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SELF-HEALING OF ENGINEERED CEMENTITIOUS COMPOSITE WITH CRYSTALLINE ADMIXTURE UNDER DIFFERENT EXPOSURE CONDITIONS

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Abstract: Self-healing mechanisms in concrete can promote healing of cracks with the material produced through the concrete itself. Engineered Cementitious Composites (ECC) is a special category of High-Performance Fiber Reinforced Cementitious Composites (HPFRCC) that has been extensively investigated for self-healing purposes. Hydration of unhydrated binders in the matrix and precipitation of calcite were recognized as the two main mechanisms to cause self-healing in ECC. In addition, the high portions of fly ash and Polyvinyl Alcohol (PVA) fibers in the mixture improves the mechanism; however, small crack widths were reported to be healed in this material. The current experimental program aims at determining the extent of self-healing in large cracks by providing favorable conditions. Crystalline Admixture (CA) is also used as a self-healing promoter to increase the crack filling capacity. Three different exposure conditions of air dry, tap water, and sea water were tested using a water permeability (WP) test. The results indicate high sealing capacities in a short period for specimens made with CA and submerged in sea water.

INTRODUCTION

Durability of concrete is generally regarded as its resistance towards a particular environment to which it will be exposed during its service life. Durability issues caused by the environment such as freezing and thawing, sulfate attacks, carbonation, and steel corrosion are able to be controlled due to the fact that they share a common ancestor, i.e., the cracks (Taylor et al. 2013). Open cracks can facilitate the ingress of harmful substances such as chlorides (Cl^-) that destroy the protective layer on the steel reinforcements, leading to steel oxidation and subsequently, structural damage (Marsavina et al. 2009). Currently, repairing conducted regularly after the construction of a concrete structure considers to be a practical method, however, there are many demerits for that. The manual repair demands high budget and labor work, and sometimes requires pausing the service which adds indirectly to the life cycle cost (Gardner et al. 2018; Das et al. 2019). In some cases, the process may become difficult or even impossible due to the location of cracks, e.g., cracks in underground concrete structures. Moreover, even though manual repair techniques have considerably improved through recent years, most of these repairs can only last ten to fifteen years (Wang et al. 2019; Li et al. 2018).

On the other hand, self-healing mechanisms in concrete have been under investigation over the last two decades, trying to replace the current repairing systems with employing a more modern mechanism capable of filling the cracks with the products coming from the concrete itself (Van Tittelboom and De Belie 2013; Huang et al. 2016; Ferrara et al. 2018; Wiktor and Jonkers 2011; Ahn and Kishi 2010). Autogenous and Autonomous mechanisms are the two major methods of self-healing recognized in cementitious material.

Autogenous healing is an inherent property of concrete that existed from the first time man used Portland cement in concrete, and it was first observed by the French Academy of Science in 1836 (212 ; De Rooij et al. 2013). The mechanism mainly relies on the hydration of unhydrated cement (binder) grains existing inside the cracks to produce further calcium silicate hydrate (C-S-H) or portlandite ($Ca(OH)_2$), and the crystallization of calcium carbonate ($CaCO_3$) which is a result of the reaction between calcium ions leaching from cracks and dissolved carbonate ions in the surrounding environment. Although, the formation of these healing products is highly dependent on the properties of the surrounding environment such as presence of moisture, pH and temperature levels, and ion concentration (De Belie et al. 2018; Ma, Qian, and Zhang 2014; Xue et al. ; Yıldırım et al. 2018). In particular, sea-water exposure has reported promising results of autogenous self-healing in cementitious materials. While the extend of healed cracks in water exposure condition is about 100-150 μm (Mahmoodi and Sadeghian 2019), the healed crack width for sea water exposure is ranging between 200-700 μm (Reinhardt and Jooss 2003; Palin, Jonkers, and Wiktor 2016; Ehsan Khan, Shen, and Dias-da-Costa 2020). This is due to the presence of magnesium (Mg^{2+}) and sulphate (${SO_4}^{2-}$) ions in sea water that can react with hydroxyl (OH^-) and calcium ions (Ca^{2+}) inside cracks and in a favorable condition, precipitate brucite and ettringite (Mehta 1980; De Weerdt, Justnes, and Geiker 2014).

As described, autogenous self-healing mechanisms are majorly functions of the chemical reaction between material in the surrounding environment and material present in the concrete. By replacing cement with Supplementary Cementitious Materials (SCM), a group of silicate and calcium-rich waste products, the chances of autogenous self-healing increases (Skibsted and Snellings 2019; Sahmaran, Yildirim, and Erdem 2013). SCMs such as fly ash and steel slag provide latent hydraulic reaction, in which silica and calcium react chemically in an aqueous alkaline environment to form portlandite or C-S-H (Holland, Kurtis, and Kahn 2016). These chemical reactions of SCMs usually proceed slower than the reaction of the cement phase; therefore, they can provide late hydration, which favors autogenous self-healing (Lothenbach, Scrivener, and Hooton 2011). Also, they provide additional hydroxyl (OH^-) and calcium ions (Ca^{2+}) to help with precipitation of calcite, brucite and ettringite in a favorable environment (Sahmaran, Yildirim, and Erdem 2013; Danner, Hjorth Jakobsen, and Geiker 2019).

One very popular category of cementitious composites in this regard is Engineered Cementitious Composites (ECC) which include high portions of fly ash in its mix design (Li 1993). The micromechanics of ECC demand for dense fraction of micro-fibers which limit the crack widths and provide nucleation sites for healing products, both nourishing the autogenous self-healing process (Herbert and Li 2013). However, studies regarding self-healing in this case are limited to relatively small crack sizes (<200μm) (Yildirim et al. 2015; Özbay et al. 2013; Sahmaran et al. 2015). In addition to SCMs, some other mineral and chemical admixtures are reported to be effective in terms of crack healing (Sisomphon, Copuroglu, and Koenders 2012). Crystalline Admixture (CA) is a group of hydrophilic admixtures containing active chemicals that form water-insoluble pore-blocking precipitates upon reaction with water. This characteristic of CA helps with the densification of C-S-H to resist water and gas penetration (reduce concrete's permeability), also the unreacted portion of this material will fill the cracks through late crystallization (Ferrara, Krelani, and Moretti 2016). When mixed with concrete at 1-4% of cement weight, it has proven to improve the self-healing capability considerably (Roig-Flores et al. 2015).

The current research is trying to measure the capacity of self-healing in ECC concrete by adding a crystalline admixture. Three different exposure conditions of sea water, tap water, and air dry are adopted as testing exposure conditions for specimens during curing periods. Since the previous studies have reported relatively high values of crack healing for specimens in sea water, in this study large crack deformations are generated to investigate the extent of self-healing when using SCMs, CA, and fibers together. To measure the self-healing efficiency through recovery of transport properties which is directly related to durability, Water Permeability (WP) test was carried out on disk specimens. Visual observations are also reported to conclude the results of the water permeability test.

1 EXPERIMENTAL PROGRAM

1.1 Material Properties

Two groups of ECC (the reference) and ECC-CA (with Crystalline Admixture) were fabricated as disk specimens of 100 mm (diameter) × 50 mm (thickness) for this study. The mix design for each group is tabulated in Table. 1. The CA used in this study is Masterlife 300D, a crystalline capillary waterproofing admixture product of BASF. Based on the literature's recommendation (Roig-Flores et al. 2015), (Roig-Flores et al. 2016), (Pazderka and Hájková 2016) 2% of cement weight was selected as the CA portion in the ECC-CA mixture.

Table 1: Mixture designs (for 1 m3)

Mixture	Amount (kg/m^3)						
	Fly ash	Cement	Sand	Water	PVA fibers	Super-plasticizer	CA
ECC	823	375	435	318	26	3	-
ECC-CA	823	375	435	318	26	3	7.5

1.2 Test Plan

The prepared disk specimens were cracked using a split tensile loading test (Figure 1.a). This test was selected to generate V-shape cracks that can have enough space for allowing the production and measuring the capacity of healing products, also can keep the integrity of the specimens and are mon common in practice (Millero et al. 2008). The loading process was recorded and further analyzed using a Digital Image Correlation (DIC) technique. The DIC results and visual observations indicate formation of V-shape cracks (see Figure 1.b). The test was conducted at a loading rate of 100kN/min and crack width was measured by DIC. Table 1. summarizes the Water Permeability (WP) test plan and exposure conditions. The WP test is carried out right after the cracking and after each 1-month exposure condition for two months, generating three WP test result for comparison over time.

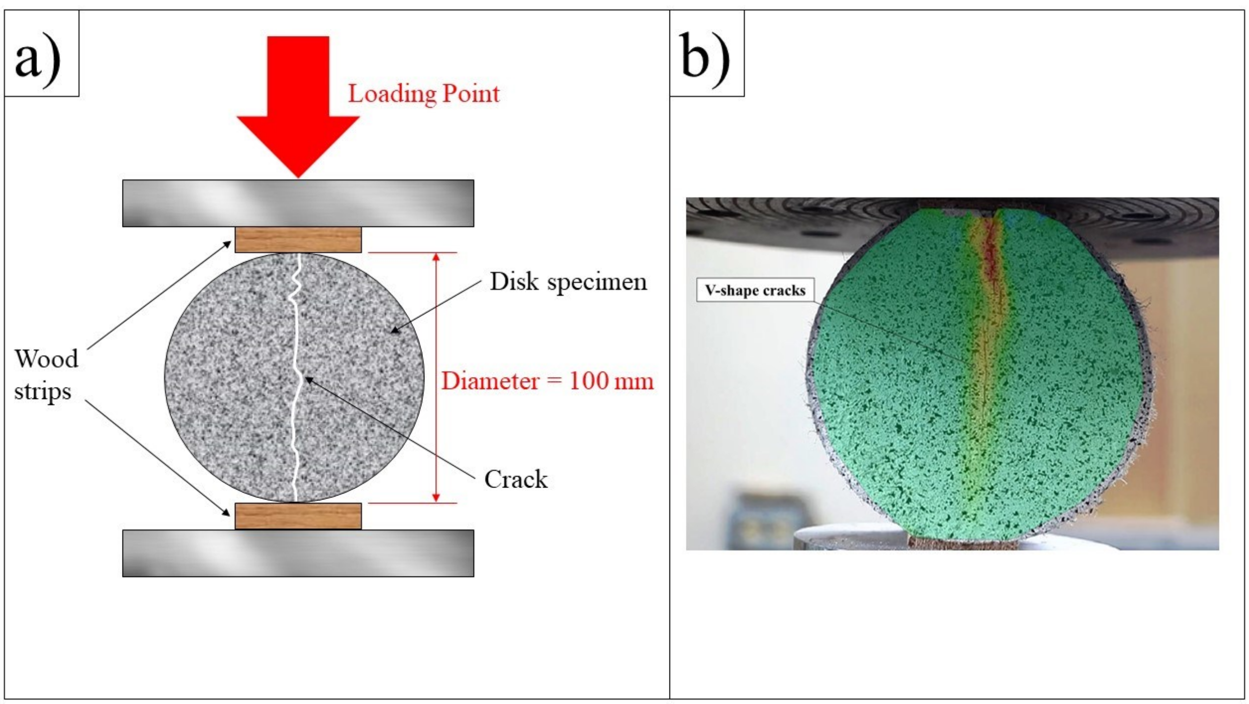

Figure 1: Tensile split loading test, a) Loading setup and, b) DIC analyzed surface indicating V-shape cracks

Table 2: ECC and ECC-CA test plan

<table>
<tr><th rowspan="2">Group name</th><th colspan="6">Test timeline</th></tr>
<tr><th>Cracking specimens with split tensile test</th><th rowspan="7">(WP1)</th><th>Exposure condition (1 month)</th><th rowspan="7">(WP2)</th><th>Exposure condition (1 month)</th><th rowspan="7">(WP3)</th></tr>
<tr><td rowspan="3">ECC</td><td rowspan="6"></td><td>Tap water</td><td>Tap water</td></tr>
<tr><td>Sea water</td><td>Sea water</td></tr>
<tr><td>Air dry</td><td>Air dry</td></tr>
<tr><td rowspan="3">ECC-CA</td><td>Tap water</td><td>Tap water</td></tr>
<tr><td>Sea water</td><td>Sea water</td></tr>
<tr><td>Air dry</td><td>Air dry</td></tr>
</table>

Note: Three specimens were tested for each case of study, making a total number of 18 specimens.

Water permeability test investigates the amount of water that passes through a crack, and is able to represent the crack filling (sealing) efficiency over time. Different techniques were adopted in the literature to evaluate the self-healing of concrete through measuring the reduction in the permeability of cracked specimens (Van Mullem et al. 2019; Edvardsen 1999). The test setup fabricated for this study is schematically shown in Figure 2 (dimensions are not to scale). The setup used in this investigation consists of three major sections, i.e., bottom and top halves of the plastic chamber and the pipe attached to it from the top. After finishing the cracking process, each disk specimen was placed inside the bottom half of the fabricated plastic chamber, and the gap between the specimen and the inner surface of the chamber was sealed with plastic wires and plumbers' putty. Next, the upper half of the chamber was placed on the bottom half, and the two parts were fixed using six bolts provided around the middle of the chamber. The water was then poured from the top of an 8mm tube attached to the chamber, and the time that water traveled inside a 1000mm length of the tube was measured with a stopwatch. The test was repeated three times for each specimen, and the average of the recorded time was used for comparison. A plastic valve was also attached to the chamber to release the air trapped inside, so the pure water pressure determines the travel time. The water permeability test was conducted after each 1-month exposure condition, and the time values obtained were further used to measure the crack healing efficiency.

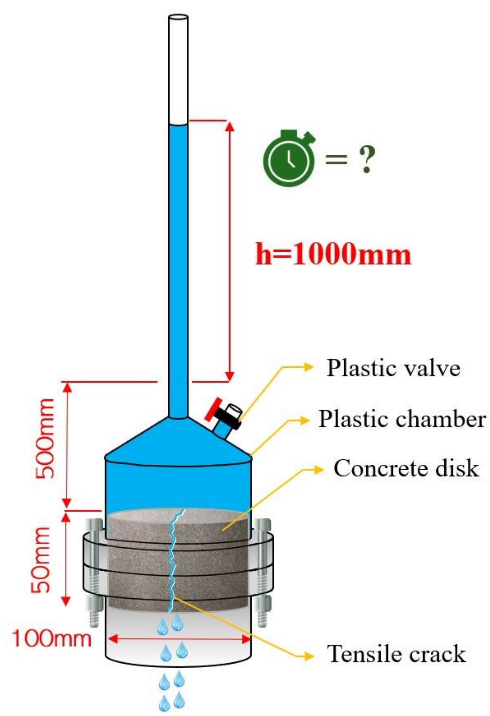

Figure 2: Water permeability test setup

2 RESULTS AND DISCUSSIONS

2.1 Water Permeability Results

The Water Permeability (WP) test setup was designed to measure the time that water traveled through the 1000mm length of the tube. After cracking, the travel time obtained through WP was reported to be between 2-55 seconds. The cracks generated in this study are not the same size, and the travel time depends on the free space within cracks. Also, ECC possesses high fractions of PVA fibers that could help with clogging the water pathway in some specimens. To normalize the results, the travel time recorded (T) through WP test was converted into flow rate (Q), using the following equation:

[1] $Q = \frac{V}{T}$

Where V is the constant volume of the tube in 1000mm length. To consider the effect of crack width on the recorded time, the calculated Q was then used for converting into velocity (U) through the following equation:

[2] $U = \frac{Q}{A}$

Equation [2] considers the effect of crack width through the area (A) perpendicular to water travel direction. To do so, it is assumed that the area in this case is a rectangle with a width equal to the maximum crack width of each specimen (W_{max}) and a unit length. Maximum crack width was obtained for specimens by means of DIC measurements, and then used to normalize the velocity. Velocity factor (U_f) is finally defined as follows to compare the results.

[3] $U_f = \frac{Q}{W_{max}}$

Figure 3 demonstrates the U_f values obtained for each disk specimen at three time intervals (month=0,1,2). The two groups of ECC and ECC-CA are categorized based on the exposure conditions, and the maximum crack width (W_{max}) for each specimen is also labeled along the specimen's name on the charts. The curves for ECC and ECC-CA in air dry condition indicate almost no reduction in the velocity factor over time. This result suggests that healing of large cracks through hydration, carbonation or crystallization mechanisms is not feasible in the absence of moisture. Even some trends in air dry condition showed an increase in the velocity factor during the first month of exposure that could possibly be due to the shrinkage of the cementitious composite, which subsequently increases the gap between the two faces of cracks. This behavior was more vivid during the first curing period of specimens in group ECC-CA, with an average of 75% increase in the U_f value. For the same period, results show 23% increase in the average U_f for ECC specimens.

As demonstrated in Figure 3, tap water curing condition is almost neutral in terms of improvement in transport properties of ECC and ECC-CA groups. Apparently, large deflections (>300μm) prevent the formation of healing products made through binder late hydration or calcite precipitation. Even if there is any small healing product formed inside the cracks, it is not impactful enough to resist the water flow. However, the results from specimens exposed to sea water prove significant reduction of velocity, specially over the first month of curing. The average reduction for ECC and ECC-CA groups were found to be 48% and 77% in the first month. During the second exposure period, the trend changed to 7-8% reduction for both ECC and ECC-CA groups. It could be concluded that due to the limited concentration of ions in the sea water, and the fact that sea water used in this experiment was kept unchanged throughout the experiment, most of the chemical reactions possibly took place during the first month and consumed the ions from the surrounding environment. Also, results confirm the high potential of sea water as a favorable surrounding environment for improved autogenous self-healing. Addition of CA to ECC makes the impact even greater. The active chemicals and minerals manufactured as CA are capable of reacting with ions transferred through sea water and precipitate new material.

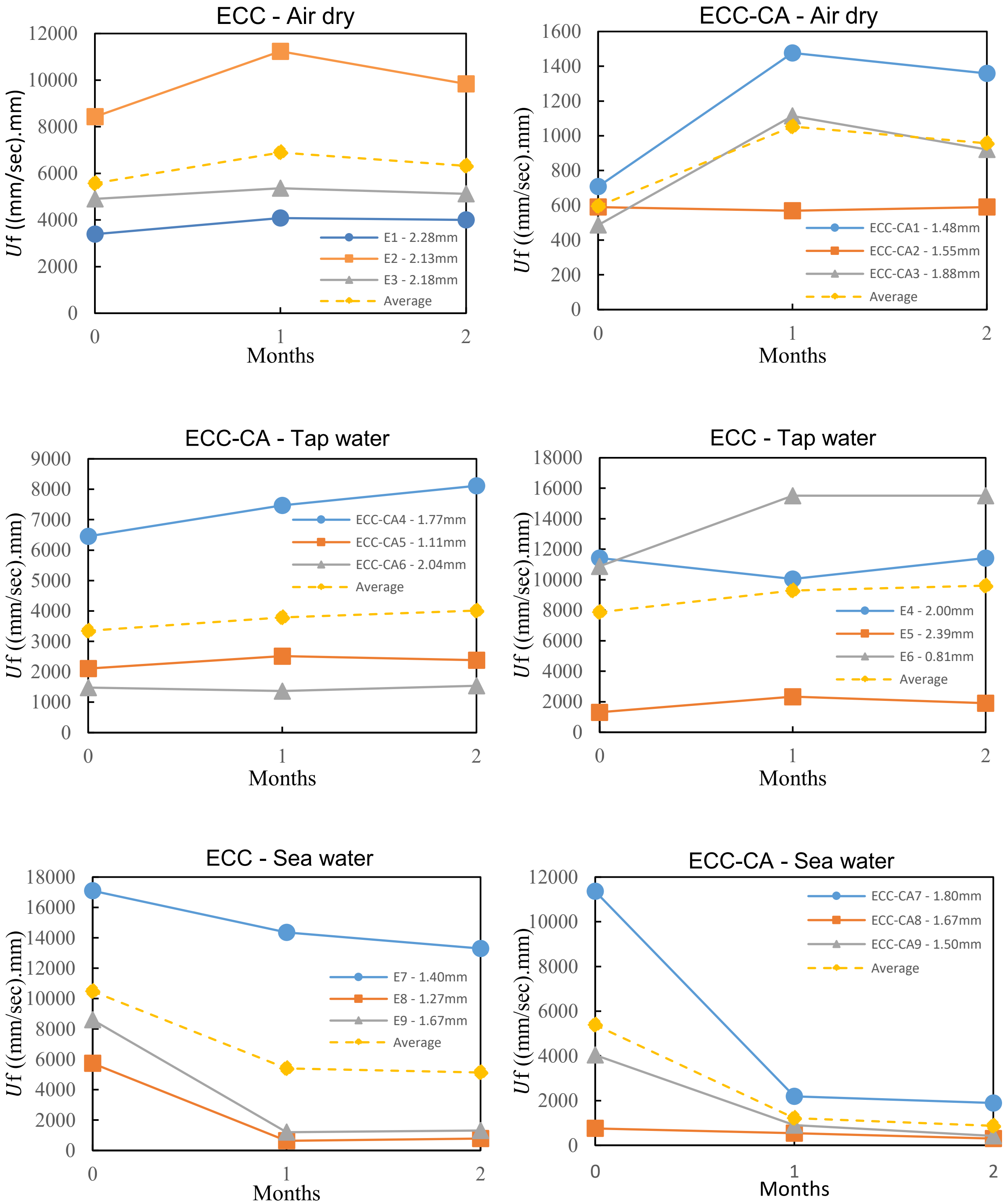

Figure 3: Velocity factor (U_f) vs. curing time for ECC and ECC-CA specimens exposed to different exposure conditions

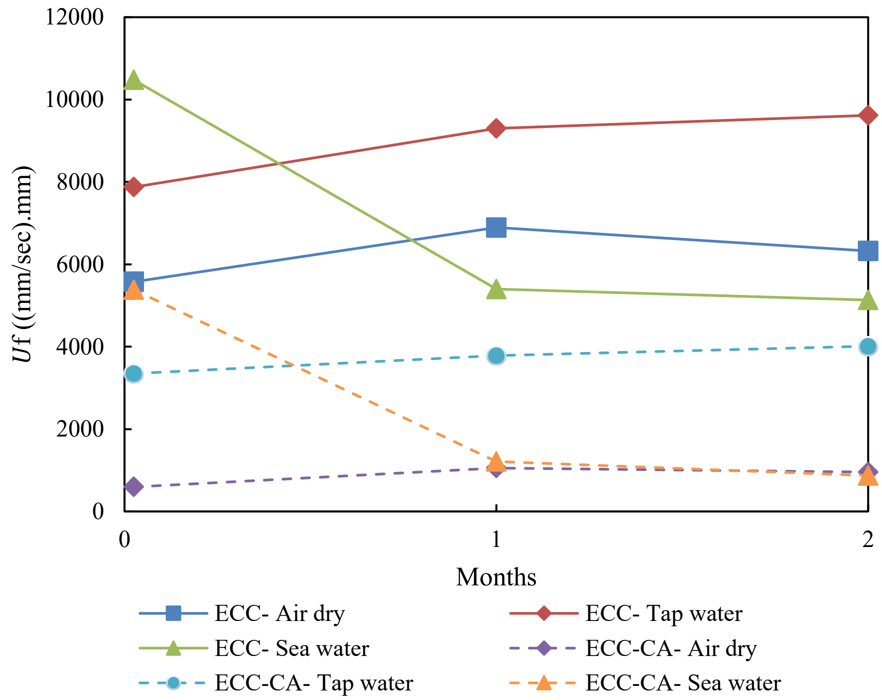

Figure 4. Average Water Permeability (WP) results for ECC and ECC-CA specimens

Figure 4 presents a comparison of average U_f values for all three exposures in ECC and ECC-CA groups. It is shown that sea water was the only exposure condition that could improve the transport properties of largely cracked specimens. The other two exposures of tap water and air dry were determined to have either no or negative impact on cracks' permeability. Generally, the ECC-CA group demonstrated lower values for U_f, indicating lower water permeability.

2.2 Visual Observations

Precipitation of healing products was visually observable for specimens exposed to sea water. Even at large deflections of about 2mm and in a short time period, the formation of self-healing products in ECC-CA exposed to sea water can be visually confirmed. These products are assumed to be precipitations of calcite/brucite.

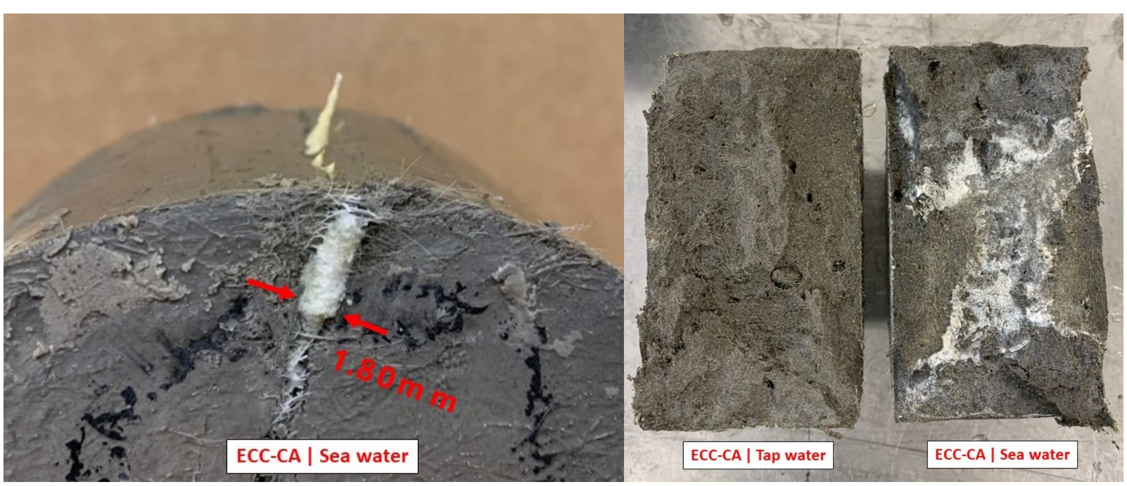

Figure 5: Visual observations indicating precipitation in the specimens exposed to sea water

3 CONCLUSION

The presented experimental program measures the extent of self-healing in ECC specimens with and without crystalline admixture. Three different exposure conditions of air dry, tap water, and sea water were adopted for this purpose. Large crack openings of ~1-2 mm were generated in disk specimens using a split tensile test to provide enough space for healing products to form. The self-healing efficiency was measured via a water permeability test conducted at different time intervals.

Water permeability results indicated that when large cracks were present in ECC specimens exposed to air dry and tap water conditions, the material was unable to fill the cracks with further hydration or calcite precipitation. This behavior remained unchanged for the ECC-CA group as well. On the other hand, the sea water exposure condition proved significant improvement in crack healing, such that cracks up to 2mm were filled in the ECC-CA group. The majority of the process took place in the first month of exposure, indicating the relatively high rate of reactions for self-healing process in the marine environment.

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