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Research Article

Sustainable High-Ductility Concrete with Rapid Self-Healing Characteristic by Adding Magnesium Oxide and Superabsorbent Polymer

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As a class of high-ductility concrete, engineered cementitious composites (ECC) have wide application prospects in engineering fields. However, the occurrence of cracks and the limited self-healing ability hinder the development of ECC. Rapid self-healing has important significance for ECC in reducing maintenance costs and prolonging service life, which are conducive to sustainable development of ECC. Therefore, the aim of this paper is to enhance the self-healing property of ECC by adding light-burned magnesium oxide (MgO) and superabsorbent polymer (SAP) on the premise of maintaining the high ductility. First, the effect of MgO and SAP on the ductility property of ECC which is the most important feature was explored with the uniaxial tensile test. The results indicated that MgO is helpful to the strength but not conducive to the ductility of ECC, while SAP has an opposite effect. The effects of MgO and SAP on the ductility of ECC can be balanced. Later, the permeability test, scanning electron microscopy (SEM), and X-ray diffraction (XRD) were used to evaluate the effects of MgO and SAP on the self-healing property of ECC. The results showed that the combined addition of MgO and SAP shows much better effect than the individual addition and can cut the healing time by half. Overall, it is concluded that ECC with MgO and SAP have the potential for self-healing, and the ductility can also be reconciled.

1. Introduction

Engineered cementitious composites (ECC) are a typical kind of high-ductility cementitious composites (HDCC). The ultimate tensile strain of ECC can be above 3%, which is 300 times larger than that of the ordinary cement concrete, and the crack width in a saturated state is below 100 μm [1–3]. Since the successful preparation of ECC, many scholars have carried out the engineering applications of ECC, such as highway bridge link slab, steel deck pavement, and ECC-concrete composite beam, retrofitting of structures in seismic regions [4–9]. The good mechanical properties and simple construction technology make ECC suitable for practical engineering projects. However, ECC is considerably affected by loading and environmental factors. Cracks may occur in ECC due to the action of loading and environment, which not only weaken the mechanical properties but also cause moisture damage.

The unhydrated or insufficiently hydrated materials at the damaged part of cement-based materials tend to hydrate further and produce products to heal a crack, which is known as self-healing behavior [10]. Huang et al. [11] pointed out that the early self-healing of cement-based materials is mainly realized by hydration of unhydrated cement. Lauer and Slate [12] investigated the effect of humidity on the self-healing behavior of cement-based materials and found that 95% relative humidity induces the fastest self-healing rate. Dhir et al. [13] showed that the rate of self-healing decreases with the increase of age. Fang et al. [14] concluded that Ca^{2+} distributed in cement-based materials and CO_3^{2-} dissolved in water form $CaCO_3$ crystals, which conduce to crack self-healing of cement-based materials. Edvardsen [15] found that the increasing rate of $CaCO_3$ crystals at cracks is related to crack width and water pressure. However, the material composition, age, temperature, humidity, crack width, and others have significant effects on the property of self-healing. In general, the crack width of ordinary cement-based materials is relatively large, and limited self-healing products are incapable of efficiently repairing those cracks.

ECC material is found to have more potential for self-healing than ordinary cementitious composites. Maalej et al. [16] proposed that ECC produces fine cracks under load, which is conducive to the self-healing of cracks. Kan et al. [17] pointed out that polyvinyl alcohol (PVA) fibers in ECC have hydrophilic properties, which provide nucleation points for the formation of self-healing products and help the formation and growth of self-healing products. But, for ECC entity engineering, the environmental temperature and humidity conditions are difficult to be adjusted; hence, it is of difficulty to achieve complete self-healing for ECC. The achievement of rapid self-healing for ECC in the natural environment has important theoretical and practical significance in reducing maintenance costs and prolonging service life. Many researchers have explored the self-healing properties of ECC through theoretical and experimental methods [18–25]. The common method used in the literature to accelerate the self-healing of ECC is adding the supplementary cementitious materials (SCM) [23]. But, the effects are not completely satisfactory and quite different among the studies. Therefore, it is also necessary to find a faster and better self-healing method for ECC.

Magnesium oxide (MgO) has been used as an expansion additive in mass concrete [20]. Addition of MgO into mass concrete makes the concrete possess microexpansion characteristics. The shrinkage of concrete, especially the temperature shrinkage, can be compensated and the crack resistance of concrete can be improved. The microexpansion feature of MgO is beneficial to crack closure and has great potential in the self-healing of cementitious materials. However, there are limited studies focusing on this.

Superabsorbent polymer (SAP) is used as curing additives in concrete [18]. SAP particles can provide plenty of water due to their ability to serve as a water reservoir. It has a three-dimensional cross-linked network structure, which can fix water inside the polymer network by swelling action. It has good water retention even under pressure and can release water slowly in dry air. As a novel self-healing technique, SAP can also be used in ECC as a self-healing agent to serve as internal water curing.

In this paper, light-burned MgO and SAP were added in ECC to enhance its self-healing property. In general, a strong chemical bonding between the polyvinyl alcohol (PVA) fiber and cement hydrates restricts the achievement of high ductility for ECC. The fly ash which is the waste produced during the production process of a thermal power plant can improve the interfacial behavior between the matrix and PVA fibers, and this is very helpful to the ductility [26–28]. Therefore, ECC in this paper were prepared with a high volume of fly ash. Due to that the ductility is the most important property for ECC, the effects of MgO and SAP on the ductility of ECC were first studied with the uniaxial tensile test. The self-healing mechanism of ECC incorporating MgO and SAP was investigated using the permeability test, scanning electron microscopy (SEM), and X-ray diffraction (XRD). The self-healing method for ECC proposed in this paper can improve its function, prolong its life, and reduce its maintenance cost, which is really conducive to sustainable development of ECC.

2. Materials and Experiments

2.1. Raw Materials. The raw materials used in this study were cement, fly ash, quartz sand, PVA fiber, water, water reducer, SAP, and light-burned MgO. The chemical composition of cement and fly ash is shown in Table 1. The particle size of quartz sand is 70–140 mesh. The SiO_2 content of quartz sand is 98.7%, and its density is 2.66 g/cm^3. The properties of PVA fiber are shown in Table 2.

Light-burned MgO has high activity and good volume stability. MgO used in this paper was produced by burning raw $MgCO_3$. The calcination temperature was 900°C, and the holding time was 2 h. The main properties of light-burned MgO are shown in Table 3.

SAP has a three-dimensional cross-linked network structure, and it can absorb and release water repeatedly. The main properties of SAP used in this paper are shown in Table 4.

The contents of MgO and SAP directly affect the performance of ECC. Superfluous MgO will cause excessive expansion, which may lead to the destruction of the internal structure of ECC, while a small quantity of MgO cannot satisfy the expected expansion effect. On the contrary, the suitable amount of SAP promotes the hydration of cementitious material and is conducive to the strength development of the composites, but the excessive amount of SAP produces many honeycomb-like voids after the loss of water, which affects the strength of the composites. According to the relevant researches [18–20] and the preliminary tests, the mix proportions of ECC used in this study are shown in Table 5.

The mixing procedure for ECC was as follows: (1) cement, fly ash, MgO, SAP, and silica sand were added into a blender and mixed at 100 rpm for 3 min. (2) Water and water reducer which were previously dissolved in water were added and mixed at 100 rpm for 1 min and then 400 rpm for 4 min. (3) Fibers were added slowly and manually along the stirring direction and mixed at 400 rpm for 10 min. The ECC specimens were then kept in a standard curing box at a relative humidity of (95 ± 5)% and a temperature of (20 ± 2)°C for 28 d.

2.2. Uniaxial Tensile Test. The uniaxial tensile test was conducted to evaluate the ductility performance of ECC, as shown in Figure 1. The sizes of tensile specimen were as follows: the total length was 330 mm, the gauge was 80 mm, the width was 60 mm, the narrow width was 30 mm, and the thickness was 13 mm [21]. The loading rate in the tensile test was 0.5 mm/min. The force and displacement were automatically collected by the universal testing machine.

Table 1: Chemical composition of Portland cement and fly ash.

Material	Chemical component (by weight (%))									
	SiO_2	CaO	Al_2O_3	Fe_2O_3	SO_3	P_2O_5	K_2O	Na_2O	TiO_2	MgO
Cement	21.26	57.82	7.67	2.88	4.04	5.26	0.78	—	0.21	—
Fly ash	56.18	2.82	31.46	3.85	0.69	0.91	—	1.32	—	1.33

Table 2: Physical and mechanical properties of the PAV fiber.

Diameter (μm)	Length (mm)	Elongation (%)	Density (g/cm^3)	Elastic modulus (GPa)	Tensile strength (MPa)
35	12	7.3	1.3	31.3	1287

Table 3: Properties of MgO.

MgO content (%)	f-CaO content (%)	Loss of ignition (%)	Water content (%)	Activity degree (s)
≥85.0	≤2.0	≤4.0	≤1.0	50–300

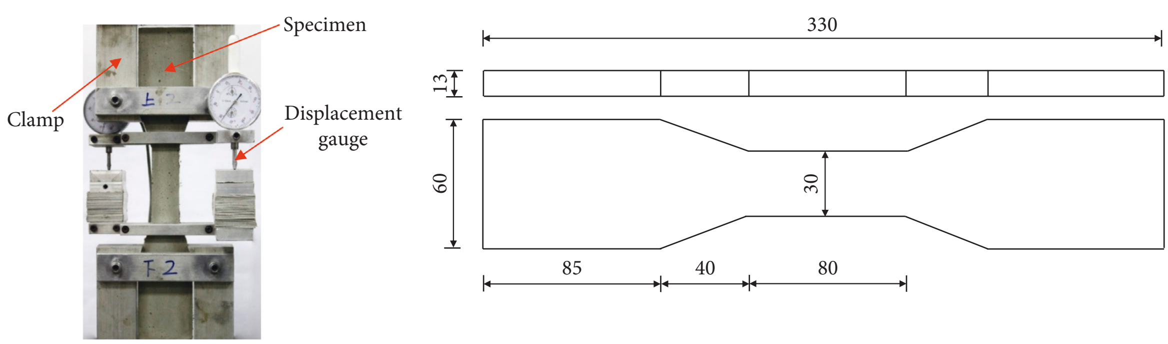
Table 4: Properties of SAP.

Particle size (mesh size)	Density (g/cm^3)	Water absorption (g/g)	Water absorption speed (s)
30–80	0.8	250–600	30

Table 5: Mix proportions (weight ratio).

Group	Cement	Fly ash	Water	Sand	Fiber[1] (%)	MgO[2] (%)	SAP[2]	Water reducer[2]
1#	1.00	3.00	1.20	1.44	2.00	—	—	0.02
2#	1.00	3.00	1.20	1.44	2.00	6.00	—	0.02
3#	1.00	3.00	1.20	1.44	2.00	—	0.40	0.02
4#	1.00	3.00	1.20	1.44	2.00	6.00	0.40	0.02

[1]Fiber content is the volume ratio to composites. [2]Contents of MgO and SAP, and water reducer is the mass ratio to cement and fly ash.



(a) (b)

Figure 1: Uniaxial tensile test: (a) test setup; (b) dimensions of the specimen (unit: mm).

2.3. Permeability Test. ECC have effective self-healing behavior in the maintenance environment of dry-wet cycles [22, 23]. Therefore, the self-healing of ECC in this study is achieved by dry-wet cycles. A dry-wet cycle includes immersion in water for 24 h and then in air for 24 h. The water permeability of ECC will decrease as the cracks heal. Therefore, the permeability test can be used to evaluation the self-healing of ECC [24, 25]. The relative seepage ratio (RSR) is defined as an evaluation index for self-healing and can be calculated with equation (1). The smaller the value of RSR is, the higher the degree of self-healing of ECC is.

$$\text{Relative seepage ratio (RSR)} = \frac{V_N}{V_{\text{original}}}, \quad (1)$$

where V_N is the seepage volume after N dry-wet cycles and V_{original} is the seepage volume before dry-wet cycles.

The test process was as follows: (1) the specimens with a diameter of 100 mm and a height of 20 mm were prepared and preloaded by splitting to produce microcracks [24]; (2) the permeability test was conducted with a water pressure of 0.2 MPa for 60 min. The devices of permeability test are shown in Figure 2.

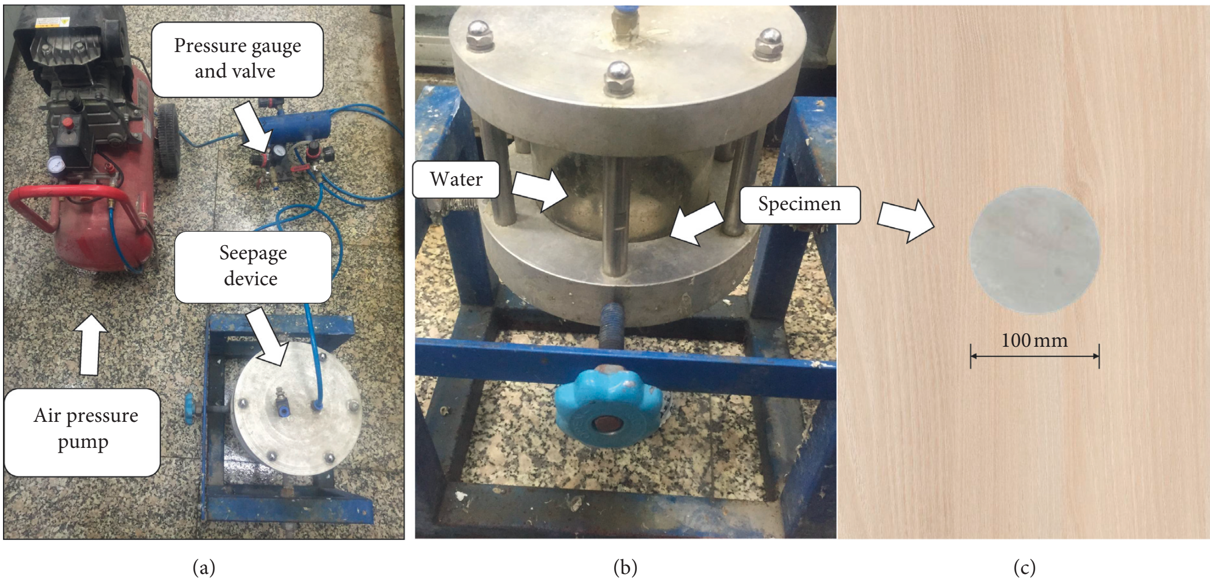


Figure 2: Permeability test: (a) test system; (b) seepage device; (c) specimen.

2.4. SEM and XRD. SEM is often used to observe the micromorphology. In this study, the cracks of ECC were observed with SEM, as shown in Figure 3. Due to the poor conductivity of the cement-based materials, the test specimens were gold-plated before observation. In addition, the energy dispersive spectrometer (EDS) was used to analyze the types and contents of elements near the crack.

In addition, XRD was used to conduct the phase analysis of self-healing products. The measurements were conducted using dried powders [29]. The specimens and the equipment used for XRD in this study are shown in Figure 4.

3. Results and Discussion

3.1. Effects of MgO and SAP on the Ductility of ECC. High ductility is the most important characteristic of ECC. Therefore, the ductility cannot be reduced significantly while the self-healing property of ECC is improved. To evaluate the effects of MgO and SAP on the ductility performance of ECC, the uniaxial tensile test was carried out. The typical tensile stress-strain curves for ECC with different mix proportions are shown in Figure 5. As can be seen from the figure, MgO and SAP do have an impact on the ductility of ECC. The order for ECC with different mix proportions based on ductility was ECC containing SAP (specimen 3), ordinary ECC (specimen 1), ECC containing MgO and SAP (specimen 4), and ECC containing MgO (specimen 2).

To facilitate quantitative analysis, the initial cracking strength, ultimate tensile strength, and ultimate tensile strain of ECC are summarized and presented in Figure 6.

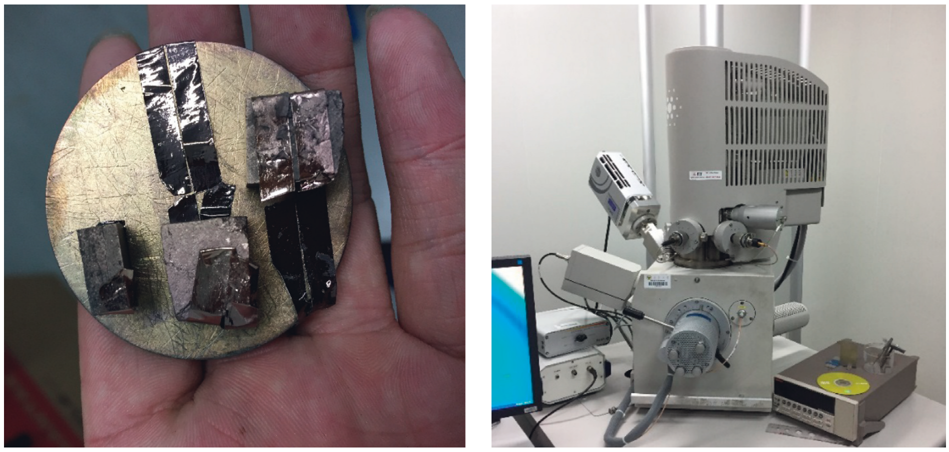
The initial cracking strength for ordinary ECC (specimen 1), ECC containing MgO (specimen 2), ECC containing SAP (specimen 3), and ECC containing MgO and SAP (specimen 4) is 2.38 MPa, 2.58 MPa, 2.23 MPa, 2.43 MPa, respectively. Also, the ultimate tensile strain for ordinary ECC (specimen 1), ECC containing MgO (specimen 2), ECC containing SAP (specimen 3), and ECC containing MgO and SAP (specimen 4) is 2.90%, 2.53%, 3.22%, 2.75%, respectively. The above data indicate that the MgO increases the strength of ECC while SAP decreases the strength of ECC. But, for the ductility of ECC, MgO reduces the ductility while SAP improves the ductility.

These above changes are directly related to the changes of MgO and SAP. The hydration of MgO produces $Mg(OH)_2$, as shown in Figure 7. The formation and growth of $Mg(OH)_2$ crystals will cause expansion and make ECC denser, which is helpful to the strength but not conducive to the ductility. Thus, ECC containing MgO (specimen 2) has larger strength and smaller deformation than ordinary ECC (specimen 1).

For the SAP, it is not active and cannot hydrate. SAP is added during batching and will initially swell and form gel-like structure. The initial swelling is also confined by the mixing and compaction processes. But, when SAP releases water, it will shrink and the honeycomb-like voids are left in the materials, as shown in Figure 8. These voids can be viewed as internal defects of ECC which has correlation with the strength and ductility of ECC [30]. The test results indicated that SAP is helpful to the ductility but leads to decrease in the strength of ECC.

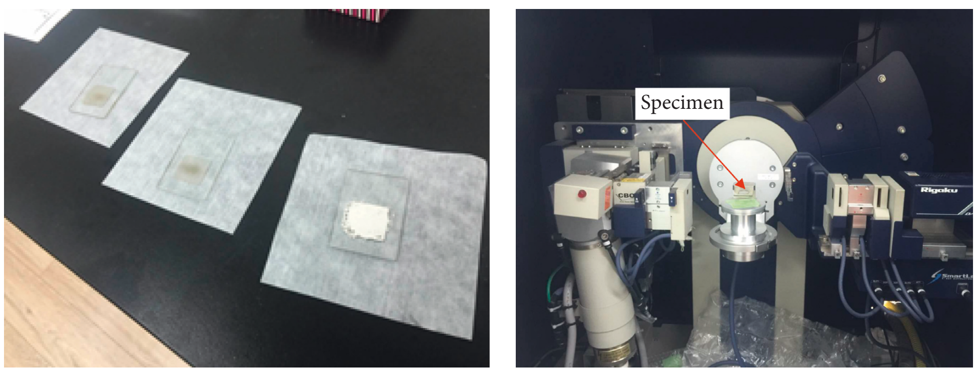
Taken together, the effects of MgO and SAP used in this study on the ductility property of ECC are balanced. The ECC prepared with MgO and SAP shows obvious ductility.

3.2. Effects of MgO and SAP on the Self-Healing Property of ECC. The changes of RSR with the number of dry-wet cycles are shown in Figure 9. As can be seen from the figure, the water permeability decreases with the number of curing cycles, initially at a rapid rate and gradually stabilized, which indicated that the self-healing of ECC with different mix proportions does occur. The control ECC specimens without MgO and SAP (specimen 1) need 10–12 cycles to complete



(a) (b)

Figure 3: SEM: (a) specimen after gold plating; (b) equipment.



(a) (b)

Figure 4: XRD: (a) specimen preparation; (b) equipment.

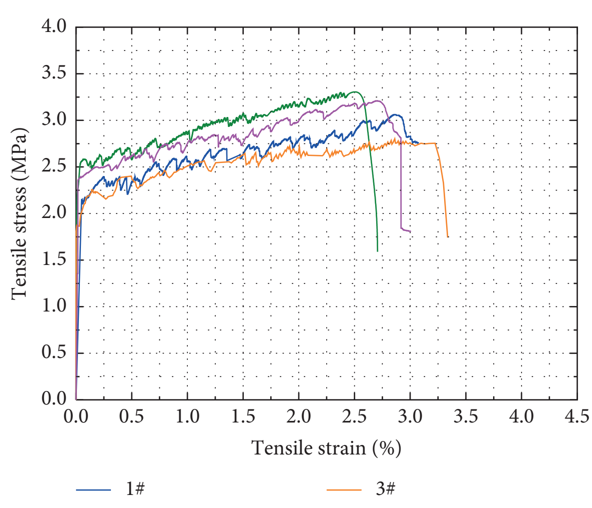


Figure 5: Typical stress-strain curves for ECC with different mix proportions.

the self-healing process; the self-healing of ECC specimens containing MgO (specimen 2) lasts 8-9 cycles; ECC specimens incorporating SAP (specimen 3) needs 7–9 cycles; ECC specimens incorporating MgO and SAP (specimen 4) only need 5-6 cycles to complete the self-healing. Therefore, it can be concluded that the individual addition of MgO or

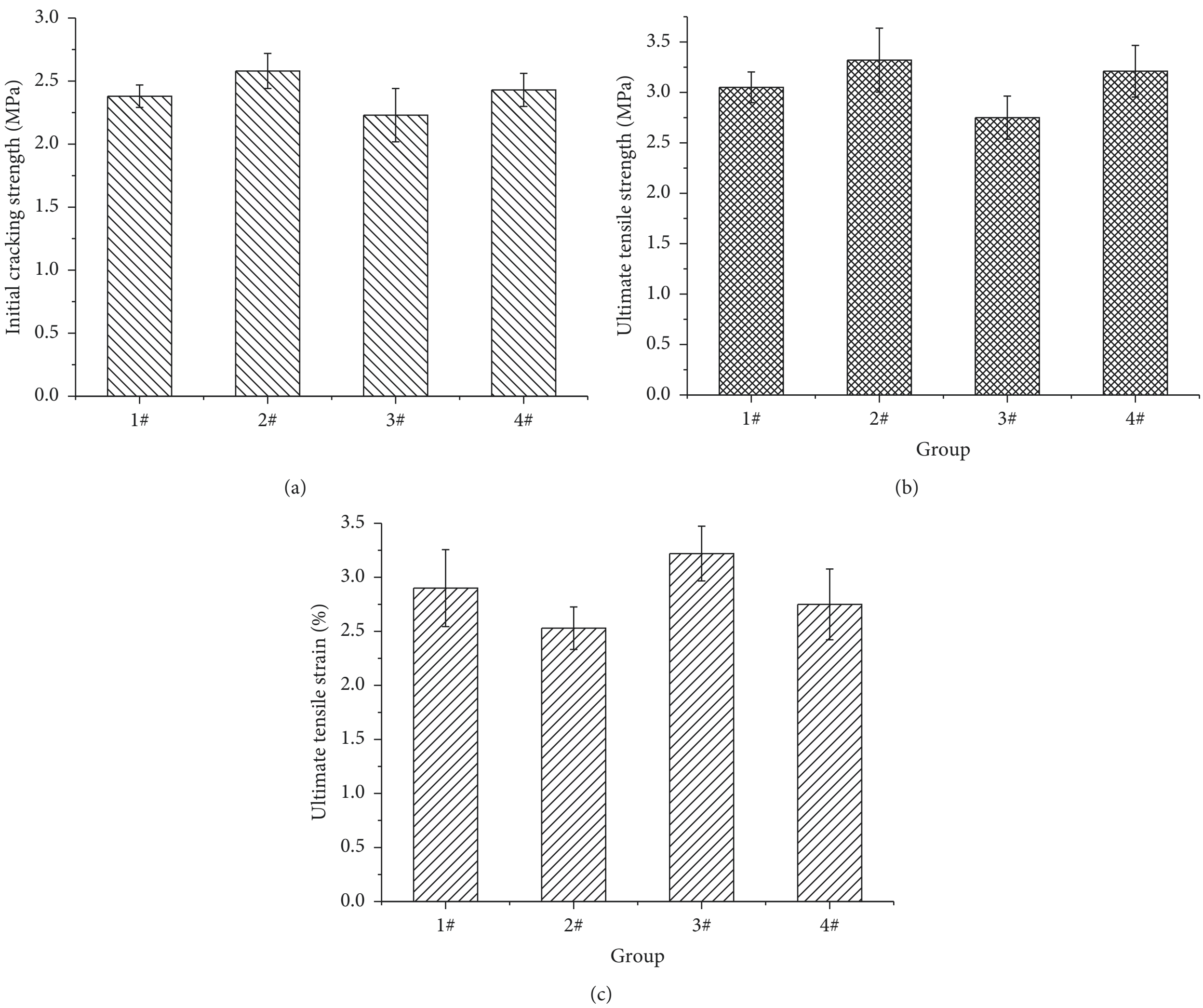


Figure 6: Results of uniaxial tensile test: (a) initial cracking strength; (b) ultimate tensile strength; (c) ultimate tensile strain.

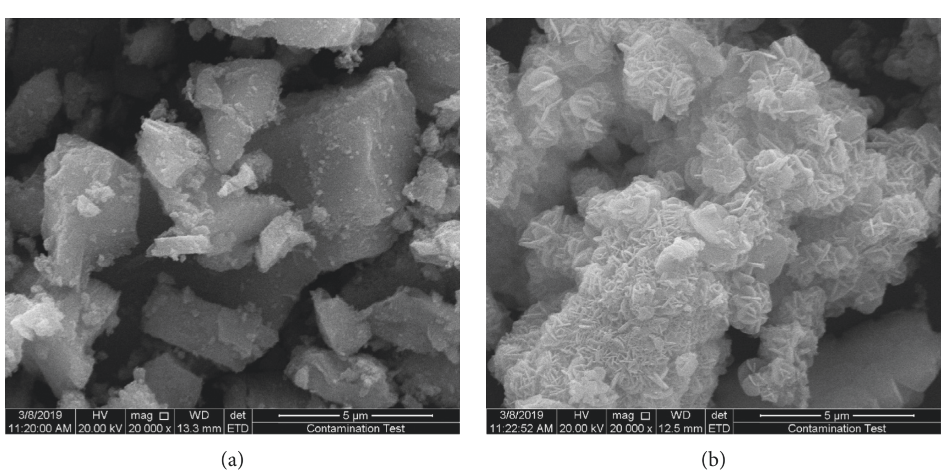


Figure 7: SEM images of MgO and its hydration products: (a) MgO; (b) hydration products.

SAP is conductive to the self-healing process. The self-healing of ECC can be improved significantly under the joint action of MgO and SAP.

Magnesium hydroxide ($Mg(OH)_2$) which is the hydration product of MgO can cause microexpansion during its hydration process [19, 20]. Both sides of the cracks will be

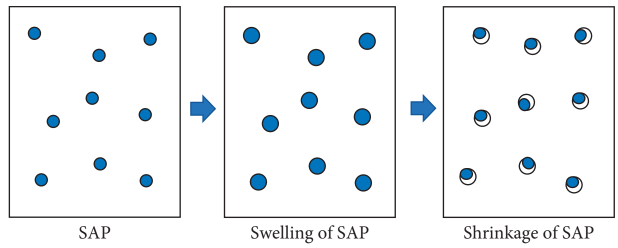


Figure 8: Schematic of the swelling and shrinkage of SAP.

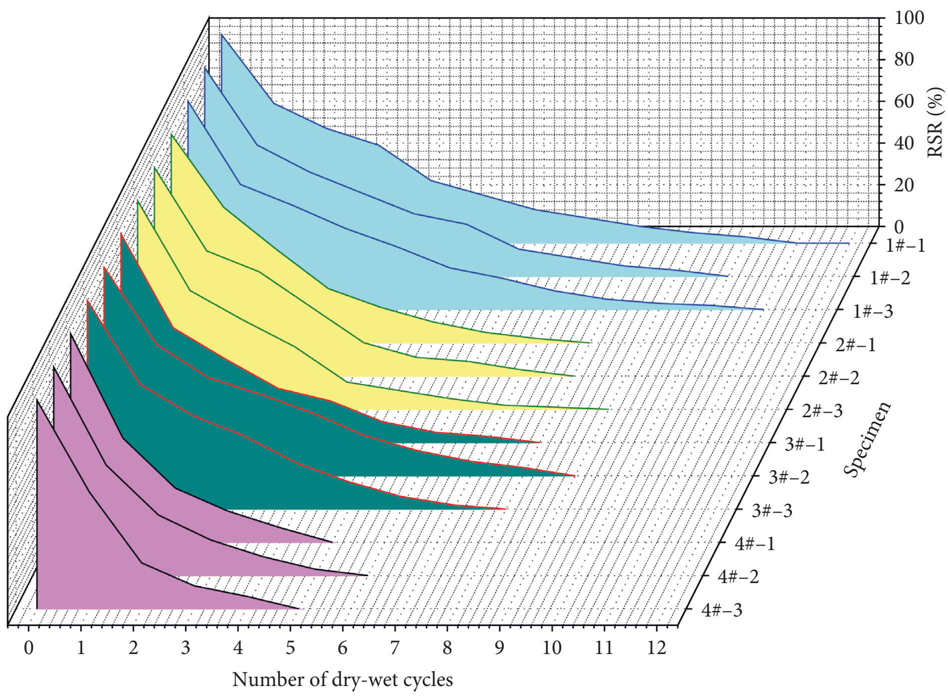


Figure 9: Variation of seepage with dry-wet cycles.

squeezed because of the expansion action, and the cracks tend to be closed. The hydration product $Mg(OH)_2$ can also be the filler in the cracks. On the contrary, SAP can store a large amount of water in the form of gel and then slowly releases [18, 22]. Therefore, under dry conditions, SAP can provide water needed for hydration of MgO and other cementitious materials and accelerate the formation of hydration products. Under the joint action of MgO and SAP, the self-healing of ECC will be faster and more effective. The mechanism is illustrated in Figure 10.

Besides, it also can be concluded from the permeability test that both actions of MgO and SAP can accelerate the self-healing process of ECC, but individual addition of MgO or SAP cannot realize the fastest healing speed. The combined addition of MgO and SAP at the same time can cut the healing time by half, which is much better than the individual addition of MgO or SAP. Therefore, the combination of MgO and SAP is recommended.

3.3. Self-Healing Mechanism of ECC Incorporating MgO and SAP. Figure 11 shows the self-healing images of the ECC specimen incorporating MgO and SAP. It can be seen from the figure that the cracks were filled with pale self-healing products.

To microscopically observe and analyze healing products in the cracks, SEM and EDS tests were conducted on self-healed ECC. The SEM images in Figure 12 also showed that the cracks in ECC were filled up after dry-wet cycles.

From a microscopic perspective, there are multiple chemical reactions in the self-healing process of ECC incorporating MgO and SAP, and they can be classified as the following: (1) continuous hydration of cementitious materials to form C-S-H; (2) formation of $CaCO_3$; (3) hydration of MgO.

The clinkers in the cement such as $3CaO{\cdot}SiO_2(C_3S)$, $2CaO{\cdot}SiO_2(C_2S)$, $3CaO{\cdot}Al_2O_3(C_3A)$, and $4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3(C_4AF)$ hydrate as expressed in equations (2) and (3). Active SiO_2 and Al_2O_3 in fly ash react with calcium hydroxide produced by clinker hydration to form cementitious material, which is called pozzolanic reaction, as shown in equations (4) and (5). At the same time, Ca^{2+} combines with CO_3^{2-} dissolved in water and forms $CaCO_3$ crystals, as shown in equations (6). Hydration reaction of MgO shown in equation (7) provides with expansion action.

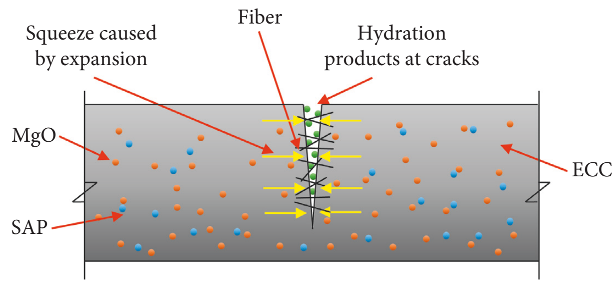
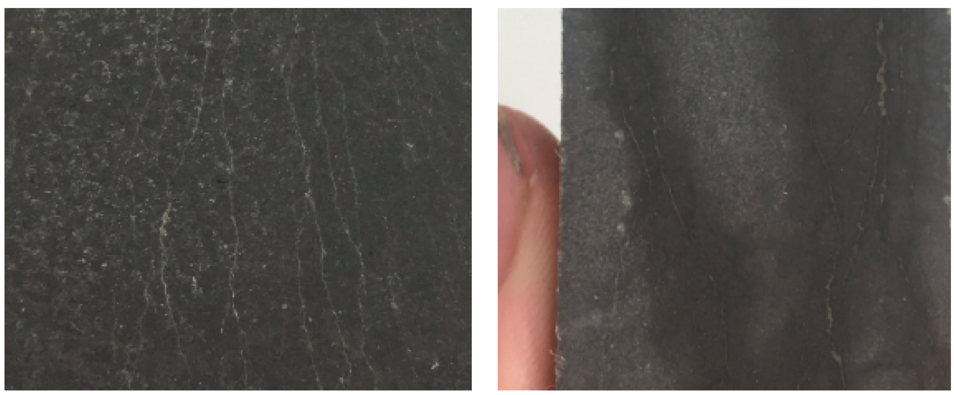
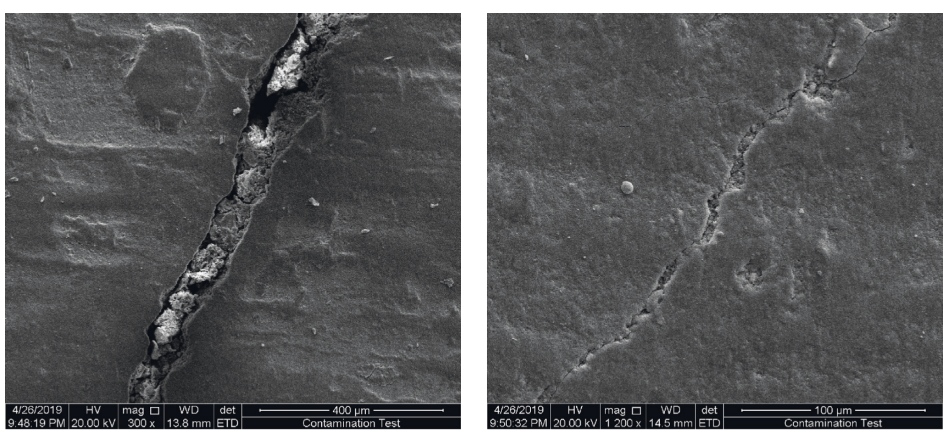


Figure 10: Typical load-displacement curves of shear test under different freeze-thaw cycles.



(a) (b)

Figure 11: Macroimages of self-healing.



(a) (b)

Figure 12: Microimages of self-healing.

$$3CaO \cdot SiO_2 + nH_2O = xCaO.SiO^2 \cdot yH_2O + (3-x)Ca(OH)_2 \quad (2)$$

$$2CaO \cdot SiO_2 + nH_2O = xCaO.SiO_2 \cdot yH_2O + (2-x)Ca(OH)_2 \quad (3)$$

$$xCa(OH)_2 + SiO_2 + m_1H_2O = xCaO \cdot SiO_2 \cdot n_1H_2O \quad (4)$$

$$xCa(OH)_2 + Al_2O_3 + m_2H_2O = yCaO \cdot SiO_2 \cdot n_2H_2O \quad (5)$$

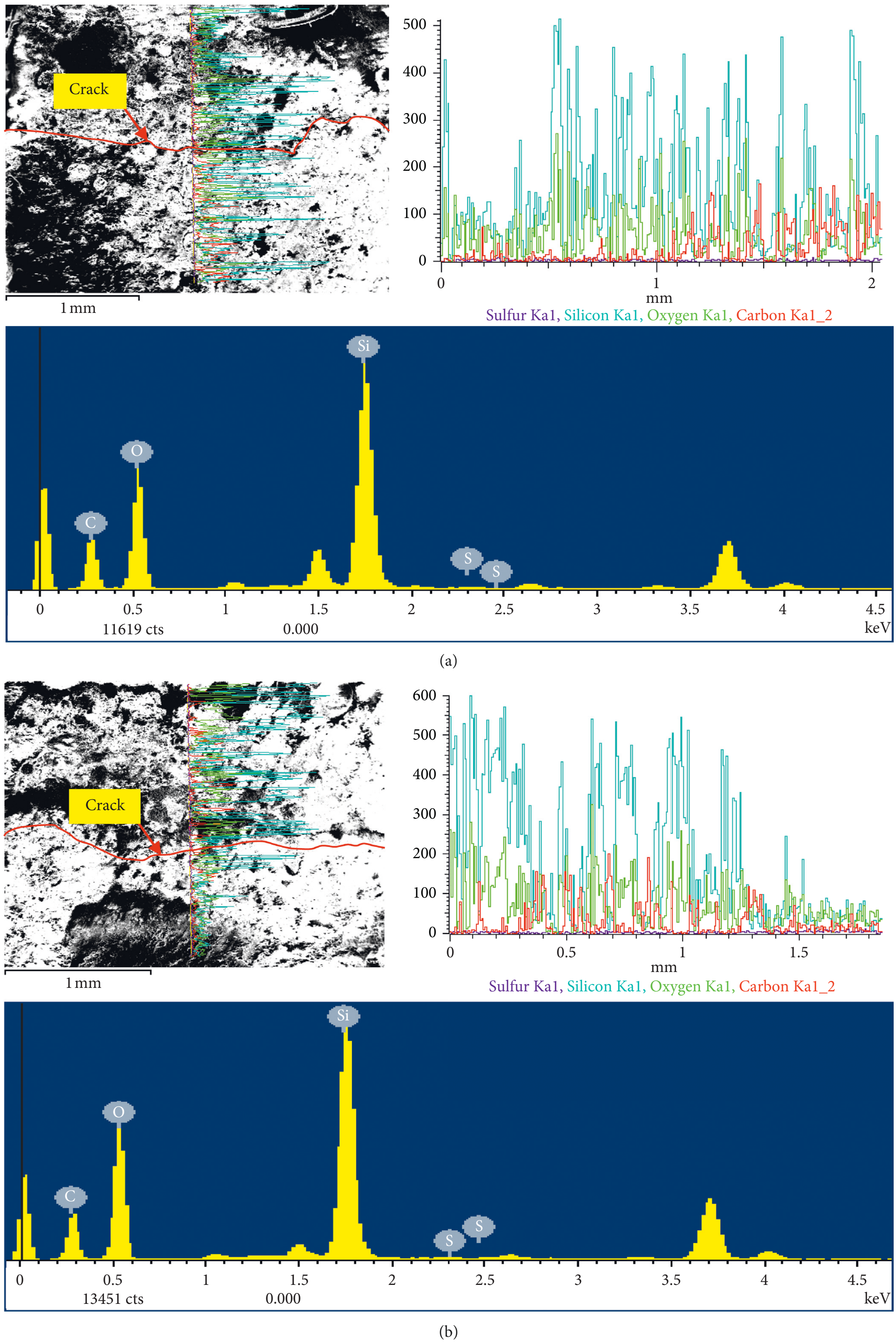
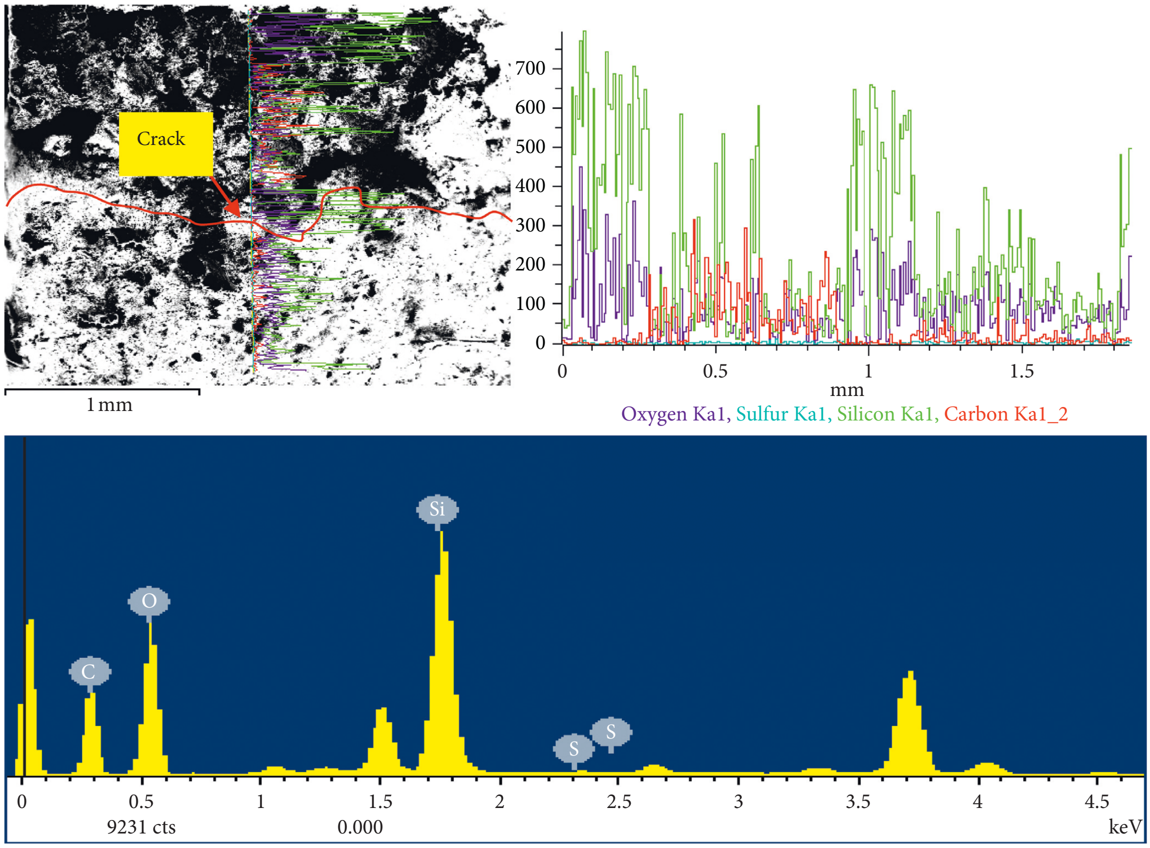


Figure 13: Continued.



(c)

Figure 13: EDS analysis for ECC incorporating MgO and SAP.

$$Ca^{2+} + CO_3^{2-} = CaCO_3 \tag{6}$$

$$MgO + H_2O = Mg(OH)_2 \tag{7}$$

In addition, under dry conditions, water needed for the hydration of cementitious materials and MgO can be provided by SAP. Further hydration will continue under dry conditions. All the above reactions and actions work together for ECC incorporating MgO and SAP and lead to the acceleration of self-healing.

To better understand the mechanism of sealing-healing, it is necessary to analyze the self-healing products. Thus, chemical analysis methods EDS and XRD are used in this study.

First, a straight-line EDS scanning perpendicular to the crack was conducted to detect the changes of main element composition after self-healing. The results along the line crossing the crack for ECC incorporating MgO and SAP are shown in Figures 13(a)–13(c), which are from different specimens. As can be seen from the figure, after the self-healing, cracks are closed, and main chemical composition remains consistent with the normal materials.

To analyze the chemical compositions of self-healing products, three different dots in the healed crack were analyzed by EDS for each specimen. Table 6 shows the proportions of the main elements of self-healing products in the self-healed crack of ECC specimens. Data for ECC before self-healing were obtained from the specimen surface near the crack, therefore reflecting the element composition of the original specimen. Based on the table, the proportions of C, which is an element of $CaCO_3$, and Si, which forms C-S-H, change significantly after self-healing cycles. The increase in the proportion of C can be explained that the contents of $CaCO_3$ increase in the healed products of cracks [18, 24]. But, the change in the amount of C-S-H is difficult to judge only based on the element changes.

To further clarify the hydration products, XRD was used to analyze the self-healing products at the cracks of ECC. The results of XRD for ECC incorporating MgO and SAP are shown in Figures 14(a) and 14(b), which are two typical types of results. As can be seen form Figure 14(a), the hydration products and calcium carbonate crystals are the main fillers of cracks. In addition, due to that a large amount of fly ash is added in ECC in this study and the hydration of fly ash is slow in the early stage, more hydration products are produced in the later stage and become the self-healing products, so sometimes, XRD results (Figure 14(b)) show C-S-H domains in the self-healing products.

The formation of hydration products is not limited to the early stage because of the addition of fly ash. Also, the literature revealed that the hydration of light-burnt MgO

Table 6: EDS analysis results of self-healing products.

Self-healing	Element (atom percentage (%))			
	C	O	Si	S
	31.92	59.69	8.32	0.06
Before	30.76	58.52	10.65	0.06
	34.88	54.65	10.45	0.02
	44.90	54.14	0.92	0.04
After	42.47	56.51	0.97	0.05
	44.26	54.81	0.91	0.02

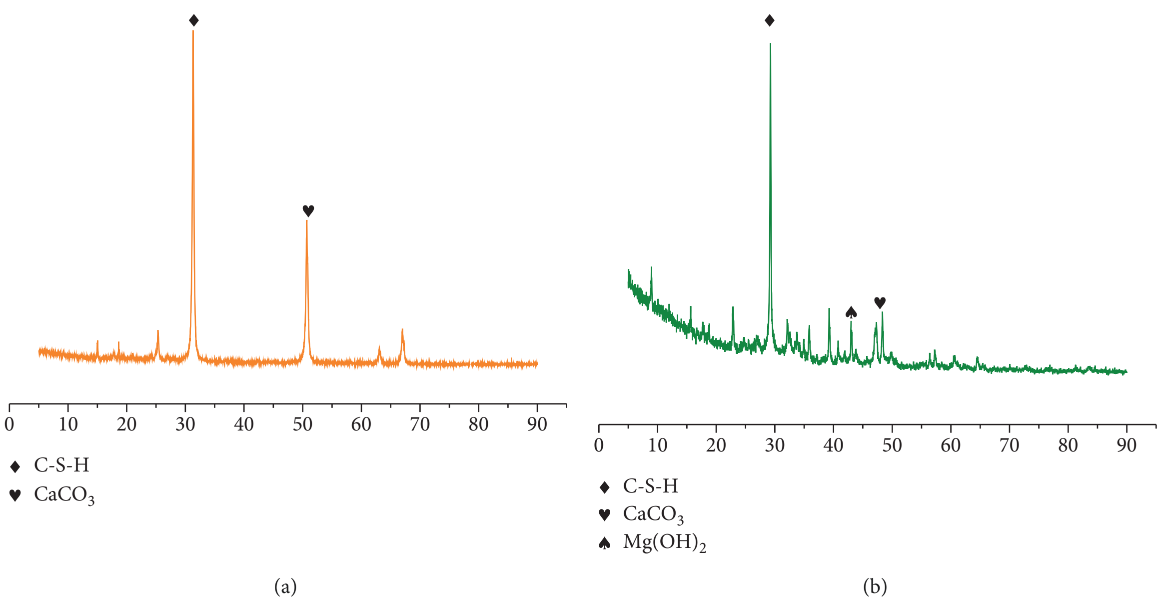


Figure 14: XRD analysis for self-healing products of ECC incorporating MgO and SAP: (a) Major products composed of C-S-H and $CaCO_3$; (b) major products composed of C-S-H.

could provide long-term expansive effect after concrete is hardened (ranging from 7 to 1000 days) [20]. Therefore, the ECC-MgO-SAP system proposed in this study can be considered to be continuous, sustainable, and durable in terms of self-healing.

4. Conclusions

In this study, light-burned MgO and SAP were added in ECC to enhance self-healing properties of ECC. The effects of MgO and SAP on the ductility performance of ECC were explored with uniaxial tensile test. The permeability test, SEM, and XRD were used to investigate the self-healing properties of ECC. Based on the obtained results, the following conclusions can be drawn:

(1) The hydration of MgO causes expansion and makes ECC denser, which is helpful to the strength but not conducive to the ductility. SAP cannot hydrate and will produce honeycomb-like voids after release of water. Thus, SAP is helpful to the ductility but leads to decrease in the strength of ECC. The ultimate tensile strain of ECC containing MgO and SAP prepared in this paper can be as high as 2.75%. The ductility of ECC can be guaranteed by adjusting the ratios of MgO and SAP.

(2) The expansion induced by the hydration of MgO makes the cracks close, while SAP provides water for further hydration even under dry conditions. The two actions can both accelerate the self-healing process of ECC. The combined addition of MgO and SAP can reduce the healing time by about 50%.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare no conflicts of interest.

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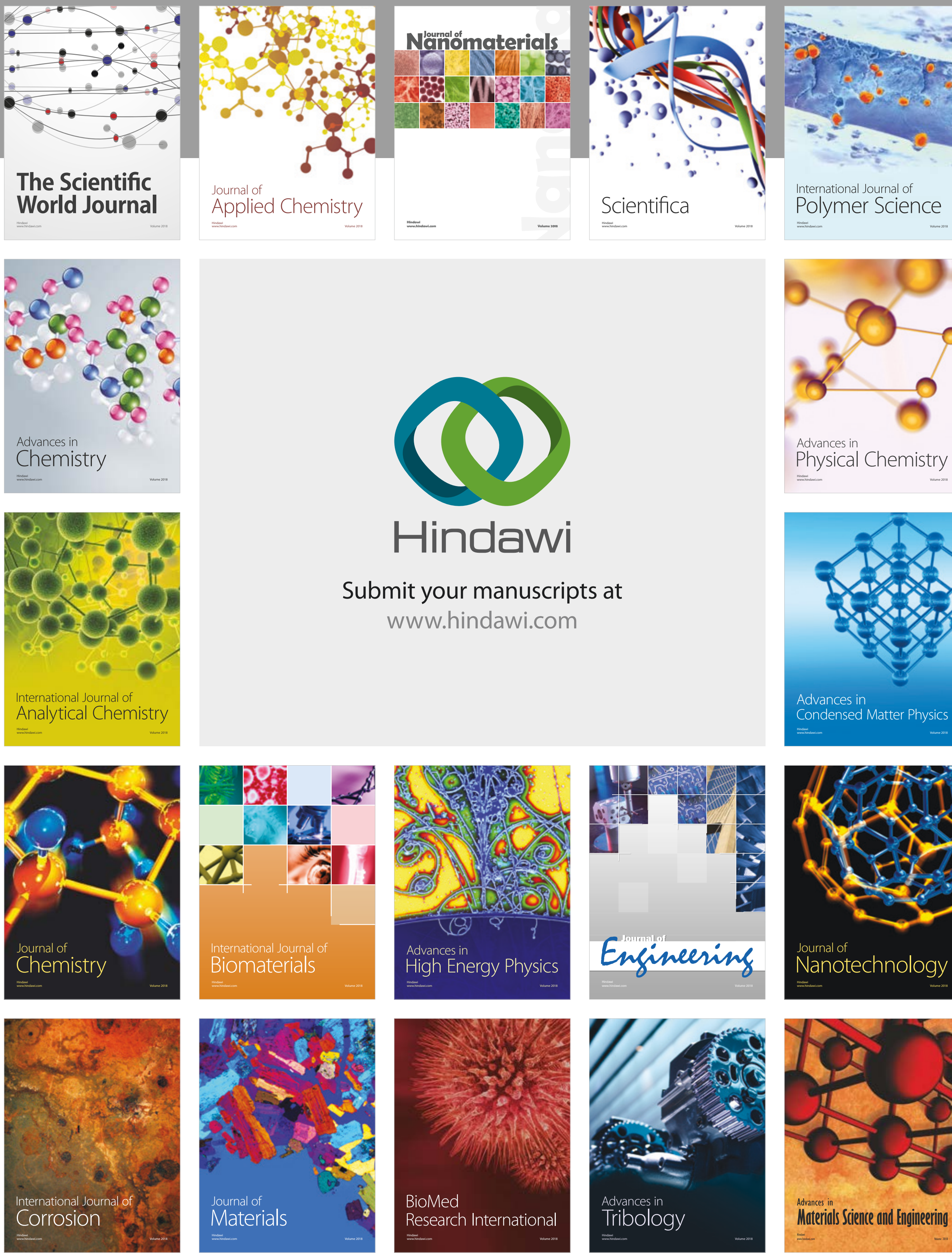
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