Self-Healing Performance and Microstructure Aspects of Concrete Using Energetically Modified Cement with a High Volume of Pozzolans

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ABSTRACT

Self-healing can increase the lifetime and durability of concrete structures. The self-healing properties of concrete made with Energetically Modified Cement (EMC), which has a pozzolan content of up to 70%, have been investigated. In such concretes, pozzolanic reactions within the cement cause the gradual formation of fresh C-S-H gel, which seals cracks as they form. The self-healing of small EMC concrete samples was tested in a laboratory, and field observations of an EMC concrete highway pavement were made. The EMC concrete exhibited fewer cracks than conventional concrete, and was observed to self-heal cracks with widths of up to 0.2 mm.

Key words:

Key words: Self-healing, cracking, pozzolanic reactions, modified cement binders

1. INTRODUCTION

1.1 Background

Many countries are experiencing civil infrastructure deterioration so severe that the annual outlay on repair and rehabilitation often exceeds the cost of constructing new infrastructure. This is partly due to the cracking (including thermal cracking, shrinkage cracking, etc.) of concrete during various stages of the hardening process. Concrete deterioration is also a problem in housing and industrial applications, where cracking is unacceptable because of its effects on durability, aesthetic value, hygiene, and acoustic insulation.

In the United States, the annual economic impact associated with the maintaining, repairing, or replacing such deteriorating structures is estimated to be $18-21 billion [1]. About half of all field repairs fail, necessitating re-repairs. Around three-quarters of these failures are attributed to a lack of durability and the remainder to structural failures. This inadequate performance is often ascribed to inappropriate material selection, the use of a poor application method, or both factors together [2].

In addition to the economic costs of repair and rehabilitation, civil infrastructure deterioration presents social and environmental costs. While these costs have not been well documented or quantified, it is generally agreed that repeatedly repairing civil infrastructure over the course of its service life is decidedly unsustainable.

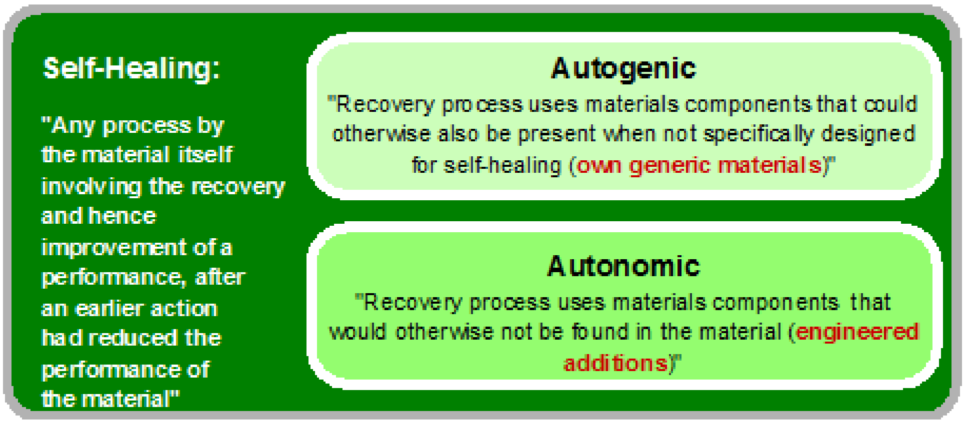
Consequently, there is great interest in concrete capable of self-healing, i.e. sealing cracks as they form. Such concrete enables the construction of more durable structures with increased lifetimes, both of which are important and desirable in a sustainable society that uses concrete as a major building material. It is therefore important to explore the self-healing capabilities conferred by new cementitious binders.

One such new cementitious binder is Energetically Modified Cement (EMC), which is formed by mechanically activating mixtures of Portland cement with a pozzolan, silica sand or blast furnace slag. A pozzolan is here defined as a siliceous or siliceous and aluminous material that has little or no intrinsic cementitious value but reacts chemically with calcium hydroxide at ambient temperatures when finely divided and exposed to water, forming compounds with cementitious properties. The EMC process was discovered in 1992 at Luleå University of Technology (LTU) and has since been developed extensively [3] - [7].

It is thus interesting to evaluate the self-healing performance of concretes incorporating Energetically Modified Cements and its relationship with the microstructural characteristics of the cement. This paper presents laboratory and field test results relating to the self-healing performance of concretes with EMC.

1.2 Self healing

Various Scandinavian groups have recently studied self-healing concretes [8] - [11]. RILEM's Technical Committee 221 [12] distinguishes between autogenic and autonomic self-healing (see Figure 1) and it has been argued that self-healing phenomena can also be classified in terms of process and action, as shown in Figure 2 [13] [14]. This article concerns autogenic self-healing, i.e. recovery processes involving material components that might be present even if the material had not been specifically designed for self-healing.



Self-Healing:

"Any process by the material itself involving the recovery and hence improvement of a performance, after an earlier action had reduced the performance of the material"

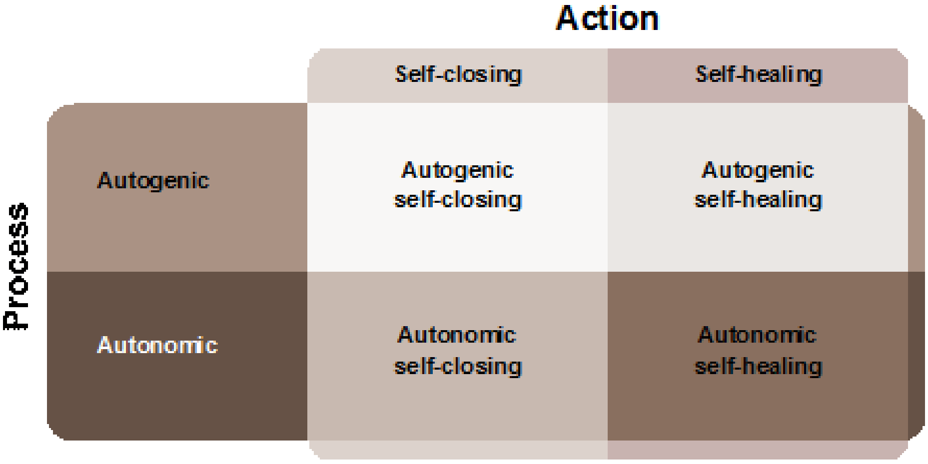
Autogenic

"Recovery process uses materials components that could otherwise also be present when not specifically designed for self-healing (own generic materials)"

Autonomic

"Recovery process uses materials components that would otherwise not be found in the material (engineered additions)"

Figure 1. Definition of Self-Healing according to RILEM-TC221 [12].



Process \ Action	Self-closing	Self-healing
Autogenic	Autogenic self-closing	Autogenic self-healing
Autonomic	Autonomic self-closing	Autonomic self-healing

Figure 2. Self-Healing defined in terms of action and process according to references [13] from [14].

Autogenic healing has been attributed to the hydration of unreacted cement (leading to fresh C-S-H gel formation, etc.), the expansion of concrete in crack flanks (i.e. swelling), crystallization of compounds such as calcium carbonate, closing of cracks by solid particles in water, and closing by spalling of loose concrete particles resulting from the cracking itself [8], [9], [14], [15]. Crystallization appears to be the most important mechanism in mature concrete. However, the hardening of many pozzolanic binders continues beyond the first month after pouring, so further hydration and C-S-H gel formation is considered to be the main reason for healing in concrete incorporating these binders. Through cracks in structures exposed to water pressure on one side can also heal via the precipitation of lime in the crack [16]. However, many applications do not involve any such one-sided pressure.

Researchers have examined the effects of diverse factors on self-healing, including crack width; the temperature; water pressure; the composition of the concrete and binder; and the chloride concentration, pH, and hardness of the water to which the concrete is exposed [8], [15], [17]. The relative importance of these factors is of course dependent on the dominant mechanism of self-healing in the structure of interest.

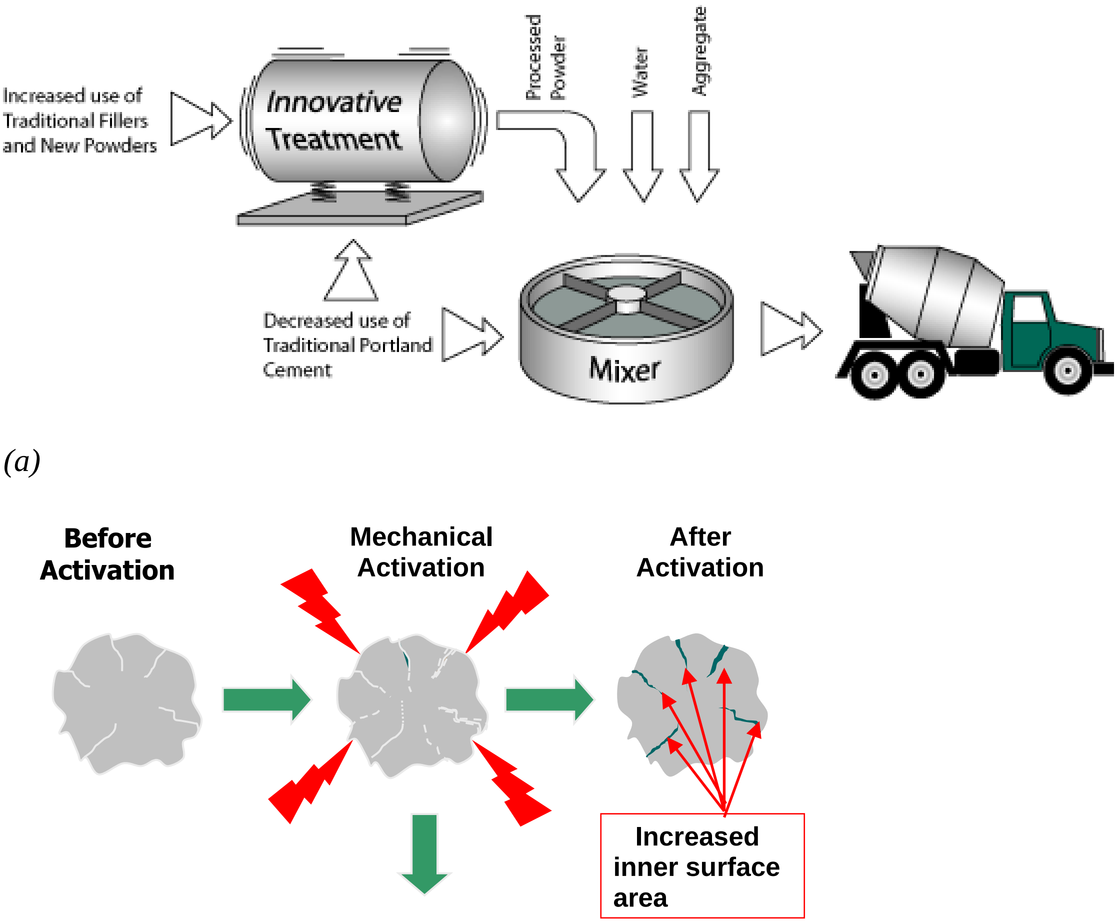
The effects of self-healing on strength are considered to depend on three factors [8], [9], [17]: (a) the moisture content in the crack and the duration of storage; water-stored concrete heals more effectively than concrete exposed to high relative humidity, (b) the initial crack width; smaller cracks seems to heal more completely whereas specimens that are completely broken clearly will not heal efficiently, and (c) the applied pressure; exposure to pressure clearly implies more effective healing than would be possible in a stress-free crack.

1.3 Modifying the binder's particulate structure – Energetically Modified Cement (EMC)

EMC is produced by processing cement with various other inorganic materials in high intensity grinding mills or commuting devices that subject the solids to severe mechanical stress and transient high temperatures, leading to particle surface modification (see Figure 3). These commuting devices have been called "mechano-chemical reactors" instead of traditional mills because the physiochemical processes that occur during comminution (which include phase transitions, melting and solidification) can induce a range of solid state chemical reactions in the treated material as well as modifying its physical structure. Binders treated in this way can be incorporated into concrete mixtures, yielding new materials with promising properties [3], [4],

[18]. Moreover, their usage makes it possible to significantly reduce the proportion of Portland cement in concrete while maintaining and sometimes even improving the concrete's properties [19]-[22]. Both traditional powders (fly ash and blast furnace slag) and new alternatives (fine quartz sand, recycled concrete) can be used, potentially reducing the content of Portland cement in concrete by more than 50 % relative to standard formulations.

A product known as CemPozz is obtained by processing mixtures of pozzolans with 2%-5% ordinary Portland cement using EMC technology. Extensive field tests in the U.S. have shown that replacing up to 70% of the Portland cement in concrete mixtures with CemPozz significantly reduces cracking and greatly increases the concrete's capacity for self-healing of cracks [20].



(a)

(b)

Figure 3. The key steps in the creation of a durable and environmentally friendly concrete using Energetically Modified Cement (EMC) materials: (a) the overall procedure (b) the mechanical activation process.

2. EXPERIMENTAL

2.1 General

This paper reports the results of laboratory experiments and field tests on the self-healing performance of different types of concrete. Laboratory tests were performed by LTU and field tests by EMC Cement BV and Texas EMC Products LLC (USA). Microstructure assessments were performed by SINTEF, Norway as a continuation of the field test project, [22]. Preliminary results of these investigations were presented at the XXII Nordic Concrete Research Symposium in Reykjavik 2014 [18].

2.2 Test Program –self-healing in laboratory

Laboratory tests were conducted in 2012 and 2013 at LTU's Complab facility. EMC concrete beams were loaded 28 days after casting using the RILEM 3-point bending protocol [24], as shown in Figures 4 and 5. The loading induced crack formation, enabling assessment of the material's self-healing capabilities. The tested concrete (total amount of binder: 350 kg/m³, w/cm ratio 0.40, max aggregate size 16 mm) contained 40% Portland cement (Byggcement, type Cem II/A-LL, 42.5, Cementa) and 60% CemPozz made from 5% Anläggningscement (type Cem I 42.5N, SR3 MH/LA, Cementa) and 95 % low calcium fly ash. The mean and maximum diameters of the CemPozz particles were 17 microns and 35 microns, respectively, meaning that their size distribution closely matched that of the product examined in the field tests.

If the loading process failed to produce sufficiently wide cracks, the beam was returned to the test frame and the procedure described above was repeated. Once an acceptably large crack had formed, it was locked by gluing a small plate of carbon fiber to one side of the specimen (Figure 5). To assess the compressive strength of the concrete, unloaded control cubes were tested for compressive strength at different ages. The cracked beams were stored in the laboratory in a water bath at a temperature of 20 – 22 °C. The cracks were inspected with an optical microscope at various points in time between 0 and 135 days after their initial formation.

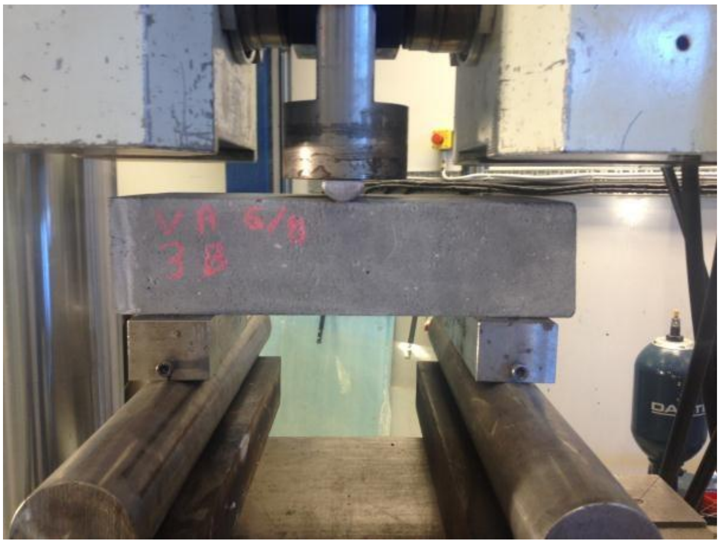


Figure 4. A concrete test beam made with Energetically Modified Cement (EMC) undergoing RILEM 3-point bending [23] at Complab, LTU.

Figure 5. The cracks were locked by gluing pieces of carbon fibre ***to*** *the sides of the beams.*

2.3 Field observation – self-healing

Field testing was performed on a highway pavement made of CemPozz concrete east of Houston, USA (see Figures 6 and 7). Three 20 km long sections of the pavement were paved with an ECM concrete made with CemPozz mixtures having different CaO contents. In addition, a fourth section was paved with a reference concrete containing no CemPozz. The reference concrete was prepared using a binder consisting of 80 % ordinary Portland cement (OPC) and 20% Class F ash (by mass) with a CaO content of 5%. The three CemPozz concretes were prepared using binders consisting of 50 % OPC and 50 % CemPozz; the CemPozz was prepared using Class F fly ash as a raw material, and had a CaO content of 2 %, 5 %, or 10 %.The regulations of the Texan road authorities limit the maximum CaO content of raw ash used in paving concrete, preventing the assessment of CemPozz with a CaO content above 10%. The total cementitious content of the CemPozz concrete was 300 kg/m^3, its OPC/CemPozz ratio was 1:1 by weight, and its w/cm ratio was 0.30.

The highway sections were paved over a period of four weeks during the summer time. Each section can be assumed to have experienced the same weather conditions and should thus have undergone the same levels of plastic and drying shrinkage. Due to the high humidity (80 – 85 %) during the paving process, the risk of plastic shrinkage cracking was considered to be rather low, and paving contractors working in this region generally do not take measures to protect against evaporation.

However, shortly after casting, all four of the concrete surfaces exhibited cracking, largely due to a combination of plastic and drying shrinkage. Three months after casting, samples with and without drying shrinkage cracks were drilled out from the pavements. Cylindrical samples with diameters of 100 mm and heights of 150 mm were tested in compression immediately after drilling and then subjected to curing in water at room temperature (20-22 °C). In addition, ten paved areas of 25 m^2 each were inspected to determine the average total number of cracks and average crack length at different time points after casting.

Figure 6. Pouring of self-healing concrete on highway IH-10, east of Houston, USA.

Figure 7. Reinforcement ahead of concrete placers on IH-10, east of Houston, USA.

2.4 Test Program – Microstructure Evaluation

To investigate the relationship between cement paste microstructure and the kinetics of crack healing, the microstructure of a cement paste made from a 50/50 blend of OPC and CemPozz was characterized at SINTEF, along with that of a paste prepared using a 50/50 blend of OPC and raw (unprocessed) fly ash The paste samples (cubes with 20 mm long sides) were prepared using CEM I 42.5N with a water-to-binder ratio of 0.40.

Paste samples for DTA/TG (Differential Thermal Analysis/Thermo Gravimetry) analysis were cured for set time periods then crushed to a fine powder and dried at 105 °C to remove physically adsorbed water. The DTA/TG experiments were conducted using a NETZSCH 409 STA with a heating rate of 10 °C/min until 1000 °C and nitrogen as a carrier gas. The sample (≈150 mg) was held in an alumina crucible during the analysis, and alumina powder was used as a reference material. The accuracy of the temperature determined for phase transitions was within ± 2 °C, while the accuracy of the mass loss measurements was within ± 0.3 mg.

MIP/HeP (Mercury Intrusion Porosimetry/Helium Pychnometry) experiments were performed using paste fragments around 5 mm in length. The MIP experiments were conducted with a Carlo Erba Porosimeter (Model 2000), which measures the sample's pore size (radius) distribution over a range of 5 - 50,000 nm, assuming cylindrical pores. The density of solid samples, ρ_s, was determined with a Micrometrics AccuPyc 1330 He-pycnometer, while the particle density, ρ_p, was determined with a Carlo Erba Macropores Unit 120. The accuracies of the total porosity and density measurements were within ± 0.5 and ± 0.01 units, respectively.

3. RESULTS

3.1 Self-healing in laboratory

Figures 8a-c show optical microscope images of a crack whose width ranges from 0.06 to 0.15 mm after different curing periods in the water bath. Due to the high chemical reactivity of CemPozz manufactured with low calcium fly ash (i.e. with a CaO content of ca. 3 %), the first signs of fresh C-S-H gel formation via pozzolanic reactions are apparent within 30 days of curing (see Figure 8b). Self-healing of the crack was complete after about 135 days, and the average penetration depth of the newly formed C-S-H gel was around 0.20 mm (see Figures 8b and c). Without any external intervention, the high volume pozzolan concrete gradually filled the cracks that had formed as the concrete set. It is believed that this was due to the ongoing formation of C-S-H gel within the concrete via pozzolanic reactions.

The high volume pozzolan concrete also exhibited continuous increases in strength over the observation period as a consequence of the ongoing pozzolanic reactions: the concrete had a strength of 85 MPa after 30 days' curing, rising to 94.5 MPa after 150 days. Such strength increases should appreciably increase the concrete's durability.

3.2 Field observation

The field studies conducted in the U.S. showed that the self-healing effect of the high-volume CemPozz concrete has a beneficial impact on concrete strength. Notably, the compressive strength of uncracked concrete samples was very similar to that of samples with self-healed cracks after 180 days of curing (see Table 1). This will undoubtedly increase the concrete's durability and abrasion resistance. The results obtained are consistent with previous findings [25], which indicated that concrete mixtures with higher fly ash contents exhibited enhanced self-healing leading to more effective recovery of compressive and bending strength as well as the dynamic modulus of elasticity after cracking.

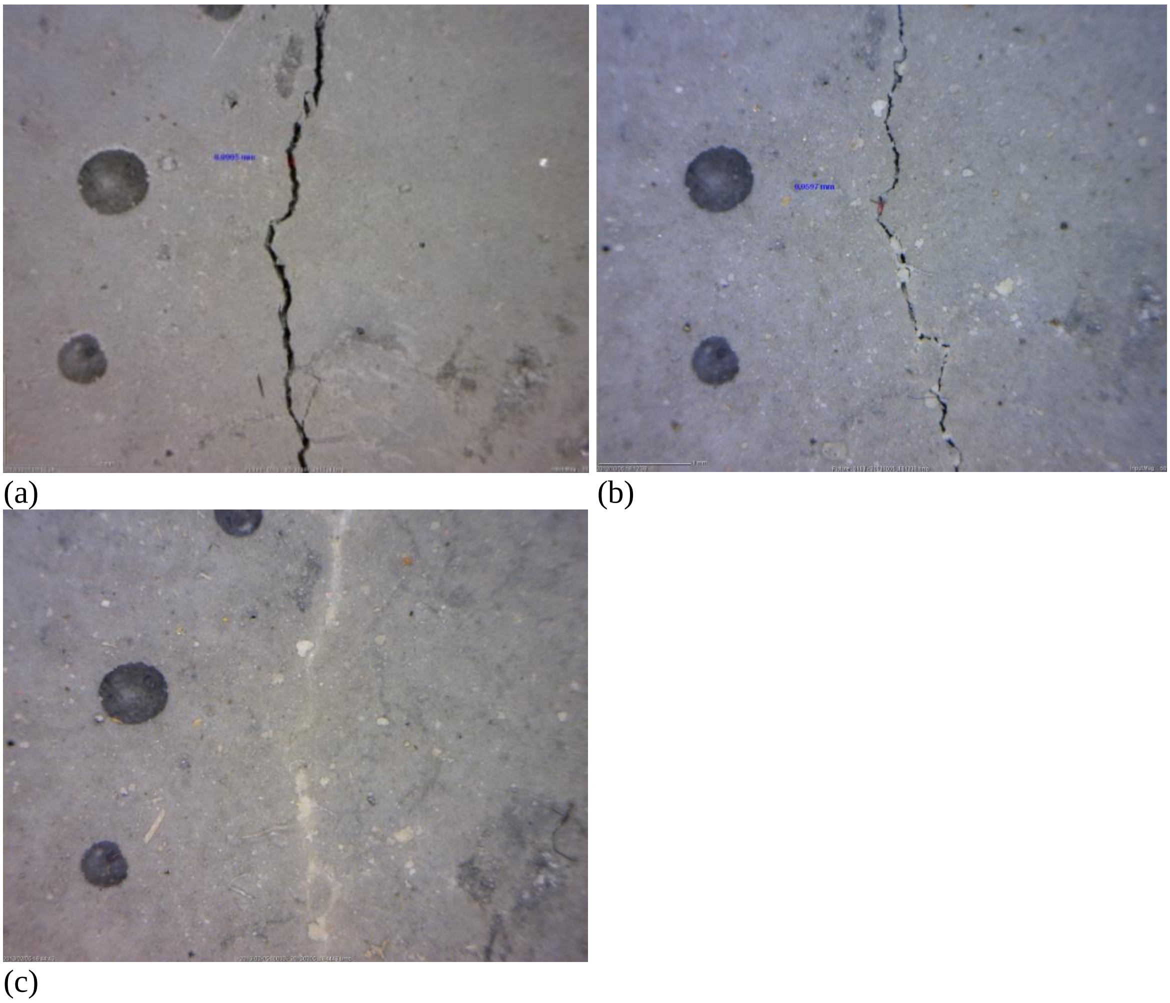

(a)
(b)
(c)

Figure 8. A concrete crack (a) shortly after its formation; (b) 30 days after its formation; and (c) 135 days after formation. Signs of [C-S-H gel] can be seen in (b) and the completely self-healed crack is observed in (c).

The strengths of the concretes examined in the field tests are shown in Table 1 and the kinetics of their self-healing processes are presented in Table 2. Visual inspection of the 25 m^2 observation areas of each pavement section clearly showed that at all ages, the 50% CemPozz concrete had substantially fewer and shorter cracks than traditional 20% fly ash concrete due to its lower levels of drying shrinkage [20]. Table 2 also demonstrates that the average number of cracks in the high volume pozzolan concrete (50 % CemPozz) decreases over time, which is a direct consequence of the self-healing of its microcracks. This increased self-healing ability is explained by the high pozzolanic activity of CemPozz manufactured with fly ashes having high calcium oxide (CaO) contents.

Table 1. Compressive strength of drilled cores collected during field tests in Texas

Type of sample	Compressive strength after 3 months in the field	Compressive strength after 3 months in the field and 3 months under water
Control without drying shrinkage cracks	45,6	53,8
Samples with drying shrinkage cracks	38,7	52,1

Table 2. Number and average lengths of cracks in the 25 m² observation areas of pavements with different CaO contents as recorded during field tests in Texas.

Curing period, months	Ref. concrete (20% fly ash), 5% CaO		CemPozz concrete, 2% CaO		CemPozz concrete, 5% CaO		CemPozz concrete, 10% CaO	
	Number of cracks	Average crack length, mm	Number of cracks	Average crack length, mm	Number of cracks	Average crack length, mm	Number of cracks	Average crack length, mm
2	12	24	8	17	9	15	6	11
3	14	22	10	18	10	15	8	12
6	12	18	7	12	5	11	5	8
9	11	17	6	10	4	8	4	6

3.3 Microstructure characterization

The results of Thermo Gravimetry, TG, and porosimetry experiments on the studied cement pastes after curing for up to 2.5 years (50/50 sealed/wet cured) are presented in Tables 3 and 4, respectively. EFAP denotes energetically modified 50/50 OPC/FAP paste, while BFAP is a 50/50 OPC/FAP blended paste.

As shown in Table 3, the total mass loss from the EFAP (energatically modified) paste was only slightly greater than that for the BFAP (blended) paste at most time points, and was actually slightly lower after 910 days' aging, probably because its denser matrix hindered its reactions. The calcium hydroxide (CH) content of EFAP exceeds that of BFAP after one day of aging but decreases more rapidly over time and is below the value for BFAP after 28 days. This indicates that the pozzolanic reaction of fly ash is faster in EFAP (in which it clearly begins after only 1-3 days of aging) than in BFAP (where the reaction apparently starts at some point between 7 and 910 days). This is understandable because the spherical shells of untreated fly ash particles are crushed during the milling process used to produce EFAP, allowing reactions to occur simultaneously on both sides of the glassy fly ash wall [22]. CH is consumed far more rapidly in EFAP paste than in BFAP: after 28 and 910 days' curing, its rate of consumption in EFAP is 13 % and 27 % greater than in BFAP, respectively.

Table 3 Thermal analysis results for Energetically modified (EFAP) and Blended (BFAP) pastes of 50% Ordinary Portland Cement (OPC) and 50% Fly Ash Paste (FAP) as a function of curing time. Results for the two pastes are separated by a slash (EFAP / BFAP).

Curing time	Total mass loss (%)	Degree of hydration (%)	CH (%)	CH/mass loss (%)
6 h / 12 h	6.89 / 7.30	28 / 29	4.11 / 3.90	60 / 53
1 day	8.64 / 7.96	69 / 64	8.65 / 6.92	100 / 87
3 days	9.93 / 9.73	79 / 78	9.28 / 8.67	93 / 89
7 days	10.38 / 10.20	83 / 82	9.18 / 8.62	88 / 85
28 days	11.15 / 10.89	89 / 87	7.37 / 8.25	66 / 76
910 days	13.46 / 14.27	108 / 114	3.99 / 5.78	30 / 41

The general trends revealed in Tables 3 and 4 are that the porosity decreases as a function of time and the specific surface area increases over time as the pores becomes smaller in size but more numerous (possibly indicating the development of gel pores). The average density of solids decreases as a function of time because the amount of crystal water in the paste increases as hydration proceeds. The EFAP paste is less porous than the BFAP paste after around 7 days due to its greater degree of hydration and more rapid pozzolanic reactions. The difference between the pastes is especially pronounced after 910 days.

Table 4 Specific surface area, S_g, particle density (ρ_p), solid density (ρ_s), mercury-accessible porosity (ε_{Hg}) and helium-accessible porosity (ε_{He}) of EFAP / BFAP pastes as a function of curing time.

Curing time	S_g (m^2/g)	ρ_p (kg/m^3)	ρ_s (kg/m^3)	ε_{Hg} (vol%)	ε_{He} (vol%)
6 h /12 h	8.4/9.7	1,300/1,231	2,588/2,519	48.2/47.7	49.8/51.1
1 day	20.0/15.5	1,302/1,243	2,373/2,359	43.7/44.7	45.2/47.3
3 days	32.8/22.7	1,349/1,313	2,264/2,260	39.3/38.4	40.4/41.9
7 days	30.6/20.7	1,377/1,383	2,235/2,248	37.6/35.9	38.4/38.5
28 days	40.2/27.2	1,349/1,371	1,931/2,102	31.7/34.7	30.1/34.8
910 days	35.7/44.7	1,324/1,180	1,609/1,856	23.2/40.5	17.7/36.4

The pore size distributions of the two samples are plotted in Figure 9, revealing that both pastes have a relatively fine pore structure: the average pore openings of EFAP and BFAP are 11 and 22 nm in diameter, respectively. However, the two pastes' pore size distributions are very different. BFAP has a bimodal distribution with substantial numbers of pores having diameters of around 600 nm and the rest being smaller than 100 nm. EFAP, on the other hand, has only a few pores with diameters above 40 nm. The reason why $\varepsilon_{Hg} > \varepsilon_{He}$ after 910 days' curing (see Table 4) is probably that the highly pressurized mercury used to determine ε_{Hg} crushes delicate structures that form in the paste after extensive curing, opening up otherwise inaccessible pores.

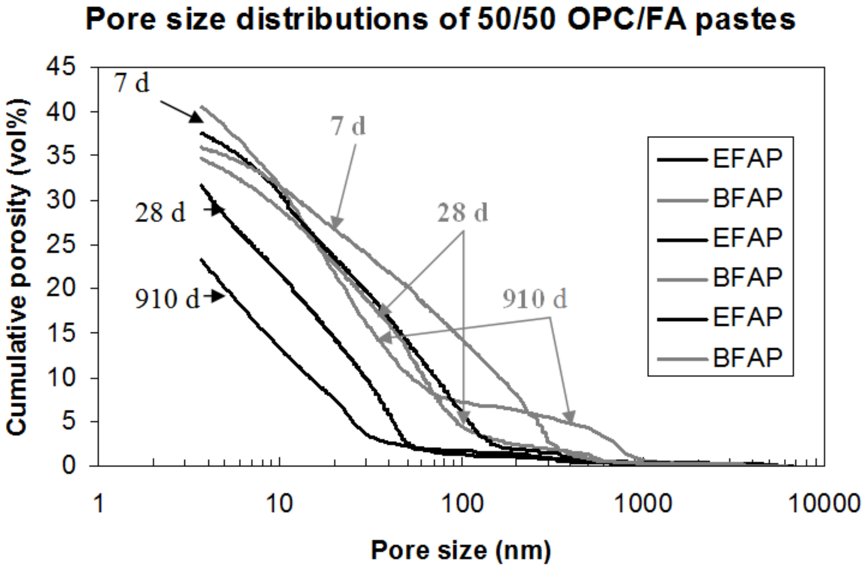


Figure 9. Pore size distribution in EFAP and BFAP pastes at 7, 28 and 910 days.

4. CONCLUSIONS AND FUTURE WORK

The results of the laboratory and field tests as well as visual inspections of the studied concretes indicate that:

1. Cracks in the surfaces of high volume pozzolan concrete beams were gradually filled in without any external intervention. It is suggested that this was due to the synthesis of C-S-H gel within the concrete, driven by ongoing pozzolanic reactions. The cracks were completely filled in after about 135 days.

2. Crack healing during field tests conducted in the United States was even more rapid than in the laboratory. This may be due to the comparatively low CaO content of the raw ash used in the concrete studied in the field test, and the higher curing temperature.

3. Self-healing of drying shrinkage cracks had a positive impact on the long-term strength of the tested concrete. After 90 days of curing, the compressive strengths of the control samples (without cracks) and concrete samples with self-healed cracks were about the same.

4. The number and length of cracks in paving made with 50 % CemPozz (EMC-processed pozzolan) decreased over time. This process was accompanied by a recovery of the concrete’s strength.

5. Observations of a highway pavement indicated that increasing the content of calcium oxide had a favorable effect on the kinetics of crack healing in concrete mixes with 50% Portland cement replacement.

6. Cement pastes containing 50% by mass of highly reactive EMC-treated pozzolans (i.e. CemPozz) exhibited high levels of hydration with lower levels of porosity and much finer pores than traditional OPC/Fly ash blends. These features are expected to increase the self-healing capacity and overall durability of EMC concrete.

Future studies will build on the results presented herein by seeking to validate the hypothesis that voids and cracks up to 0.2 mm wide can be filled by self-healing, and will assess the effect of such self-healing on the durability of concrete structures. In addition, efforts will be made to identify boundary conditions that can be used to select appropriate EMC concrete mixtures for various applications such as road construction.

5. ACKNOWLEDGEMENTS

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