, which is essentially an extremely aggressive environment for concrete. Two concrete samples of 100×100×100mm were formed for each concrete composition, kept in water at 20°C for 2 weeks and then maintained for a further 14 days under conditions of 20°C and 65% relative humidity. The mass of the samples was then recorded, and the samples then fully immersed in an ammonium sulphate solution at 148g/l for 100 days. After this time, the concrete samples were maintained for 14 days at 200°C and 65 % relative humidity then weighed and the difference in masses determined.

2.5. Water permeability and capillary absorption

Concrete cubes of 150×150×150mm were formed for each composition and tested for water permeability. The samples were placed on a special stand on which they were exposed to water pressure that was increased by 0.2 MPa each day for 5 days until 1.0 MPa pressure was reached. After 28 days, the permeability was determined by seeing whether the water passed through the sample in accordance with GOST 10060:2012 then the depth of water penetration was concluded upon according to EN 12390-8.

To determine the effect of the crystalline additive Betocrete-CP-360-WP on the capillary water absorption of concrete, 6 prisms of 40×40×160mm were formed using a standard mortar composition of 450g of cement, 1350 g of sand and 225 g of water with the same Lithuanian and Latvian cements and crystalline additive. After 1 day of hardening, the samples were weighed then tested according to EN 480-5. Then, 3 samples were placed vertically in a container where the water was at 3mm depth from the bottom of the sample. The other 3 samples of the same composition were kept in the air for 6 days in a mode chamber. After 7 days, all samples were weighed and the circular absorption, CA, was calculated from the difference in masses and the area of the sample.

2.6. Frost resistance

Frost-resistant concrete is usually produced using an air-immersive additive; therefore, in order to assess the effect of the crystalline additive Betocrete-CP360-WP on frost resistance, a concrete mix with the same composition as the initial samples was created but with the addition of the air-immersive additive at 1.05kg. For mixtures of LT-P and LV-P frost-resistant concretes, their sliding, temperature, density and air content were determined in accordance with the procedures described in EN 12350-2, EN 12350-5, EN 12350-6 and EN 12350-7.

Subsequently, cubes were formed with this concrete mixture and the compressive strength and resistance to volumetric refrigeration were tested on samples of size 100×100×100 mm, then resistance to surface cooling was tested on samples of size 150×150×150 mm.

The frost resistance test of concrete was carried out according to the volumetric method 1428-17:2016. A total of 12 samples of size 100×100×100 mm was formed for each composition. After 28 days in water, the first 3 samples were tested for compressive strength. The take-off samples were placed in a special refrigerating chamber where three daily freeze and thawing cycles were carried out. After 150, 200 and 300 cycles, three samples were tested. The freeze-thaw cycle temperatures were used as follows: freezing temperature -18 ± 2°C and thawing temperature +20 ± 2°C. The time interval used for freezing and thawing was 4 hours.

Determination of frost resistance of concrete according to the surface method was carried out in accordance with SS 137244:2019. Samples 50 mm high were cut from concrete cubes of 150×150×150 mm and the cut surfaces were frozen after having a 3 percent sodium chloride solution poured onto it. The samples were stored in a refrigerator which carried out one freeze and thaw cycle in one day. After 56 cycles, the masses coming off the concrete surface as a result of exposure to ice were weighed. Frost resistance was assessed based on mass loss (kg/m^2).

2.7. Self-healing

In order to determine the self-healing effect of cracks on concrete with the crystalline additive Betocrete-CP360-WP, a specific test procedure was used to artificially create a crack and continuously expose the concrete samples to a constant water pressure through a 1-metre pole over 24 hours for up to 28 days, as shown in Fig. 1. Once the artificial crack was created, the samples were fixed on either end to tighten the detached parts and a slit of 0,4mm width was left. The test was stopped for LT-P after 2 weeks (14 days) and after 3 weeks (21 days) for LV-P.

Fig. 1. Testing mechanism for self-healing capability of concrete samples containing crystalline admixture.

3. Results

3.1. Influence of crystalline additive on the physical properties of a fresh concrete mixture

As seen from Table 2., the use of the crystalline additive Betocrete-CP-360-WP had no significant effect on the temperature, air content or density of the concrete mix; however, it did influence the flowability of the fresh concrete. The initial slump for both types of cement was classified as S4 but was then reduced to S2 for the Lithuanian cement AB "Naujoji Akmene" CEM 1 42.5N and S3 for the Latvian SIA "CEMEX" CEM 1 42.5N. After an additional 30 minutes, the Lithuanian cement maintained the S2 class, but the Latvian cement reduced its slump class to S1, showing that the crystalline admixture does have an effect of flowability. This means designers must be aware when using this crystalline additive because it will affect the workability. As workability is generally inversely proportional to concrete strength, we could relate these results with that of the compressive strength tests. As seen in the compressive strength test, samples containing Betocrete-CP-360-WP have lower compressive strengths, which is supported by their high workability.

Table 2. Slump, temperature, air content and density of concrete mixes.

Parameters	LT	LT-P	LV	LV-P
Temperature of mixture, °C	18	18	18	18
% air content of mixture	2.5	3.2	2.5	2.9
Initial Slump, mm	170	70	160	100
Slump after 30 min, mm	170	60	60	40
Mixture density, kg/m3	2395	2397	2399	2405

3.2. Influence of the crystalline additive on the strength and density of concrete

As shown in Fig. 2., the use of the crystalline additive affects the early strength of the concrete. After 1 day of hardening, the strength was significantly less than the control sample. The compressive strength improves after 7 and 28 days and is only slightly less than that of the control sample. The density of concrete in samples with a crystalline additive is also reduced slightly with ranges from about 0.46 to 1.6%, which isn't too significant. Concrete manufacturers must bear in mind that the use of the crystalline additive Betocrete-CP360-WP will lower the early strength of the concrete, so this must be evaluated by preparing a schedule for removing formwork and concrete in winter conditions.

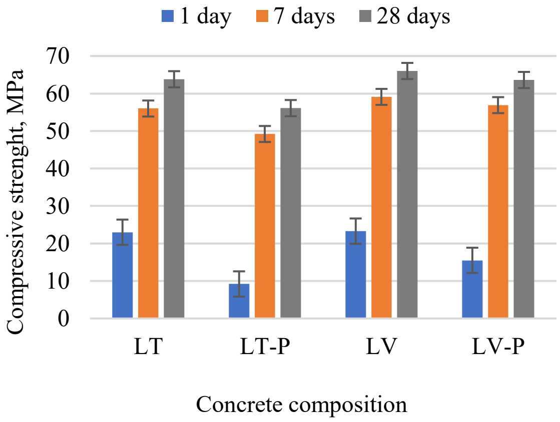

Fig. 2. Compressive strength and density after 1, 7 and 28 days of hardening.

3.3. Influence of crystalline additive on concrete shrinkage deformations

Table 3. shows the changes in concrete mass and relative deformations according to the tests carried out in part 2.3. The + sign indicates that the mass was increasing, and the samples were shrinking, whereas the - a sign that the mass was decreasing, and the samples were expanding. As shown, the use of the crystalline admixture Betocrete-CP-360-WP has no significant effect on shrinkage deformations. More influence is observed according to cement type used.

Table 3. Results of changes in concrete mass and relative deformations.

Parameters	LT	LT-P	LV	LV-P
Δg 7 d, %	+0.89	+0.94	+0.94	+0.76
Shrinkage 7 d, %	+0.004	+0.004	+0.017	+0.012
Δg 28 d, %	-0.49	-0.38	-0.15	-0.22

Deformation 28 d, %	+0.018	+0.019	+0.014	+0.013

3.4. Influence of the crystalline additive on the chemical resistance of concrete

After 100 days of maintenance in a chemically aggressive ammonium sulphate solution, concrete with crystalline additive Betocrete-CP-360-WP had higher compressive strengths for both cement types and as much as 40.7 MPa with the Latvian cement as compared to its control sample with only 28.4 MPa. Also, with the Latvian SIA "CEMEX" CEM I 42.5 N and the crystalline additive, the concrete became more resistant to chemical effects as compared to the other samples. The concentration of the ammonium sulphate solution is too aggressive, so all concrete samples became poor in appearance after as seen in Fig. 3.

Table 4. Change in concrete mass (%) and compressive strength after 100 days in a chemically aggressive ammonium sulphate solution.

Name of the concrete composition	Weight loss, %	Crushing strength after 28 days, MPa
LT	4.88	33.4
LT-P	5.88	34.1
LV	4.41	28.4
LV-P	2.56	40.7

Fig. 3. Concrete samples after 100 days in ammonium sulphate solution (148 g/l).

3.5. Influence of the crystalline additive on concrete water permeability and capillary absorption

According to the results of this test, it was seen that the samples were impermeable up to the W10 water tightness class. In concrete samples that were produced using the crystalline additive Betocrete-CP-360-WP, the water penetration depth was lower by as much as 30%, which leads to the conclusion that the additive decreases the water permeability of concrete. The capillary absorption for the samples was similar in all compositions with about 0.01% decrease, at most. Fig. 4. Shows how the penetration depth was measured.

Fig. 4. Depth of water penetration of concrete samples.

3.6. Influence of the crystalline additive on the frost resistance of concrete

The compressive strength of the samples did not decrease significantly even after 300 freeze-thaw cycles. As such, we can conclude that the crystalline additive does not have any influence on the property of frost resistance of concrete when mixed with an air-immersive additive. According to the surface method of testing the frost resistance, it can be seen that after 56 cycles, the mass loss is very similar for all samples (with ranges between 0.15 to 0.20 kg/m$^{2)}$ and also insignificant as in the first frost resistance test; therefore, it is safe to say that the crystalline additive Betocrete-CP-360-WP has no effect on frost resistance.

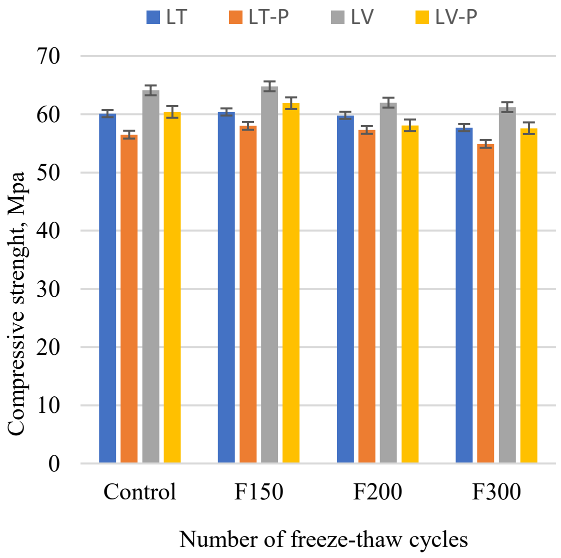

Fig. 5. Compressive strength of concrete samples after 0, 150, 200 and 300 refrigeration-heating cycles

3.7. Influence of the crystalline additive on the self-healing of concrete

According to the self-healing capability test conducted where a constant water pressure was maintained on concrete blocks containing Betocrete-CP-360-WP, it can be concluded that the crystalline additive promoted self-healing within the concrete. Aside from the initial crack, the gaps or pores within the concrete samples were also assessed to determine whether self-healing had occurred. After a maximum of 21 days of being exposed to a constant pressure of 1 atmospheric pressure, it can be seen that the initial crack as shown in Fig. 6 was completely healed (Fig. 7).

Fig. 6. Initial crack created by tensile split test.

Fig. 7. Crack completely healed after 21 days of being exposed to constant water pressure.

The pale orange paste that can be seen is the calcium-silicate-hydrated gel that is formed as a result of continued hydration that fills the crack, therefore creating the healing effect.

As shown in Fig. 8., gaps/pores as wide as 0.54 mm can be healed to as much as 0.18 mm width which is an estimated 67% healing capability.

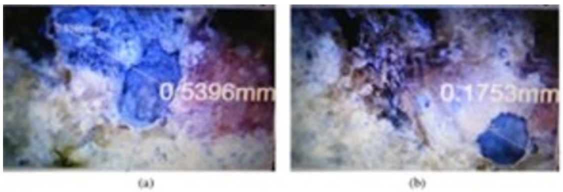

Fig. 8. Initial gap/pore (a) filled by crystals (b) after healing process.

4. Discussion

The use of the crystalline additive Betocrete-CP-360-WP had no effect on the temperature, air content or density of the concrete mix; however, it did influence the flowability of the fresh concrete, with the initial slump for both types of cement being classified as S4 class and then reduced to S2 class for the Lithuanian cement and S3 class for the Latvian cement. The slump for the Latvian cement was further reduced to S1 after the additional 30 minutes of mixing. Similarly, Gojevic et al. [23] concluded that the addition of the crystalline admixture reduced the slump of the fresh concrete mix by as much as 37.5%, similar to the results achieved with the Latvian cement. In addition, it was also found that the admixture had no effect on the air content or density of the concrete. On the contrary, Geraldo et al. [12] saw the addition of a crystalline admixture, not specified, resulting in reduced slump. This may be attributed to the mixture design as the water to binder ratio was quite high.

In concrete samples that were produced using the crystalline additive Betocrete-CP-360-WP, the water penetration depth was lower by as much as 30%, which leads to the conclusion that the additive decreases the water permeability of concrete, which was also seen by other researchers [3, 4, 21-23].

The early-age compressive strength was significantly lower for samples containing the crystalline additive, LT-P and LV-P (as shown in Fig. 2.) Cobos et al. [3] found that including a crystalline additive at as much as 0.45% or 0.9% cement mass resulted in about a 12-hour delay in cement hydration through measuring the electrical resistance of said samples during hydration, as well as assessing the setting time using the VICAT apparatus, which then ultimately influences the early strength of concrete. Cobos et al. [3] also then found that after 7 days of hardening, the properties of the material are no longer affected by the crystalline additive. As Gojevic et all explain [23], the use of the crystalline additive has no significant effect on the compressive strength due to the lower workability and higher air content, which is also presented in this work.

In consideration of the chemical resistance of the concrete mix, we see that the addition of the crystalline admixture does not significantly weaken the concrete. Instead, we see that with the Latvian cement, the concrete performs better than the control in a chemically aggressive environment and achieves a significantly higher compressive strength when tested after 28 days. It is, however, important to note that the samples lose their aesthetic appearance when exposed to this environment. Drochytka et al. including other researchers [13] similarly found that with coatings of crystalline admixture, the concrete exposed to the aggressive agents CO_2, SO_2, SO_4^{2-}, Cl^- had increased protection against the conditions. At a depth of 15 mm, even over a time as long as 18 months, the pH did not significantly change which would mean reinforcement can remain protected. [24]

In reference to the self-healing ability promoted by the crystalline additive, as can be seen from Fig. 8., the gap of 0.5396mm (a) was reduced in size by about 67.5% (b) under the water test while being filled by a crystal.

This goes to show the crystalline additive Betocrete-CP-360-WP promotes healing in concrete due to the crystallization process. The reaction between the active compound of the crystalline additive and tricalcium silicate in cement in the presence of the moisture caused the formation of calcium-silicate-hydrated (C–S–H) gel bounded with crystalline deposits and a precipitated pore blocker in the micro-pores and capillaries, resulting in the increasing resistance to the penetration of water under pressure. Betocrete-CP-360-WP causes formation of a hydrophobic layer or simply blocks the pores. This hydrophobic layer then creates a permanent water-impermeable effect, which we then refer to as the healing ability. Additionally, a crack created as in Fig. 6 via the tensile splitting method (resulting in a gap of 0.305 mm), was completely healed as seen in Fig. 7. The gel or paste of C-S-H formed due to crystallisation is visible confirming the improved self-healing ability of samples containing the Betocrete-CP-360-WP crystalline admixture. The aesthetic quality of samples after self-healing has occurred is to be analysed in the future as a desirable outcome would be minimal effort in maintenance of concrete structures. Further testing is also required to increase the crack width that can be healed with the autogenic mechanism. However, experimentation showed that the self-healing capability of concrete increases in the presence of Betocrete-CP-360-WP and makes it a viable option for use in the creation of concrete structures. More research is needed to conclude on the overall effects of this crystalline additive over time.

5. Conclusions

- The crystalline additive Betocretre-CP-360-WP has no effect on density and air content of concrete, but it does influence the workability. Hence, it is necessary to design the concrete mix carefully to allow for ease of transportation and concreting procedures.
- The early age compressive strength of concrete is significantly lowered in the presence of the crystalline admixture. However, over the hardening process, it is seen that the strength reached for concrete containing Betocrete-CP-360-WP are similar to that of the control mix, with no real significant difference.
- The use of Betocrete-CP-360-WP can be highly effective in reducing the effects of corrosion of concrete which is exposed to harsh chemical environments. The structural integrity may be maintained to a certain extent and the reinforcement is protected from corrosion, but the external appearance may be affected.
- Addition of the crystalline admixture has a positive effect on the self-healing ability of concrete. In the presence of Betocrete-CP-360-WP, the gaps from cracks within the concrete are healed by as much as 67.5% over a 28-day period, which means that over longer lengths of time, the gaps may be completely filled.
- The use of crushed concrete dust at 3 % by cement mass did not prevent the achievement of self-healing concrete. However, further testing is needed to determine at what percentage the crushed concrete will start to have an effect on the properties of concrete samples.

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