Cement & Concrete Composites 32 (2010) 686–693

Contents lists available at ScienceDirect

Cement & Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

Influence of curing condition and precracking time on the self-healing behavior of Engineered Cementitious Composites

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ARTICLE INFO

Article history:
Received 18 March 2010
Received in revised form 14 July 2010
Accepted 17 July 2010
Available online 25 July 2010

Keywords:
Self-healing
Engineered Cementitious Composites (ECC)
Precracking time
Curing condition
Nanoclay

ABSTRACT

This paper investigates the self-healing behavior of Engineered Cementitious Composites (ECC) with focus on the influence of curing condition and precracking time. Four-point bending tests were used to precrack ECC beams at different age, followed by different curing conditions, including air curing, 3% CO_2 concentration curing, cyclic wet/dry (dry under 3% CO_2 concentration) curing and water curing. For all curing conditions, deflection capacity after self-healing can recover or even exceed that from virgin samples with almost all precracking ages. After self-healing, flexural stiffness was also retained significantly compared with that from virgin samples, even though the level of retaining decreases with the increase of precracking time. The flexural strength increases for samples pre-cracked at the age of 14 days and 28 days, presumably due to continuous hydration of cementitious materials afterwards. Furthermore, it is promising to utilize nanoclay as distributed internal water reservoirs to promote self-healing behavior within ECC without relying on external water supply.

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1. Introduction

Self-healing phenomenon of cement-based materials has been observed in natural environments for many years. For civil infrastructures, e.g. bridge decks, pavements and tunnel lining, where water and carbon dioxide are normally available, concrete is able to heal its own damage with chemical products by itself. The main cause of self-healing was attributed to the formation of calcium carbonate, a result of reaction between calcium ion in concrete and carbon dioxide dissolved in water [1,2]. Continuous hydration of unhydrated cementitious materials was another mechanism for the self healing to occur [3]. Due to the limited availability of self healing products, the control of crack width was found essential in obtaining consistent and robust self-healing behavior in cementitious materials [4,5]. To consistently control the crack width, even with the presence of steel reinforcement is relatively difficult in normal concrete. Engineered Cementitious Composites (ECC), a new class of ductile fiber reinforced concrete material, can however achieve self-controlled tight crack width with the aid of micromechanics theory [6]. Li and coworkers [4,7–9] have investigated the self-healing behavior of ECC under a number of exposure conditions, including water submersion, wetting and drying cycles, chloride ponding and high alkaline environment. The mechanical and transport properties can be largely recovered, especially for ECC specimens preloaded to below 1% tensile strain.

In an effort to develop green ECC with local waste materials, Zhou et al. [10] have developed a number of ECC mixtures with blast furnace slag (BFS) and limestone powder, all characterized with 2–3% tensile strain capacity and tight crack width (typically below 60 μm). With these green ECC mixtures, Qian et al. [5] have demonstrated via four-point bending test that it is possible to achieve self-healing behavior if the pre-cracked samples were submerged in water for a certain period, while this is not the case for air cured samples.

Besides exposure conditions, the influence of precracking time on healing behavior may be of interest to researchers due to the randomness of cracking time within concrete structures as well as the decreasing quantity of unhydrated cementitious materials with time. In studying the self-healing potential of early age concrete, Heide and Schlangen [11] observed a better recovery of flexural strength in concrete beams pre-cracked at earlier age, with all samples clamped and then submerged in water. In studying the influence of early-age compressive load on the healing behavior of concrete, Jin et al. [12] found the age of preloading has more significance compared with that of the preloading level.

While water is normally assumed readily available in most self healing studies, this may not necessary be the case at all times. Some researchers attempted utilizing internal water reservoirs to promote self-healing behavior rather than relying on external water supply. For instance, De Rooij et al. [13] tried employing

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doi:10.1016/j.cemconcomp.2010.07.015

pre-wetted and coated short wood fibers as water reservoirs in cementitious materials to promote self-healing behavior. The wood fiber was installed with water and/or other healing compounds within its hollow cell structures and then coated to prevent premature water vaporization. Valcke et al. [14] tried to use nanoclay as internal water supply for pre-cracked mortar samples to heal itself. According to their study, in a homogenized clay/water mix, the nanoclay takes up ~95–98% of its own mass as interlayer water and several times more water of its own mass to get good dispersion and avoid flocculation. These large amount of extra water are considered free water, available for cement hydration.

In this paper, the influence of curing condition and precracking time on the self-healing behavior of ECC was investigated. Four different curing conditions were employed in the investigation, including air curing, 3% CO_2 curing, cyclic wet/dry curing (dry under environment of 3% CO_2 concentration) and water curing. The precracking time varied from 14 days, 28 days and 56 days in order to reveal the effect of timing of damage on the self-healing behavior of ECC. Furthermore, nanoclay was added in the mixtures to investigate its feasibility to act as internal water reservoirs to promote self-healing behavior of ECC, eliminating the dependence on the external water supply. In the following sections, the experimental program of this investigation will be introduced in details, including material preparation and four-point bending test. Furthermore, experimental results on mechanical behaviors, such as deflection capacity, flexural strength, stiffness and crack pattern will be presented and discussed. Finally, overall conclusions will be drawn based on the experimental results and discussion.

2. Experiments

2.1. Material proportion and specimen preparation

In Table 1 Ma and Mb were the mixtures investigated in this paper, while the rest mixtures M1-4 from Qian and Zhou et al. [5,10] is shown here for comparison purpose. Portland cement CEM I 42.5 N was used in the mixture along with blast furnace slag (BFS), limestone powder and nanoclay. The nanoclay is a purified Na-montmorillonite with a small particle size (ca 500 nm). The purpose for introducing small amount of nanoclay is to investigate the feasibility of utilizing its water retaining capacity to enhance the self-healing behavior of ECC, without relying on external supply of water. As can be easily seen from Table 1, indeed significant amount of extra water is needed to achieve similar visual workability in Ma and Mb in comparison with M0 due to the addition of nanoclay. The polyvinyl alcohol (PVA) fiber with a length of 8 mm and a diameter of 40 μm was used in the content of 2% by total volume (1.3% by weight).

The solid materials, CEM I 42.5, BFS, limestone powder and nanoclay were first mixed with a HOBART mixer for 2 min thoroughly. Water and superplasticizer were then added and the mixture was mixed at low speed for 1 min, followed by high speed for 2 min. Finally, fibers were added at low speed and the mixture was mixed at high speed for another 2 min. Coupon specimens with the dimension of 240 mm × 60 mm × 10 mm were cast for four-point bending tests. After 1 day curing in moulds covered with plastic paper, the specimens were cured under sealed condition (RH 98%) at 20 °C for another 13, 27 or 55 days, respectively before precracking.

Once the precracking time was reached, each coupon specimens was evenly cut into four pieces with the dimension of 120 mm × 30 mm × 10 mm. These specimens were used in four-point bending test. The support span of four-point bending test set-up was 110 mm and the middle span was 30 mm. The deflection capacity was calculated based on the average results of at least five measurements. The deflection capacity was defined as the deflection at which point the bending stress reaches maximum (MOR).

2.2. Four-point bending test (FPBT)

The overall program for FPBT is shown in Fig. 1, including three precracking ages (14 days, 28 days and 56 days) and four curing schemes (B–E) and control scheme A, resulting in 15 combinations. In the scheme A, five samples of both mixtures were bent until final failure to derive the flexural stress-deflection relation. The results from this scheme form a reference value to determine how much healing is occurred in terms of deflection capacity, strength and flexural stiffness. For control scheme A, time of precracking has no meaning since all samples were tested until final failure.

In case of scheme B, C and E five samples from both mixtures were bent (pre-cracked) up to 1.5 mm and unloaded. The deflection of 1.5 mm is well below ultimate deflection of the mixture at different precracking ages. Afterwards the pre-cracked samples were further cured in air (RH 30% at 20 °C, scheme B), CO_2 of 3% concentration (RH 50% at 20 °C, scheme C) and water (scheme E), respectively for 28 more days before testing to final failure. The curing condition D is the combination of scheme C and E, meaning 2 days water curing followed by 2 days drying in 3% CO_2 to simulate the natural cycle of raining and extended drying.

Scheme B looks to investigate the feasibility of utilizing nanoclay alone to promote selfhealing, without relying on the external water supply, as in the case of scheme E. In the case of scheme C, it simulates the effect of accelerated carbonation on the self-healing behavior of ECC. In the FPBT, the sample was tested under deformation control at a constant rate of 0.01 mm/s.

3. Results and discussion

3.1. Flexural strength and stiffness

The flexural strength from mixture Ma and Mb under different curing conditions and precracking time is compared in Figs. 2 and 3. The flexural strength from air, CO_2, wet/dry and water curing (conditions B–E) is larger when compared with that of reference (condition A) for precracking time of 14 and 28 due to continuous

Table 1
Mix proportion of ECC by weight percentage.

Material	Cement (CEM-I 42.5 N)	Limestone powder	Blast furnace slag	Nanoclay	Water	Super-plasticizer	PVA fiber
Ma	**25.0**	**20.0**	**30.0**	**0.6**	**22.5**	**0.7**	**1.3**
Mb	**24.5**	**19.6**	**29.3**	**1.2**	**23.2**	**0.9**	**1.3**
M1	26.5	21.2	31.8 (Fly ash)	0.0	18.3	0.9	1.3
M2	21.0	31.5	25.2	0.0	20.6	0.5	1.3
M3	18.6	37.2	22.3	0.0	20.3	0.3	1.3
M4	15.1	45.2	18.1	0.0	20.0	0.3	1.3

2% PVA fiber content by volume is approximately 1.3% by weight. Mixes M0-4 have been studied by Qian et al. [5] and Zhou et al. [10].

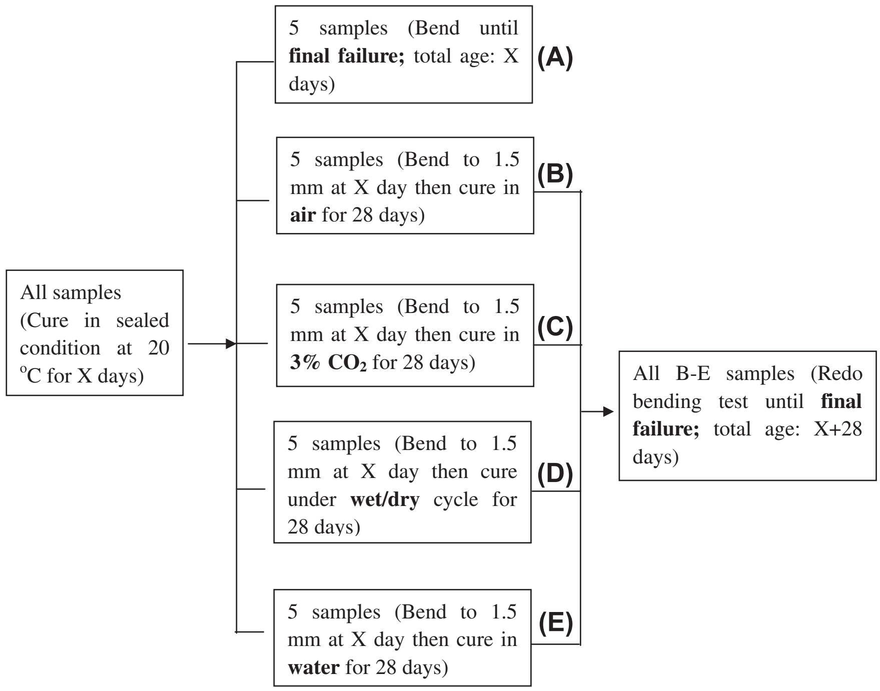

Fig. 1. Bending test program of self-healing cementitious composites (*X*: precracking time, varies from 14, 28 and 56 days).

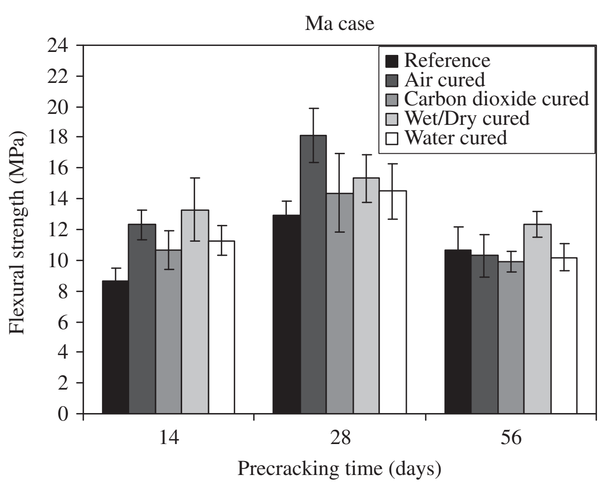

Fig. 2. Comparison of flexural strength of mixture Ma for different curing conditions and precracking time.

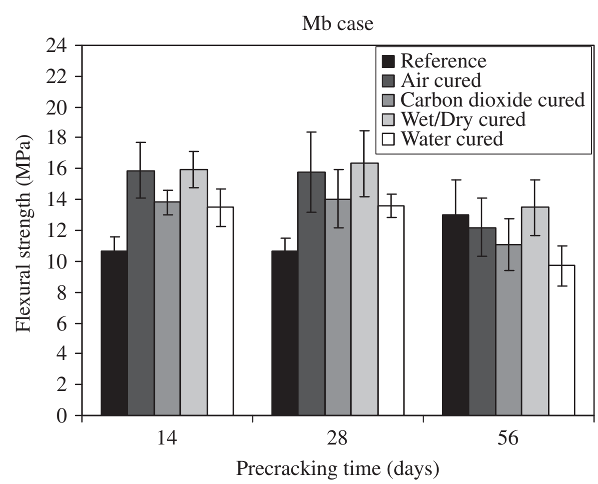

Fig. 3. Comparison of flexural strength of mixture Mb for different curing conditions and precracking time.

hydration of unhydrated cementitious material. As expected this trend is neutralized when the precracking time reaches 56 days.

When the samples were pre-cracked to 1.5 mm at 14 and 28 days, the deflection capacity of all samples under air, CO_2, wet/dry and water curing (conditions B–E) was not reached. With the continuous hydration of cementious materials in both mixtures, the flexural strength was enhanced in these conditions compared with that of the reference (condition A), which was totally fractured at age of 14 and/or 28 days. In the case of precracking time 56 days, the flexural strength of samples under these conditions is generally comparable to that of reference (condition A) due to the high maturity of the sample.

The effect of self-healing can also be seen from the level of flexural stiffness retained after air, CO_2, wet/dry and water curing (conditions B–E) compared with the reference sample as shown in Figs. 4 and 5. The flexural stiffness is defined as the initial slope of flexural stress-deflection curve when the flexural stress is between 1 and 6 MPa, where the slope is nearly linear for all cases. As shown in Figs. 4 and 6, the level of stiffness retained under air, CO_2, wet/dry and water curing is generally comparable with experimental results of Li and Yang [4] for the precracking age of 28 days, even though the actual curing conditions may not be the same.

As shown in Figs. 4 and 5, the level of stiffness retained after air, CO_2, wet/dry and water curing (conditions B–E) decreases with time of precracking for both mixtures due to the gradual exhaustion of cementitious material for continuous hydration. The flexural stiffness of the wet/dry curing (condition D) is much higher compared with the other curing conditions, caused by the cycle of wetting and drying under 3% CO_2 concentration. This curing con-

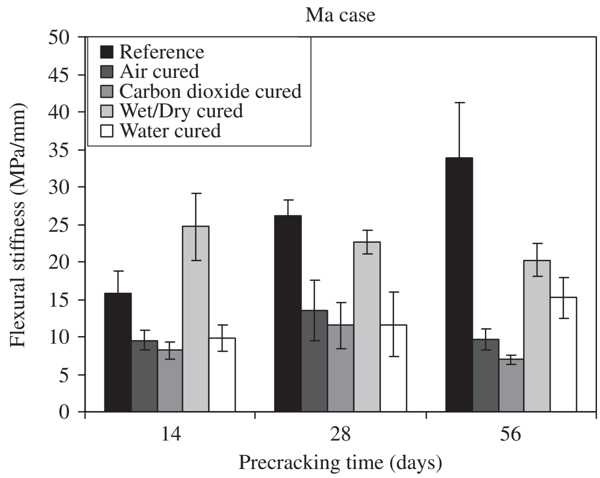

Fig. 4. Comparison of flexural stiffness of mixture Ma for different curing conditions and precracking time.

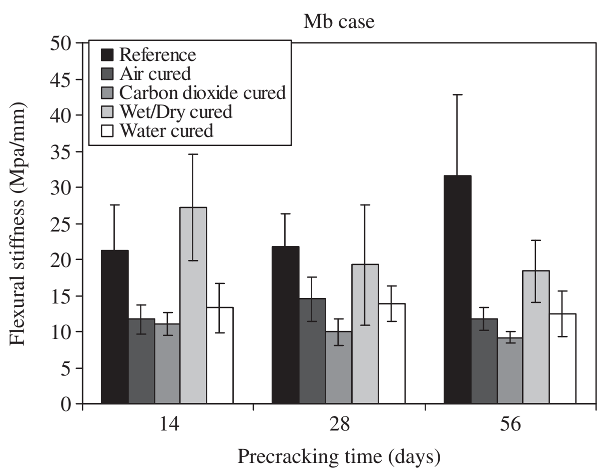

Fig. 5. Comparison of flexural stiffness of mixture Mb for different curing condition and precracking time.

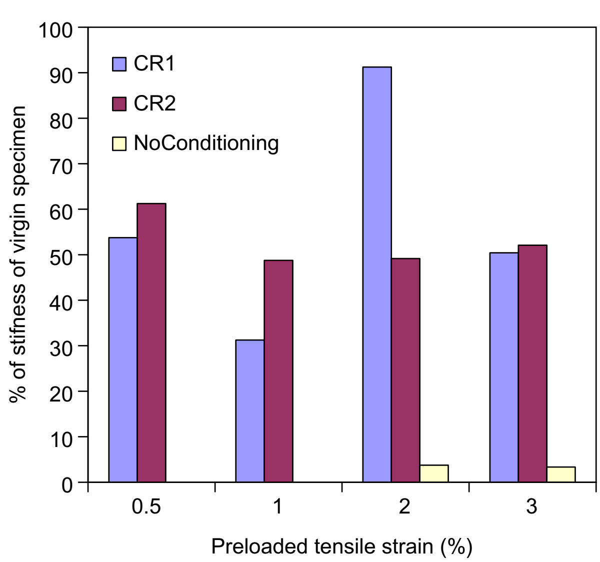

Fig. 6. Stiffness recovery of ECC pre-cracked at 28 days under different conditioning regime (Li and Yang [4]).

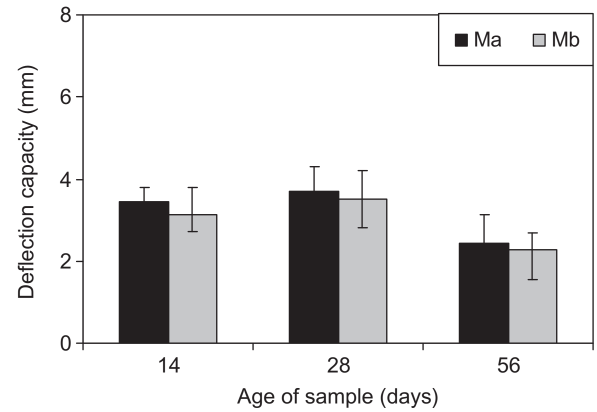

Fig. 7. Development of deflection capacity with time (Reference case).

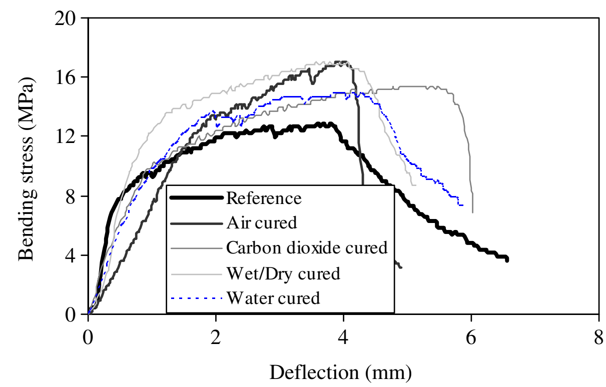

Fig. 8. Bending stress-deflection curves of mixture Ma for precracking age of 28 days.

dition can effectively promote the interaction of water, CO_2 and remaining unhydrated cementitious material inside ECC material (including matrix and fiber-matrix interface), therefore resulting in enhanced flexural stiffness. Furthermore, it can be seen in Figs. 4 and 5 that the flexural stiffness of reference A increases with precracking time.

As shown in Figs. 4 and 5, it can be seen that the 14-day flexural stiffness of wet/dry curing condition is higher than that of reference A at the same age, but smaller compared with flexural stiffness of the reference samples at 56 days. This is as expected, as from the literature [4,7], the main component of self healing products are usually calcium hydrates or calcium carbonates, which is typically weaker than that of CSH from the cement hydration. Even if CSH is formed inside the crack due to rehydration of unhydrated cement, the bond strength between the crack surface and new CSH is most likely weaker compared with the tensile strength of the matrix.

3.2. Deflection capacity

The deflection (deformation) capacity is of major concern for ECC type material since its structural application mainly requires high deformation and energy dissipation capacity. As can be seen from Fig. 7, the deflection capacity for reference samples (scheme A) first increases from 14 days to 28 days and then decreases due to the continuous evolution of fiber, matrix and fiber/matrix interface properties. The optimal balance of these three aspects of ECC material leads to peak deflection capacity at 28 days. Afterwards, the increased matrix strength results in reduced deflection capac-

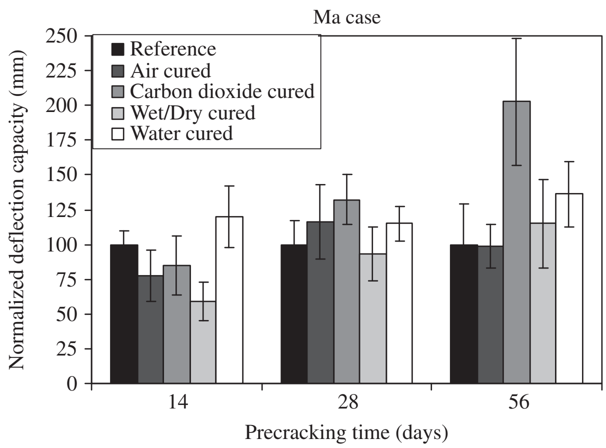

Fig. 9. Comparison of normalized deflection capacity of mixture Ma for different curing conditions and precracking time.

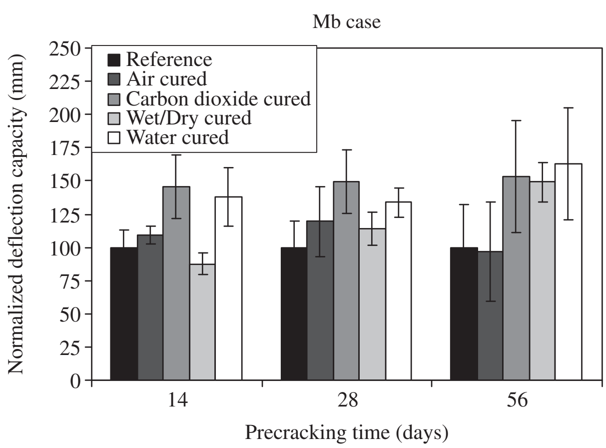

Fig. 10. Comparison of normalized deflection capacity of mixture Mb for different curing conditions and precracking time.

ity. Eventually a stable deflection capacity (ductility) is expected due to maturity of matrix toughness and fiber/matrix interface properties. This has been confirmed in long term durability study of ECC by Lepech and Li [15]. Their results indicated a stable ductility after about 1 or 2 months.

Fig. 8 shows a comparison of the typical bending stress-deflection relations under four curing conditions (air, CO_2, wet/dry and water curing) with reference for mixture Ma at precracking time of 28 days. Figs. 9 and 10 indicate under these four curing conditions almost all samples can fully recover its deflection capacity; in most cases even exceed the deflection capacity of reference case (condition A). This is very significant especially considering these samples have been pre-cracked to 1.5 mm deflection. It can be seen that that samples under air curing, CO_2 curing, cyclic wet/dry curing and water curing can reach about 80–120%, 85–200%, 60–150% and 115–160% of the deflection capacity of the reference samples. This recovery level is significantly higher than that of M2-4 in Fig. 11 (Qian et al. [5]). In addition to the water retaining effect of nanoclay, this can be further explained that the M2-4 has much less cementitious material compared with Ma and Mb. Therefore the self-healing potential of Ma and Mb due to continuous hydration is much greater than that of M2-4.

Furthermore, it should be noted that the air cured sample shows reasonable recovery with different precracking time, which seems to indicate that the water retaining capacity of nanoclay

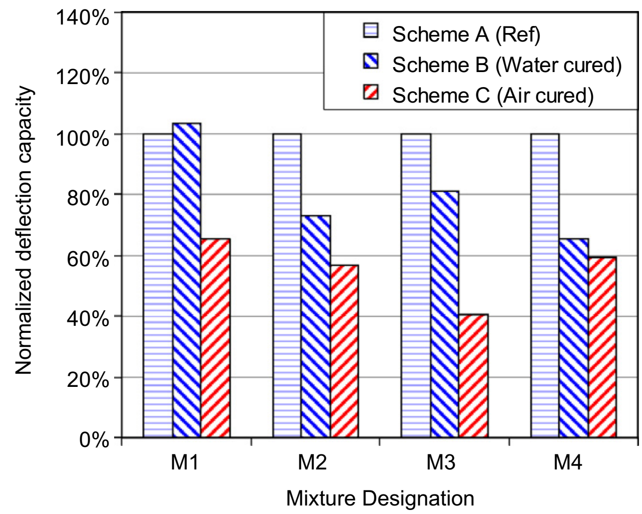

Fig. 11. Comparison of normalized deflection capacity for different mixtures with precracking time of 28 days (Qian et al. [5]).

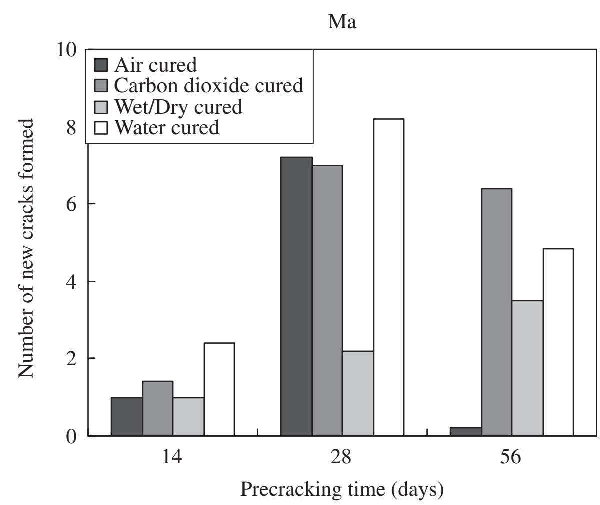

Fig. 12. Comparison of number of new cracks formed for mixture Ma with different curing conditions and precracking time.

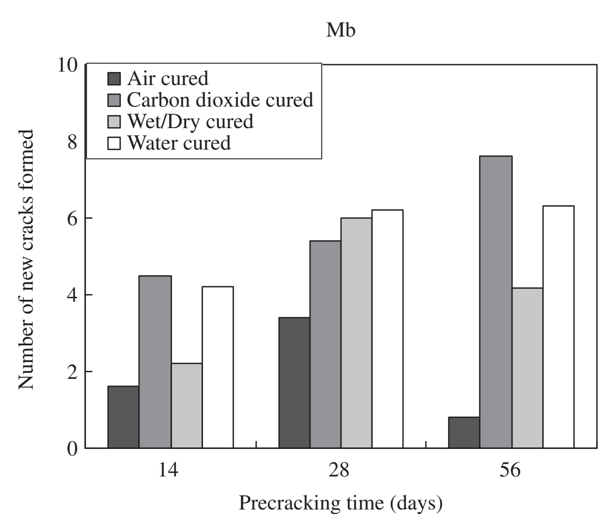

Fig. 13. Comparison of number of new cracks formed for mixture Mb with different curing conditions and precracking time.

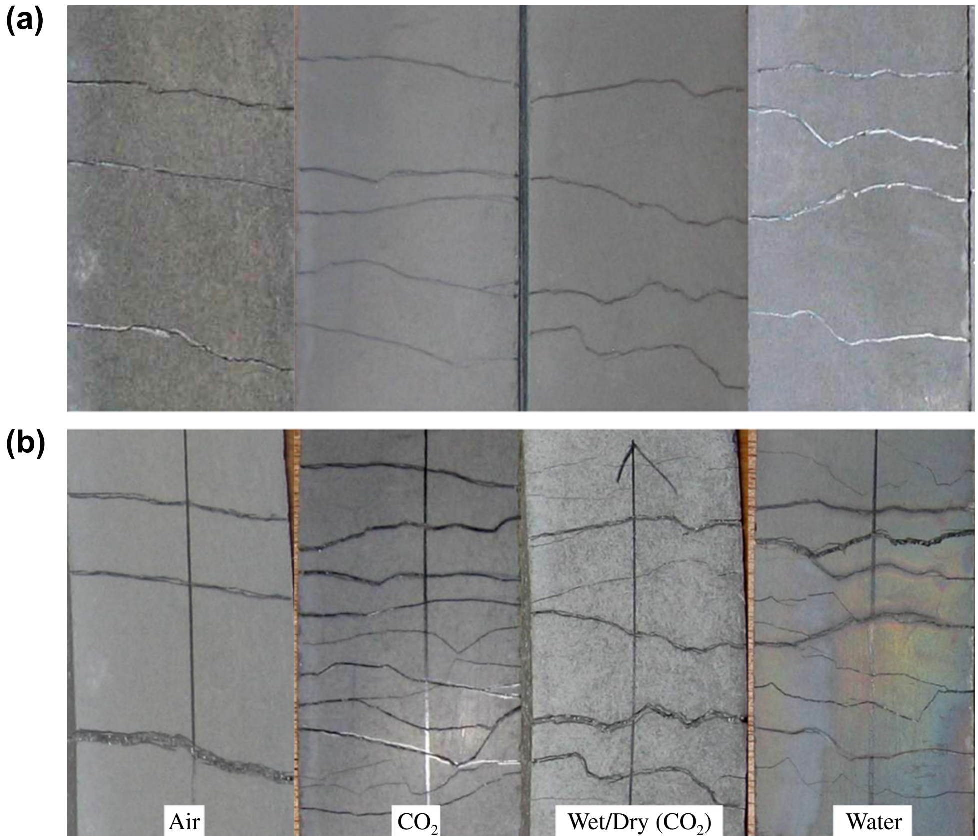

Fig. 14. Comparison of crack pattern for mixture Ma with precracking age of 56 days: (a) before curing and reloading and (b) after curing and reloading.

provides internal water supply for further hydration to occur. Valcke et al. [14] indicated that the nanoclay (the same type as used in this investigation) can absorb water of about 100% of its own weight between layered structures and requires about seven times more water to have a homogeneous water/clay mixture. The extra water is free water and can potentially be utilized for cement hydration.

As can be seen from Figs. 9 and 10, the samples of cyclic wet/dry curing (curing condition D) show lower deflection capacity compared with that of CO_2 cured and water cured (conditions C and E). In Figs. 2 and 3 and Figs. 4 and 5, it can be seen that the flexural strength under wet/dry curing (condition D) are slightly higher compared with that of CO_2 and water curing, while flexural stiffness are much greater in wet/dry curing. Flexural stiffness can be roughly correlated with flexural first cracking strength as both properties are closely related to matrix strength. Therefore, the deflection capacity in wet/dry curing is reduced probably due to the shrunk margin between flexural first cracking strength and flexural strength in wet/dry curing.

3.3. Cracking pattern

As shown in Figs. 12 and 13, the variation of number of the newly formed cracks after reloading with curing conditions generally follows the same pattern as that of the deflection capacity with curing conditions as in Figs. 9 and 10. In almost all cases, new cracks formed much less under air curing and cyclic wet/dry curing compared with the CO_2 and water curing conditions. The new cracks formed the least in mixture Ma at precracking time of 14 days, which is well correlated with the corresponding deflection capacity of the same mixture at same precracking time. Due to the unevenness of crack width of different mixture at different age, however, the number of new cracks formed cannot be directly proportioned with the deflection capacity, as evident by comparison of Figs. 9 and 10 and Figs. 12 and 13.

Crack pattern for mixture Ma and Mb is compared before and after curing and reloading with precracking age of 56 days. The pre-cracked sample was marked by pencil to reveal the number of crack formed during preloading stage, as shown representatively in Figs. 14a and 15a. The samples after curing and reloading are shown in Figs. 14b and 15b. From the comparison between these two figures, the number of new crack formed after curing and reloading can be clearly shown. From these figures it can be seen that the new crack formed the least in the air cured samples while many new cracks formed in the samples with other curing conditions (CO_2, wet/dry and water curing), contributing to the enhanced deflection capacity.

From Figs. 16 and 17, the percentage of final cracks occurring at new locations is compared for air, CO_2, wet/dry and water curing (conditions B–E) and different precracking time. The actual locations of final cracks can be easily differentiated between preexisting crack and new crack by comparing within Figs. 14 and 15. It is very consistent that there is no final crack occurring at new locations for air cured samples for all precracking time and mixtures. On one hand, the number of new cracks formed is generally much less in air cured samples compared with other curing conditions (CO_2, wet/dry and water curing) in Figs. 12 and 13. On the other hand, the low chance for air cured samples also suggests that the healing under air curing, even though with internal water supply from nanoclay, has relatively weak strengthening effect, making it difficult to shift final fracture sites from preexisting cracks to new locations.

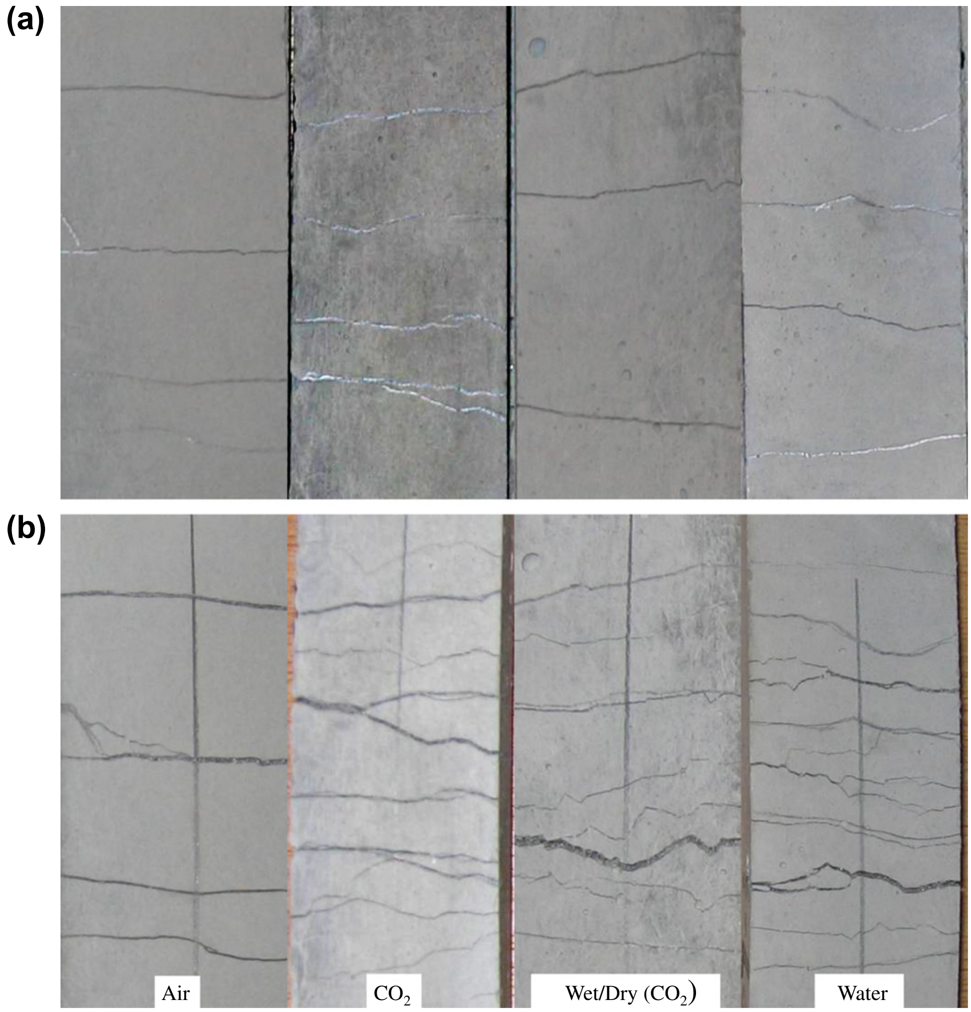

Fig. 15. Comparison of crack pattern for mixture Mb with precracking age of 56 days: (a) before curing and reloading and (b) after curing and reloading.

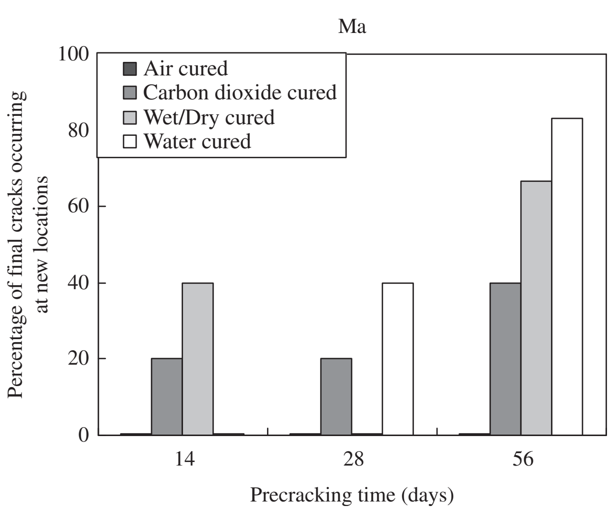

Fig. 16. Percentage of final cracks occurring at new locations for mixture Ma with different curing conditions and precracking time.

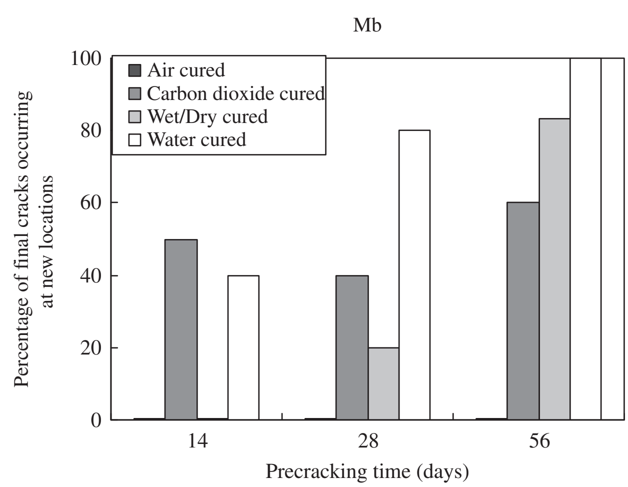

Fig. 17. Percentage of final cracks occurring at new locations for mixture Mb with different curing conditions and precracking time.

4. Conclusions

This paper investigates the influence of curing condition and precracking time on the self-healing behavior of ECC. Based on the experimental results, following conclusions can be made:

In most cases, the reloading deflection capacity for air, CO_2, wet/dry and water curing (B–E) is equal to or even exceeds that from the reference (condition A) due to the self-healing of ECC. Their recovery levels are significantly higher compared with that of the previous mixtures M2-4 by Qian et al. [5] (Fig. 11) due to the addition of nanoclay and higher cementitious material content in the mixtures (Ma and Mb compared with M2-4).

Furthermore, it should be noted that the air cured sample shows reasonable recovery of deflection capacity with different precracking time, which indicates that it may be possible to utilize the water retaining capacity of nanoclay to internally cure and heal the damage along the microcracks. The next step for study is to further demonstrate the above preliminary finding with ESEM and NMR, etc.

The level of stiffness retained under air, CO_2, wet/dry and water curing (conditions B–E) is generally comparable with experimental results of Li and Yang [4] for the precracking age of 28 days, even though the actual curing conditions may not be the same. The level of stiffness retained after these four curing conditions decreases with time of precracking for both mixtures due to the gradual exhaustion of cementitious material for continuous hydration.

It is very consistent that there is no final crack occurring at the new location for air cured samples for all precracking time and mixtures. This suggests that the healing under air curing, even though with internal water supply from nanoclay, has relatively weak strengthening effect, making it difficult to shift final fracture sites from preexisting cracks to new locations.

Acknowledgements

The authors would like to thank Dutch Ministry of Economic Affairs for their financial support for this work (IOP Self healing materials – Healing Built-in Building Materials). Furthermore, we would like to thank Kuraray Co, Japan for donating PVA fiber and Dr. Hartmut Fischer from TNO Science and Technology for providing Nanoclay and helpful suggestions for this research.

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