Studying the Combination Effect of Additives and Micro Steel Fibers on Cracks of Self-Healing Concrete

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ABSTRACT

In this study, the effect of the combination of micro steel fibers and additives (calcium hydroxide and sodium carbonate) on the size of cracks formation and healing them were investigated. This study aims to apply the use of self-healing phenomenon to repair cracks and to enhance the service life of the concrete structures. Micro steel fibers straight type were used in this research with 0.2% and 0.4% by volume of concrete. A weight of 20 and 30 kg/m^3 of $Ca(OH)_2$ and 2 and 3 kg/m^3 of Na_2CO_3 were used as a partial cement replacement. The results confirm that the concrete cracks were significantly self-healed up to 30 days re-curing. Cracks width up to 0.2 mm were completely self-healed after re-curing for 90 days by using the combination of micro steel fiber of 0.4% by volume of concrete and 25 kg/m^3 of $Ca(OH)_2$ and 2.5 kg/m^3 of Na_2CO_3 as a partial replacement of cement. Products of Self-healing are observed by Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX). It was found that self-healing occurred mainly due to precipitation of calcium carbonate.

Key words: healing agents, cracking of concrete, self-healing concrete.

دراسة ألتأثير المشترك لكل من المواد المساعدة على شفاء الشقوق والالياف الحديدية الدقيقة على تكون الشقوق وشفاؤها في الخرسانة ذاتية الشفاء

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الخلاصة

في هذا البحث تم دراسة اصلاح شقوق الخرسانة ذاتيا باستعمال مواد مضافة تساعد على التئام الشقوق وتأثير ذلك على خواص الخرسانة ، حيث يهدف البحث من خلال تطبيق طريقة الشفاء الذاتي لشقوق الخرسانة الى تقليل اصلاحات الخرسانة وكلفتها و تحسين ديمومتها من خلال معالجة الشقوق و تقليل الاضرار الناتجة عنها.تم استخدام الالياف الحديدية الدقيقة و المواد المضافة المساعدة على شفاء الشقوق والمتمثلة ب(هيدروكسيد الكالسيوم وكاربونات الصوديوم) والتي تم استبدالها بنسب من وزن السمنت ،تم اضافة الالياف الحديدية بنسب 0.2% و 0.4% و المواد المساعدة على الشفاء بنسب استبدال 20كغم /م3 الى 30 كغم/م3 لهيدروكسيد الصوديوم ،وبنسب استبدال 2كغم/م3 و 3كغم/م3 لكاربونات الصوديوم و من ثم دراسة التأثير المشترك لكل من الالياف الحديدية الدقيقة والمواد المساعدة على الشفاء. النتائج اعطت تغير واضح جدا بسمك الشقوق بعمر 30يوم واعطت التئام تام للشقوق بعمر 90يوم ، حيث ان الشقوق لغاية سمك 0.2ملم تم شفاؤها تماما وافضل نتائج كانت للخلطات الحاوية على الياف حديدية بنسبة 0.4 والمواد المساعدة على الشفاء بنسبة 25 كغم/م3 من هيدروكسيد الكالسيوم و 2.5كغم/م3 من كاربونات الصوديوم. اظهرت نتائج فحص المجهر الالكتروني الماسح لتحليل المادة المالئة للشقوق ان كاربونات الكالسيوم هي المادة التي ترسبت في الشقوق وأدت الى غلقها وشفاؤها.

الكلمات الرئيسية: المواد المساعدة على الشفاء، تشقق الخرسانة، الخرسانة ذاتية الشفاء.

1. INTRODUCTION

1.1 General

Concrete is widely used as a building material due to its high strength, durability, availability, versatility. These properties added to the low production cost and recyclability make concrete the most commonly used building material in the world. However, concrete is quasi-brittle material, effective in compression and quite week in tension, and susceptible to many sources of damage, caused by external loading (dynamic or static loading), differential thermal exposure cycles, drying shrinkage, chemical attacks, corrosion, and other environmental conditions. Cracks are one of various types of damages, **Ramos et al., 2013.**Concrete elements, under bending or in tension loading condition, easily crack. Cracks formation is considered a natural feature of the concrete material. Because of this, reinforcements are installed. Inert reinforcements are activated once the concrete cracks.

1.2 Micro Cracks

Cracking plays an important role in concrete's response to load in both tension and compression. As matter of fact, reinforced concrete structures are linked with cracks from the beginning of their service life. The stresses transfer capability of these cracks may powerfully influence the service life of some concrete structures such as foundations, underground structures, and offshore structures, especially when water gets through cracks, this promotes corrosion process and accelerates the damage of concrete structures, **Homma et al., 2009.**

The crack width should not exceed a recommended limit. Too wide cracks may reduce the ability of the concrete material to protect the reinforcements against corrosion. The serviceability limit of concrete structures is mainly governed by the extent of their damages, **Tittelboom et al., 2013.**

Cracks in concrete structures may indicate main structural problems and also may ruin the monolithic construction appearance. In addition, they can expose reinforcements to oxygen and moisture, and so make them more susceptible to corrosion.

However, if it was possible to know and understand the reasons of the different behaviors of concrete structures, subjected to mostly similar loading conditions, it might have the basis to design high durability concrete structures with low or negligible repair and maintenance costs needed. Moreover, the limit of serviceability of the concrete material by cracking could be overcome by cracks width control methodologies. The improvement of service life of structures would decrease the demand for concrete cracks rehabilitation and repair. The employment of concrete self-healing technologies has high capability as an excellent repair technique for cracked concrete exposed to a water leakage of underground structures such as foundations, pipes, culverts, and tunnels, **Ahn et al., 2010.**

Fine cracks in fractured concrete, if admitted to close without tangential displacement may heal completely under moist conditions. This is known as autogenous healing. It is primarily due to the occurrence of hydration of the unhydrated cement clinker, which becomes exposed to water upon the opening of the cracks. Healing is also aided by the creation of insoluble calcium carbonate from the reaction of calcium hydroxide in hydrated cement if carbonation takes place. Some mechanical blocking of the cracks may also occur if very fine material is suspended in the water, **Neville, 2011.**

1.3 Self-Healing Phenomena

Self-healing concrete is one of the modern and smart concretes, in which the cracks can be healed by themselves. These are due to un-hydrated cement particles interaction with moisture in the crack, **Truong et al., 2013.**

Edvardsen, 1999, explained that calcium carbonate crystallization within the crack fracture surface was the major mechanism for self-healing of matured concrete. In particular, a calcite formation in the region of water-effecting cracks takes place in the material arrangement $CaCO_3$-CO_2-H_2O corresponding to the following reactions

$$Ca^{2+} + {CO_3}^{2-} \leftrightarrow CaCO_3 (pH_{water} > 8) \quad (1)$$

$$Ca^{2+} + {HCO_3}^{-} \leftrightarrow CaCO_3 + H^+ \qquad (7.5 < pH_{water} > 8) \quad (2)$$

A reaction between the calcium ions Ca2+, obtained from the concrete materials, and the in-water available carbonates CO_3^{2-}, or bicarbonates HCO_3^- produces the water-insoluble $CaCO_3$. Furthermore, CO_2 partial pressure in the water, pH value of the water, and water temperature favor the $CaCO_3$ precipitation in the crack. Calcium carbonate is a compound almost like stalactite, and has high cut off performance with great stability **Edvardsen, 1999.**

Also, there is the man-created self-healing concrete ability (autonomic healing). It includes a replacement of a small amount of cement or sand materials by self-healing additives. The self-healing concrete material is known as one of the recent smart concretes that can heal its cracks and other minor imperfections by itself. The healing is the results of the interaction of the moisture with the unhydrated cement clinker and, or other materials in the region of crack surfaces under some special circumstances, **Truong et al., 2013.**

2. MATERIALS

The materials used in this research are:

2.1Cement

Ordinary Portland cement produced in Iraq supplied from Taslooja cement factory was used in this work. It was stored in a dry and shaded place to avoid exposure to the atmospheric conditions like humidity. The chemical properties of the cement are shown in **Tables 1**; results conform to the, **Iraqi specification No.5/1984.**

2.2Fine Aggregate

Natural sand supplied from Al-Ekhadir quarry was used in concrete mixes of this work. The grading and physical and chemical properties of fine aggregate are shown in **Tables 2 and 3**. The test results indicated that the sand grading is within the limits specified by the **Iraqi Standard IQS No. 45/1984** and lies in Zone 3.

2.3 Coarse Aggregate

Natural gravel was used as coarse aggregate with a nominal aggregate size of (5-19 mm) for all mixes. It was obtained from Al-Nibaee region. The grading and physical and chemical properties of the used aggregate conform to **Iraqi Standard IQS No. 45/1984** as shown in **Tables 4** and **5**.

2.4Mixing Water

Ordinary drinking tap water was used throughout this work in the mixing of concrete.

2.5Calcium Hydroxide ($Ca(OH)_2$)

Conventional type of $Ca(OH)_2$ supplied from supplier was used.. **Fig. 1** shows Calcium hydroxide used in tests.

2.6 Crystallization Material Na_2CO_3

Conventional type of Na_2CO_3 supplied from supplier was used. **Fig. 2** shows Na_2CO_3 used in tests.

2.7 Micro Steel Fiber

Micro steel fiber straight type was used in this research with 0.2% and 0.4% by volume of concrete. The properties of the micro steel fiber used in research are shown in **Table 6** according to supplier. **Fig. 3** shows micro steel fiber used in tests.

3. MIX DESIGN

Design of Concrete mixes is made according to the **American Method ACI 211-91** to achieve cubs with compressive strength of 35 MPa , with slump of 75 to 100 mm, water to cement ratio (w/c) equal to 0.45, fineness modulus of fine aggregate equal to 2.4, maximum size of coarse aggregate equal to 19 and non-air-entrained concrete with unit mass equal to 2280 kg/m3. According to the mix design procedure, the mix proportion is (1:1.217:2.281). The mix proportions by weight of the concrete materials are given in **Table 7.**

4. THE EXPERIMENTAL PROGRAM

The experimental program consists of 7 concrete mixes **Table 8** gives the details for each mix.

4.1 Mixing Procedure

The mixing process was performed by a hand mixer. The saturated surface dry fine aggregate was placed in smooth, clean, non-absorbent metal pan, after that the cement was mixed with the required quantity of $Ca(OH)_2$ and Na_2CO_3 added to the fine aggregate. The saturated surface dry coarse aggregate was added to the mix, and the whole dry materials were well mixed for about two minutes. The required amount of water was added gradually and the whole constituents were mixed for further two minutes to get a homogenous mix, for the case of fiber reinforced concrete, the micro steel fibers were added manually to the mix. Then, the constituents' materials were mixed for about three minutes, until a homogenous concrete was obtained.

4.2 Curing

All specimens were cured in water with laboratory environments.

4.3 Cracking of Specimens

A compressive force was applied on the specimens of (100×100×100) mm concrete cubes using a digital compression machine till 40% of the ultimate load of each cube to form compressive micro-cracks in the concrete cubes. The cracking load was chosen according to **ACI Committee 224R-09 ,2009.**

4.4 LABROTATRY TESTS

4.4.1 Ultrasonic pulse velocity test

Concrete cubes (100×100×100) mm were used to determine the ultrasonic pulse velocity (UPV) test according to **ASTM C 597-02**, using a device commercially known as PUNDIT. Transducers (55 KHZ) were used to transmit and receive ultrasonic waves the direct method was applied, in which the transducers were placed on opposite faces of the concrete cube to be tested after lubricating with grease to get a smooth surface and to ensure the transition of the greatest amount of energy to the specimen. The method of measuring (U.P.V.) depends on the time (T) needed for the waves to pass a distance (L) in concrete. The following equation is used to calculate UPV:

$V = L/T$

Where:

V: Ultrasonic pulse velocity (km/sec);

L: Average length of specimen (mm);

T: Transit time (microsecond).

4.4.2 Microscopic observation

Cracks are introduced at age of 30 days from casting, and all the cracked cubes were observed by microscope and cracks widths were measured for all the cubes and the cracks up to 0.2 mm were fixed and measured again after 30 and 90 days from the day of cracking. A microscope with a lens that magnifies and clarifies the micro cracks up to 10X was used. The crack meter microscopic devise is shown in **Fig. 4.**

4.1.6 Scanning electron microscope

Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX) was used to observe the products of Self-healing. Two specimens of (2×2×1) cm were used; the first was from the cracked surface specimen of concrete mix M2, and the second sample was taken from the cracked surface specimen of concrete mix M6. The test was carried out in Physics Department in College of Science / University of Nahrain.

5. RESULTS AND DISSCUSIONS

5.1 Ultrasonic Pulse Velocity (UPV)

Ultrasonic pulse velocity test gives information about microstructure properties of concrete like density, porosity, and micro cracks. Also, there is a relationship between the increases of UPV and the increases of compressive strength, **Zhong and Yao, 2008.** So, the UPV was used to show the difference between concrete mixes and demonstrate the deterioration after cracking and the recovering of compressive strength and micro cracks of deteriorated samples after curing. Cubes specimens having dimensions (100× 100×100) mm were used for all concrete mixes (M1, M2, M3, M5, M6, M7, and M8) to measure the time between the transmitter and receiver transducers; from measured time, the wave velocity was calculated. The speed was calculated for each cube before cracking (at age of 28 days from casting), immediately after cracking (at age of 28 days from casting), and after 90 days of curing from cracking day (after healing). The changes in velocity before and after cracking reflect the inner micro cracks as the UPV related to the microstructure properties. For all concrete mixes the results show that the UPV decreases after cracking due to micro cracks producing. After 90 days of curing, the results of concrete mix M1 show very slight increasing in UPV as shown in **Fig. 5**. This could be most likely attributed to the very simple development in hydration process by hydration of unhydrated cement particles. For concrete mixes M2 and M3 the results show very clear increasing in UPV, and that indicates recovering of compressive strength, and healing of micro cracks due to the products results from self-healing reactions. Also, it indicates that the percentage of increasing in UPV increases with increasing of healing agents percentage so that the results of UPV of M3 are higher than that of M2 as shown in **Fig. 6**, and **Fig. 7**. For concrete mixes M5 and M6 the results show simple increasing in UPV and that refers to simple development in compressive strength. Also, the micro steel fiber bridges across some micro cracks and bonds the two sides of micro crack leading to simple healing in crack due to hydration of the unhydrated cement particles, **Homma et al., 2009**, but it still has a simple effects and appears through very small micro cracks. Also the results show that the concrete mix of higher micro steel fiber content M6 gives better regaining in UPV results after curing than concrete mix M5 as shown in **Figs. 8** and **9.** For concrete mixes M7 and M8 the results show a complete recovering of UPV and give the best results in comparison with other mixes. This could be most likely attributed to the recovering in compressive strength and healing of micro cracks resulting from the combination effect of micro steel fiber and healing agents. Concrete mix M8 gives higher recovering than mix M7 due to the higher Vf of micro steel fiber and higher content of healing agents as shown in **Figs. 10** and **11.**

5.2 Microscopic Observation

Cracks width was measured for the specimens of all concrete mixes after cracks production and after curing of 90 days from cracking. The measured cracks width for reference concrete (M1) after cracking and after curing of 90 days from cracking shown in **Fig. 12**. According to microscopic observation, no healing of cracks appears after curing, so, crack width after curing did not change in most of specimens. For concrete mixes M2 and M3 the crack width decreased due to the precipitation of products of self-healing, and the results indicate that the amount of healing agents affects the self-healing of cracks so concrete mix M3 give better results than concrete mix M2 and that attributed due to the higher concentration of Ca^{2+} and CO_3^{2-} ions. That leads to increasing the precipitation of self-healing products $CaCO_3$ as shown in **Figs.13** and**14.** In concrete mixes containing micro steel fiber without healing agents (M5) and (M6)the results show small changes in crack width before and after curing and that refers to the effect of micro steel fibers in bridging across the crack which decreases the crack width.

That leads to decreasing the distance between crack faces, connecting the particles to facilitate and accelerating the healing of cracks due to hydration products of unhydrated cement particles, **Homma et al, 2009**, but the healing appears only in small micro cracks. The concrete mix with higher percent of Vf of micro steel fiber (M6) shows the better results as shown in **Figs. 15 and 16**. For concrete mixes containing a combination of micro steel fiber and healing agents, the microscopic reading of cracks width give the better result due to the precipitation of self-healing products and the effect of micro steel fiber in bridging across the crack and facilitating the healing of micro cracks. The results gave a very remarkable changing in the width of cracks even in cracks of (0.25-0.3) mm and the microscopic image shows a very clear healing of cracks especially in concrete mix M8, with higher content of healing agents and higher Vf of micro steel fiber as shown in **Figs. 17** and **18. Figs. 19** and **20** show the microscopic images for self-healed cracks from concrete mixes M7 and M8**.**

5.3 Scanning Electron Microscope Results

The products of Self-healing are observed by Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX). For this observation, two samples were taken from the cracked surface of specimens of two concrete mixes. The first one was from the cracked surface of concrete mix M2, and the second sample was taken from the cracked surface of concrete mix M6 to observe the self-healing products for each sample. **Fig. 21** and **Table 9** show the EDX analysis for the chemical composition of self-healed zone in cracks of sample from concrete mix M6. The SEM image shows distribution of self-healing products along the crack surfaces resulting from partial self-healing of crack. The results show that self-healing products are composed of (C-S-H), and $CaCO_3$. This indicates that the hydration of unhydrated cement particles lead to producing (C-S-H) in the micro cracks, **Huang et al, 2013** and also the combination of calcium ions (Ca^{2+}) which are derived from the concrete and the carbonate ions (CO_3) which are derived from the in water dissolved CO_2 lead to precipitate $CaCO_3$ (Edvardsen, 1999).

6. CONCLUSIONS

Based on the results obtained from experimental investigations, the following conclusions can be stated:

1. The self-healing of cracks was confirmed.
2. The UPV is increased as the age of curing in water increases and the maximum increasing in UPV for all concrete mixes was at 90 days. The percentage of increasing in UPV increases with increasing of healing agents' percentage.
3. The compressive strength increased for micro steel fiber reinforced concrete specimens, and higher Vf gives higher percent of increasing since the first week.
4. Specimens of concrete with higher percentage of calcium hydroxide, and sodium carbonate give better sealing of cracks than other specimens. The increase in compressive strength appears slightly in the first week and the sealing percentage increases with increasing the time of curing.
5. The specimens of concrete with only micro steel fibers give a slight decreasing in cracks due to further hydration of unhydrated cement particles.

6. The best result of cracks sealing was for specimens having a combination of micro steel fibers and with healing agents ($Ca(OH)_2$, and Na_2CO_3), due to the further effect of micro steel fibers in addition to the effect of healing agents.
7. The healing products are mainly composed of calcium carbonate for concrete mix with calcium hydroxide, and sodium carbonate additives, so the healing of cracks is mainly attributed to the precipitation of calcium carbonate due to the reaction of calcium ion with carbonate ions.
8. The results of UPV of self-healed specimens show high recovering in compressive strength and the maximum recovering occurs in concrete mixes containing a combination of micro steel fibers and healing agents ($Ca(OH)_2$, and Na_2CO_3).

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Table 1. Chemical composition and main compounds of ordinary Portland cement used in this research.

Oxide composition	Result	Limits of IQS No. 5/1984
Lime (CaO)	62	-
Silica (SiO_2)	20.1	-
Alumina (Al_2O_3)	4.24	-
Iron oxide (Fe_2O_3)	4.16	-
Sulfate (SO_3)	2.15	≤ 2.8%
Magnesia (MgO)	3.65	≤ 5%
Loss on ignition (L.O.I.)	3.42	≤ 4%
Lime saturation factor (L.S.F.)	0.89	0.66-1.02
Insoluble residue (I.R.)	0.71	≤ 1.5%
Main compounds (Bogues eq.) of cement ASTM C 150-00		
Tricalcium silicate (C3S)		59.02
Dicalcium silicate (C2S)		29.65
Tricalcium aluminate (C3A)		4.21
Tetracalcium aluminoferrite (C4AF)		12.65

Table 2. Grading o fine aggregate.

Sieve size (mm)	Passing % of sand	Limits of IQS No. 45/1984/Zone 3
4.75	97.4	90-100
2.36	90.2	85-100
1.18	79.1	75-100
0.6	61.88	60-79
0.3	21.7	12-40
0.15	3.22	0-10

Table 3. Physical and chemical properties of natural sand.

Properties of sand	**Test result of sand**	**Limits of IQS No. 45/1984**
Fineness modulus	0.2	-
Specific gravity	2.414	-
Absorption	1.01%	-
SO_3	0.28 %	≤ 0.5%
Dry rodded density	1680 kg/m³	-

Table 5. Grading of coarse aggregate.

Sieve size (mm)	**Passing % of gravel**	**Limits of IQS No. 45/1984 (5-20) mm**
37.5	100	100
20	100	95-100
10	41.8	30-60
4.75	2.5	0-10

Table 6. Physical and chemical properties of coarse aggregate.

Properties of gravel	**Test result of gravel**	**Limits of IQS No. 45/1984**
Specific gravity	2.606	-
Absorption	0.8%	-
SO_3	0.06%	≤ 0.1%
Dry rodded density	1600 kg/m³	-

Table 6. Properties of micro steel fiber.

Length	13 mm
Diameter	0.2 mm
Density	6800 kg/m^3
Tensile strength	2600 MPa
Aspect ratio	65

Table 7. The mix proportion by weight of concrete materials.

The materials	The mix proportion by weight (kg/m^3)
Water	205
Cement	456
Coarse aggregate	1040
Fine aggregate	555

Table 8. Details of mix design for concrete mixes .

Mix No.	W/C	Micro steel fiber % by volume	$Ca(OH)_2$ kg/m^3 as partial Replacement of cement	Na_2CO_3 kg/m^3 as partial replacement of cement
M1	0.45	0	0	0
M2		0	20	2
M3		0	30	3
M5		0.2	0	0
M6		0.4	0	0
M7		0.2	25	2.5
M8		0.4	25	2.5

Table 9. Chemical composition of self-healing products of M6.

Oxides	Percentage (%)
CaO	73.9
SiO_2	20.8
Al_2O	3.9
MgO	1.4

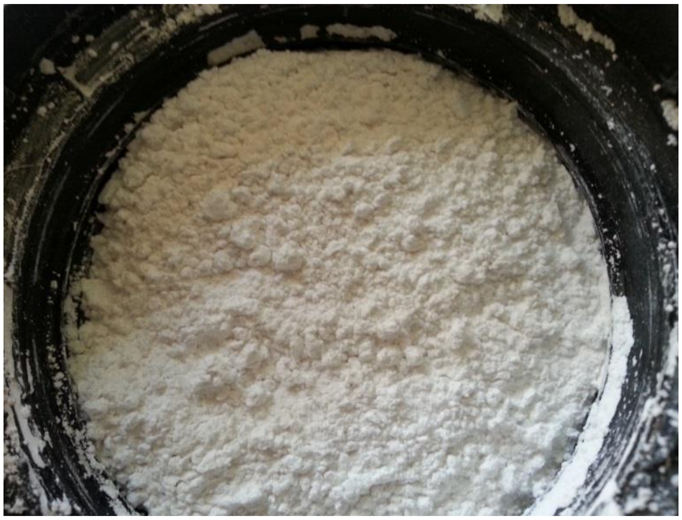

Figure 1. Calcium hydroxide.

Figure2. Sodium carbonate.

Figure3. Micro steel fibers.

Figure4. Microscopic observation device.

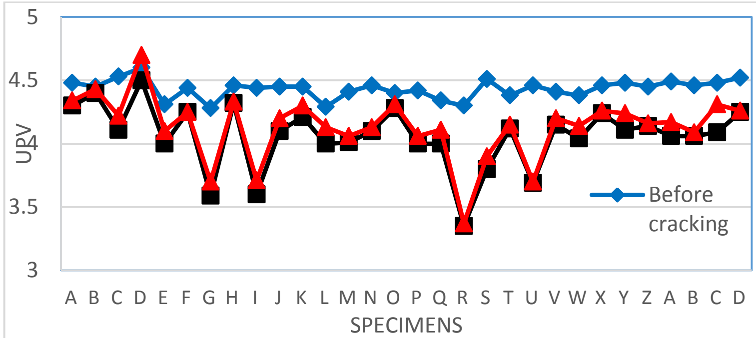

Figure5. The changes of UPV before and after cracking for M1.

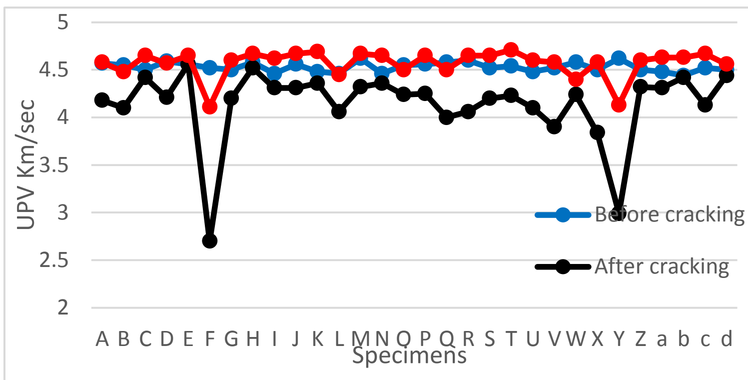

Figure 6. The changes of UPV before and after cracking for M2.

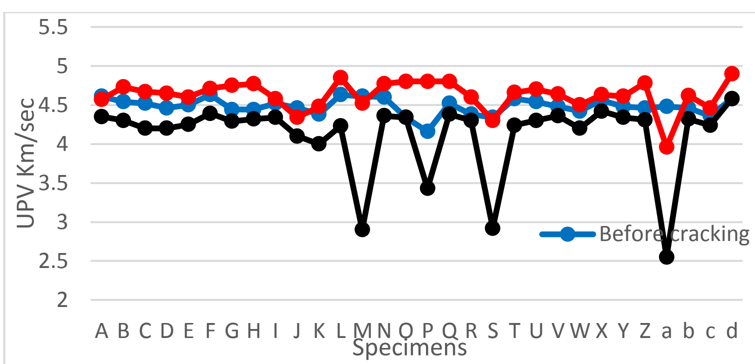

Figure 7. The changes of UPV before and after cracking for M3.

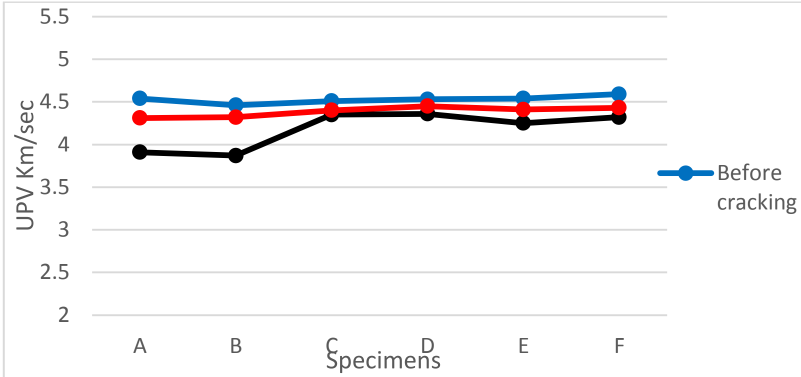

Figure 8. The changes of UPV before and after cracking for M5

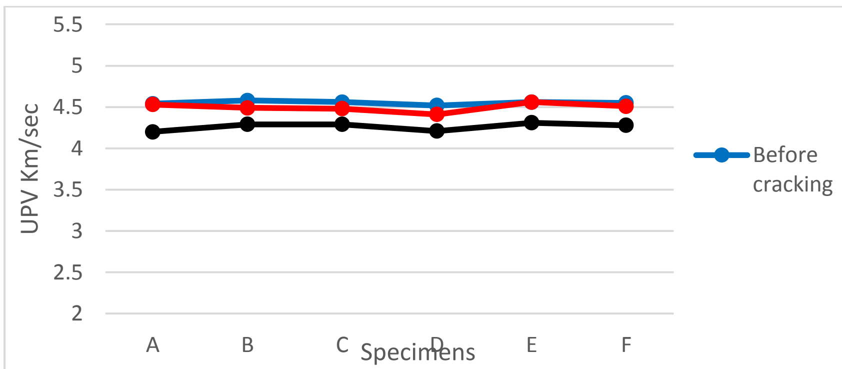

Figure 9. The changes of UPV before and after cracking for M6

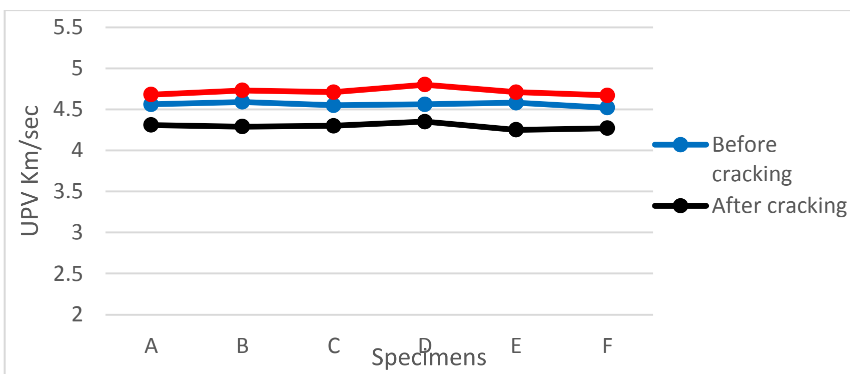

Figure 10. The changes of UPV before and after cracking for M7.

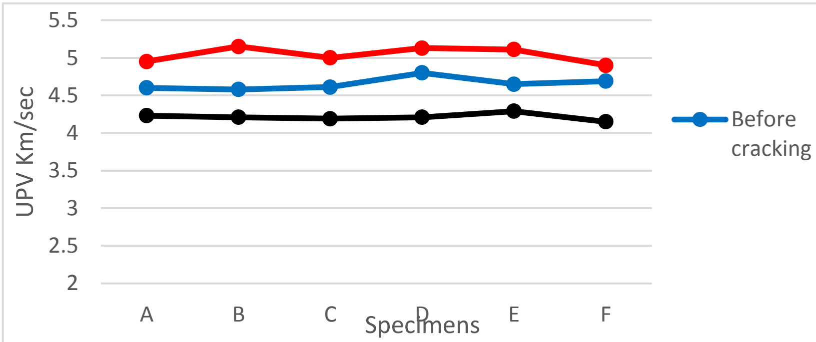

Figure 11. The changes of UPV before and after cracking for M8.

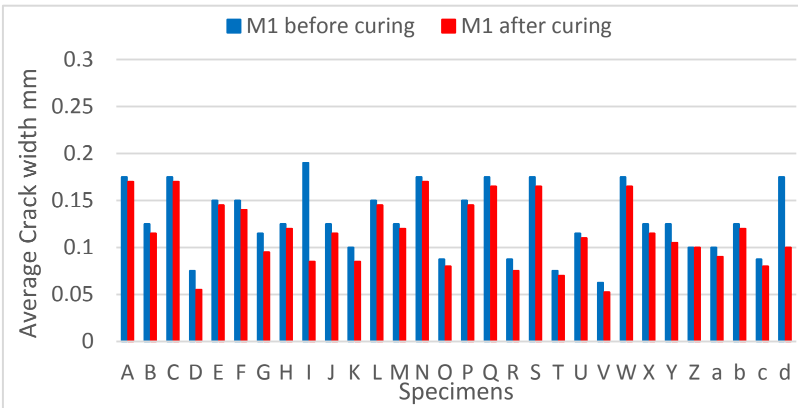

Figure 12. Changes of crack widths for concrete mix M1.

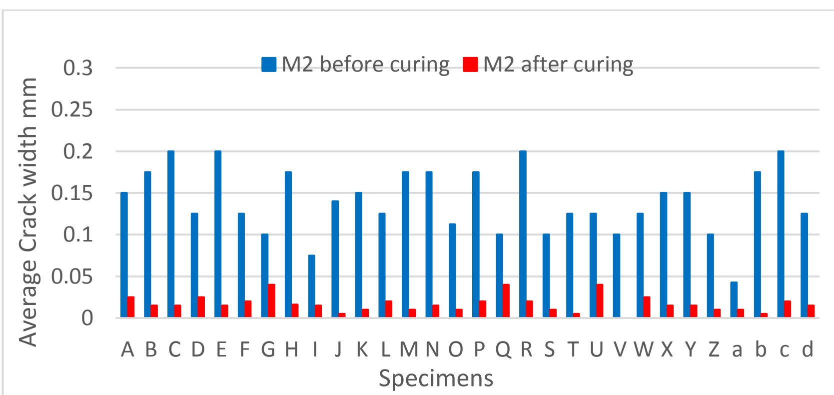

Figure 13. Changes of crack widths for concrete mix M2

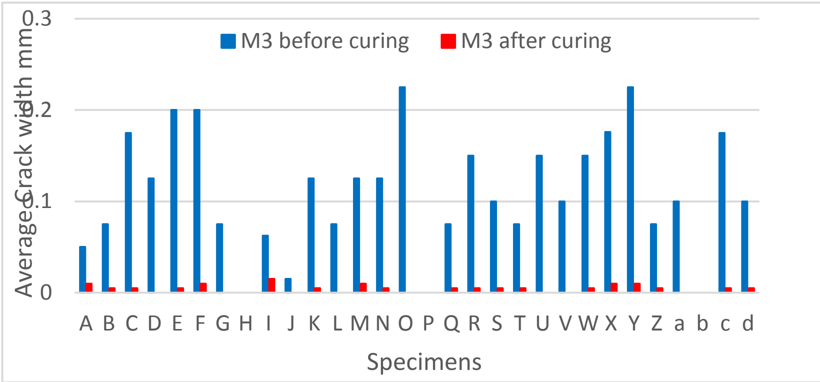

Figure 14. Changes of crack widths for concrete mix M3

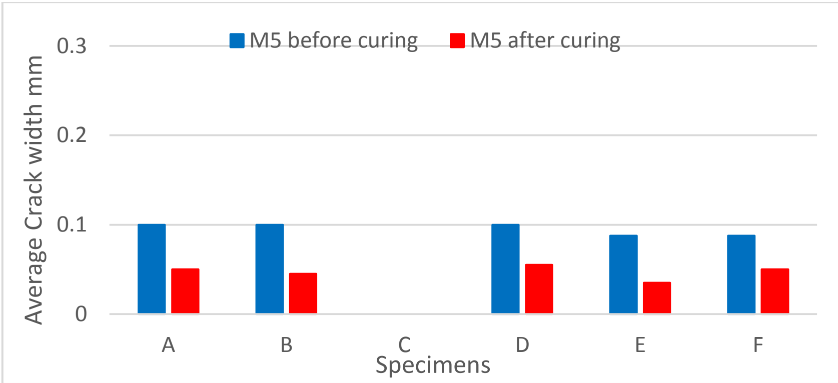

Figure 15 . Changes of crack widths for concrete mix M5.

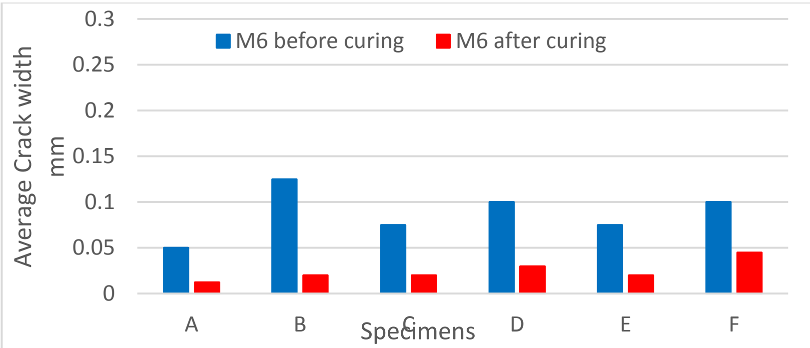

Figure 16 . Changes of crack widths for concrete mix M6.

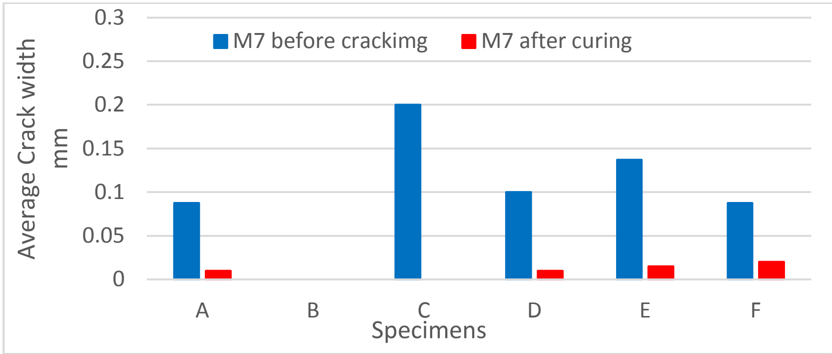

Figure 17 . Changes of crack widths for concrete mix M7.

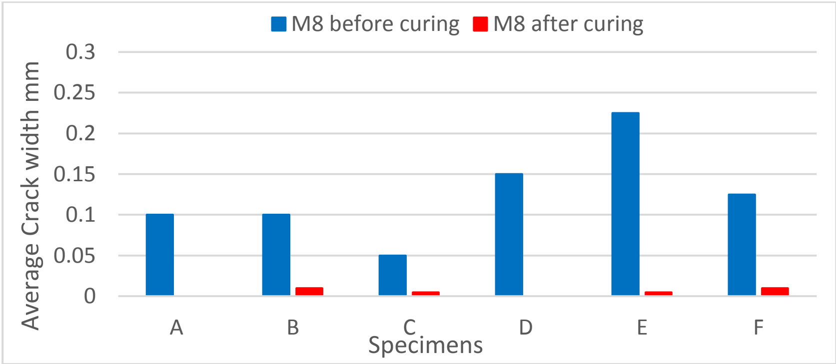

Figure 18. Changes of crack widths for concrete mix M8.

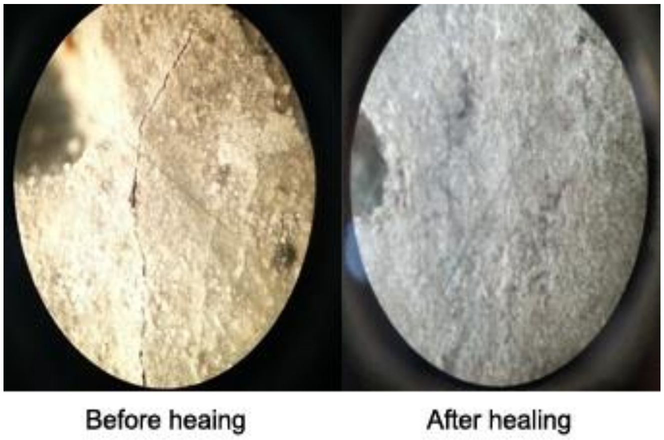

Figure 19. Microscopic image for self-healed sample from concrete mix M7.

Figure 20. Microscopic image for self-healed sample from concrete mix M8.

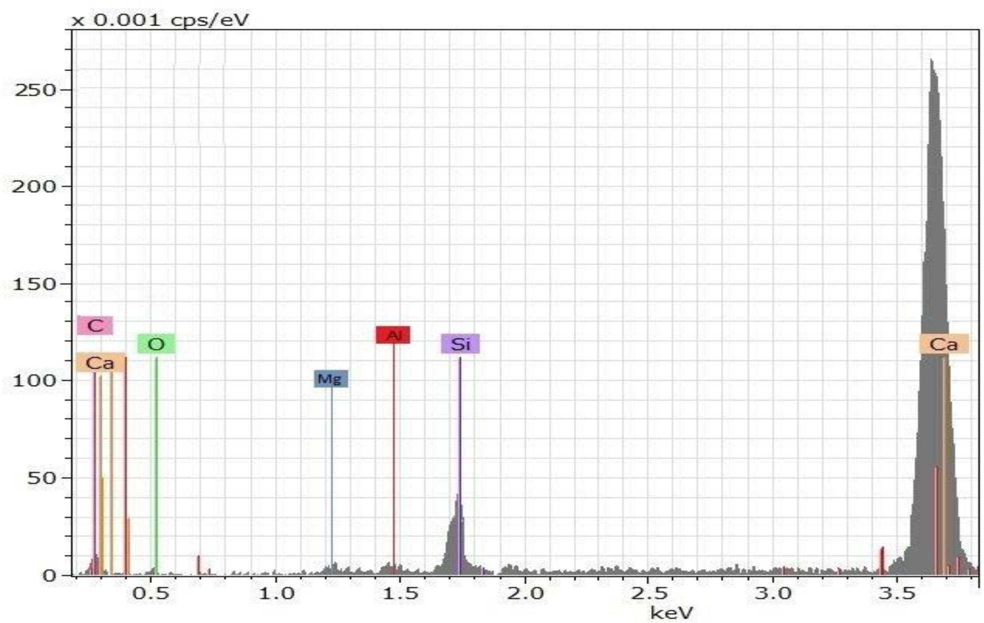

Figure 21. EDX analysis for self-healing products of M6.