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Effect of Crack Repair by Bio-Based Materials Using Alginate and Bacillus Subtilis under Wet and Dry Environment Part-I

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Abstract. *This study examined crack repair methods using alginate gel films (1.5 wt.%) mixed with healing agents under wet and dry actions. The healing agents consist of Bacillus subtilis (natto) as an aerobic microorganism and glucose as an organic carbon source, thereby producing insoluble calcium carbonate in the gel films in the presence of calcium ions. In this study, repaired surface in cracked mortar specimens (ϕ50×100mm) was observed by microscope. In addition, water absorption test was carried out in assessing the effectiveness of crack repair for the cases of alginate gel films with or without calcium carbonate precipitation induced by the microbial activities. Based on the observations made by microscope, precipitates in white color were clearly observed after 8 weeks on the cracked surface of specimens especially under wet conditions. In the case of the specimens with higher sealing rate of crack repair, the alginate gel films were remained intact under dry and wet actions, which is advantageous to keep moisture by swelling. Water absorption test was carried out using repaired mortar specimens with average crack size of 0.5 mm. Based on the results obtained, water absorption rate is substantially decreased in the specimens with increasing sealing rate of crack repair. The results suggest that higher crack sealing rate associated with calcium carbonate precipitation leads to higher resistance against water absorption tested in accordance with ASTM C 1585.*

Keywords: *Self-Healing, Aerobic Microorganisms, Bio-Composite, Water Absorption.*

1 Introduction

Recently, repair materials associated with microbial induced calcium carbonate precipitation (MICCP) have been intensively studied in the field of self-healing concrete *e.g.* (Jonkers *et al.*, 2010). We proposed a liquid based repair system comprising dry yeast, organic carbon sources, calcium sources mixed with Tris alkali buffering solution (Kawaai *et al.*, 2016; Putri *at el.*, 2016). The grout used for repairing cracks in concrete is supposed to seep into deeper zones in cracks, joints and gaps spatially distributed in larger areas. Then, calcium carbonate precipitation mainly contributing to the sealing effect can be formed in the cracks well before 24 hours (Kawaai *et al.*, 2016). It has been reported that the precipitation rate was largely dependent on the concentration of constituents, pH levels, and ambient environment such as temperature. The effect of temperature is significant in facilitating the precipitation process (Putri *et al.*, 2016), thus leading to a larger amount of precipitates formed compared to those tested in normal room conditions.

Apart from the grout system, the activity of Bacteria added to mixing water was observed to be limited owing to the pore solution with highly alkaline environment (Jonkers, 2011). In order to protect them from corrosive agents and the severe environment, much research has been carried out in the development of encapsulation techniques including expanded clay particles, glass tubes, superabsorbent polymer, more recently alginate-based systems (Jonkers, 2011; Tittleboom *et al.*, 2011; Wang *et al.*, 2014; Palin *et al.*, 2016).

According to past research reported by Kawaai *et al.*, (2017), precipitation of calcium carbonate in alkaline environment has been confirmed by precipitation tests using aerobic Bacillus subtilis (natto) encapsulated in calcium alginate capsules. Sodium alginate extracted from brown seaweed could provide viscosity in aqueous solution, which varies depending on the concentrations. When sodium alginate dissolved in a liquid is used for repairing cracks in concrete, there is a strong possibility that the alginate reacts with calcium ions available on the cracked surface, thus forming a polymer comprising calcium alginate via ion-link on the cracked surface. This could result in in-situ encapsulation for the microorganism and nutrients in the cracks. Generally, there are two types of microorganisms, anaerobic and aerobic microorganisms. The former can metabolize an organic carbon sources in an oxygen-free environment. On the other hand, the latter requires oxygen for the metabolic activity. In this study, we selected Bacillus subtilis (natto) as an aerobic microorganism. The liquid-based mixture is supposed to be applied to concrete structures in tidal zones under marine environment. Dissolved oxygen is expected to be available during the wet and dry cycles. Based on the above background, this study examines the effect of crack repair using the viscosity modified mixture containing the Bacillus subtilis (natto) and nutrients via liquid-based approach under wet and dry conditions

2 Experimental Programs

2.1 Materials

First, culture solution of Bacillus subtilis (natto) containing sodium alginate is stirred using a stirrer for 30 minutes until the sodium alginate dissolves. Subsequently, Tris buffer solution or glucose is added. The concentrations of each constituent of the mixtures are shown in Table 1.

The experimental parameters include the presence (N1 mixtures) and absence (W mixture) of Bacillus subtilis (natto), the concentration of sodium alginate is specified as 1.5 wt.% for all the mixtures tested. The use of Tris buffer solution with concentrations of 0.1 mol/L is also considered as a testing parameter. In this study, three mixtures were totally prepared.

Table 1. Concentrations of each constituent of mixtures.

Mixtures		Sodium Alginate (%)	Glucose (mol/L)	Tris buffering solution (mol/L)
N1	N1-G1-T0	1.5	0.4	-
	N1-G1-T1	1.5	0.4	0.10
W	W -G0-T0	1.5	-	-

2.2 Cultivation of Bacillus Subtilis (natto)

In this study, Bacillus subtilis (natto) was cultivated using culture media mainly comprising NH_4Cl, NH_4NO_3, Na_2SO_4, $MgSO_4$, $CaCl_2$, K_2HPO_4, KH_2PO_4 in this study. The round rod shape of Bacillus subtilis (natto) forms a spore per a bacterial cell in the bacterial body. Spores are formed when it becomes an environment not suitable for growth such as oxygen, water and nutrient sources. And then, they become dormant and inactive. However, if the environment is set, the spore dormancy state ends, and germination begins. And then it becomes vegetative cell and the activity starts. Therefore, the Bacillus subtilis (natto) has higher resistance to environmental fluctuation compared to other microorganisms. In this study, the culture solution of the Bacillus subtilis (natto) after 24 hours was used as repair mixtures. Before preparing the mixtures, the dissolved oxygen concentration in the culture solution was measured to confirm the metabolic activity of Bacillus subtilis (natto) without an aeration apparatus.

2.3 Specimen Preparation and Exposure Tests

In this study, a mortar specimen with a water cement ratio of 0.5 and unit water content of 316 kg/m^3 and unit weight of crushed sand 1264 kg/m^3 was prepared using a cylindrical mold of ϕ 50×100 mm. Specimens were demolded after 24 hours and sealed curing was carried out until the age of 28 days. In order to simulate the cracks generated in the concrete member, split cracks were induced using a loading machine. The experimental set-up is similar to tensile strength test. The crack width was targeted around 0.5 mm. And then, the repair mixtures were poured into the cracks. After 1 week, exposure tests were carried out either through wet conditions (12 hours) and dry conditions (12 hours) or only wet conditions (24 hours). Wet cycles were executed using distilled water containing NaCl (3%) for both cases.

2.4 Measurement of Crack Width and Sealing Rate

The crack width and sealing rate by MICCP are measured using microscope (Dino-Lite Edge AM7915, ANMO) as shown in Figure 1. The five points highlighted in the figure are consistently measured using two specimens subjected to each exposure condition. The sealing rate is calculated based on the ratio of widths occupied with the precipitates over crack widths at each measurement point.

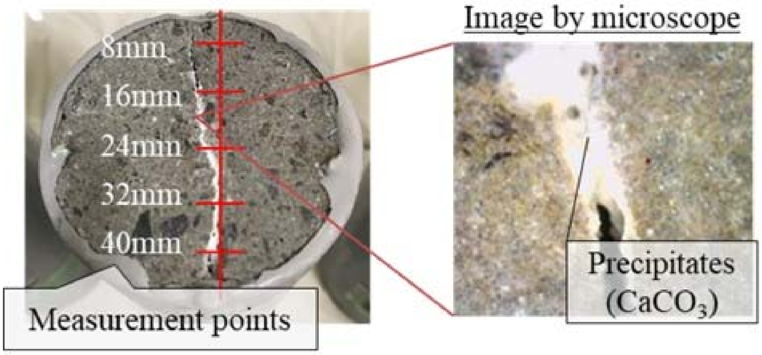

Figure 1. Measurement of crack width and sealing rate by microscope.

2.5 Measurements of Rate of Absorption of Water

The rate of absorption of water was measured in accordance with ASTM C 1585-13 using the specimens subjected to wet and dry cycles or wet condition. The test is carried out in such a way that the repaired surface was exposed to the distilled water and the water is basically absorbed through the capillary suction as shown in Figure 2. The measurements were carried out up to 7 days at predetermined intervals. Based on the changes of weight of the specimens, the rate of absorption of water is calculated using Eq. (1).

$$I = \frac{m_t}{ad} \tag{1}$$

where I: rate of absorption of water, m_t: changes of weight of the specimen, a: area of the immersed surface, d: density of water.

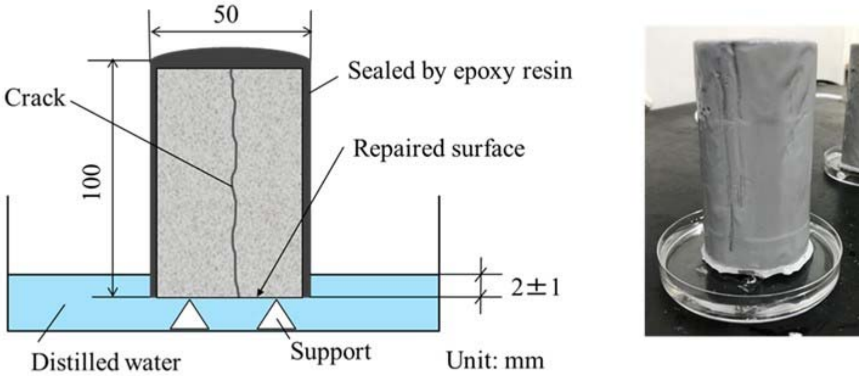

Figure 2. Testing set-up of absorption of water.

3 Experimental Results and Discussion

3.1 Exposure Tests Under Wet and Dry Cycles

For the mortar specimens repaired with each mixture, the materials formed on the cracked surface were firstly observed by microscope. The observations were made before repaired, 7 days later before the specimens were exposed to the dry and wet cycles and 56 days after the exposure. It should be noted that the average crack width was about 0.57 mm for the specimens tested. As can be seen in Figure 3, precipitates in white colour were not observed by microscope on the specimens for all the mixtures tested after the specimens were exposed to the room conditions controlled at 20°C for a week. Subsequently, precipitates were clearly observed on the specimens repaired with each mixture. However, the distribution of precipitates was largely varied, thus resulting in differences in the sealing rate. The sealing rates were 25.8% (N1-G1-T1), 40.0% (N1-G1-T0), 10.0% (W-G0-T0), and 13.8% (No repair). As observed by microscope, the sealing rate is clearly higher in the cases of specimens repaired with the N1 mixtures. It should be noted that the self-healing could naturally take place in the absence of the microorganism owing to the unhydrated cement and carbon dioxide dissolved in the distilled water. The difference observed in the sealing rate could be also affected by the gel films formed on the cracked surface. It is partly because the moisture kept in the gel films can be released to the crack even under dry conditions in the exposure conditions. Thus, the sealing effect could be obtained even under the dry conditions in such a case. As observed on the repaired surface after the exposure tests, the higher sealing rate obtained in the case of N1-G1-T0 mixture can be explained by the fact that the gel films were present and remained intact through the dry and wet actions.

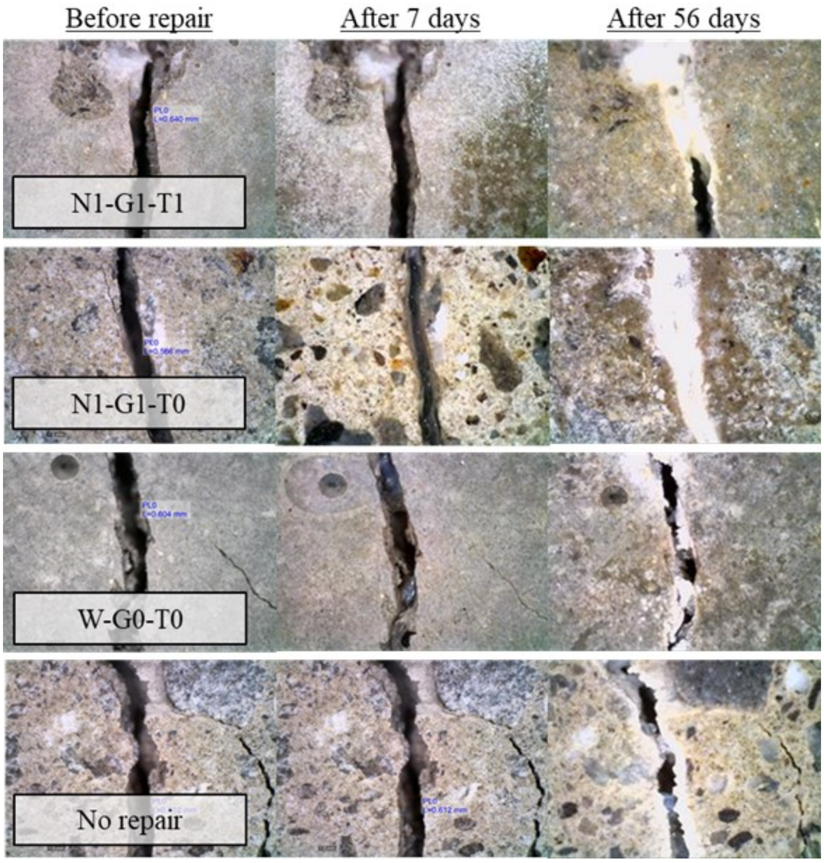

Figure 3. Observations by microscope on the repaired surface (wet and dry condition).

3.2 Exposure Tests Under Wet Condition

For the specimens exposed to the wet condition, the average crack width ranged from 0.48 to 0.51 mm. As shown in Figure 4, similar to those observed in the specimens subjected to the wet and dry conditions, precipitates in white color were clearly observed by microscope on the specimens especially repaired with the N1 mixtures after 56 days exposure. The sealing rates were 53.0% (N1-G1-T1), 74.4% (N1-G1-T0), 35.4% (W-G0-T0), and 37.9% (No repair). The effect of repair with respect to the sealing rate was found to be higher in the case of the wet conditions compared to those observed in the specimens exposed to the wet and dry environment. This could be attributed to the higher availability of moisture and dissolved oxygen for the MICCP processes in the N1 specimens. It should be noted that the gel films were remained intact especially in those specimens. This contributed to the bio-composite formation accompanied by the calcium carbonate precipitations inside the gel films. The specimens repaired with each mixture and exposed to the exposure tests were further tested by the water absorption tests, which is discussed in Section 3.3.

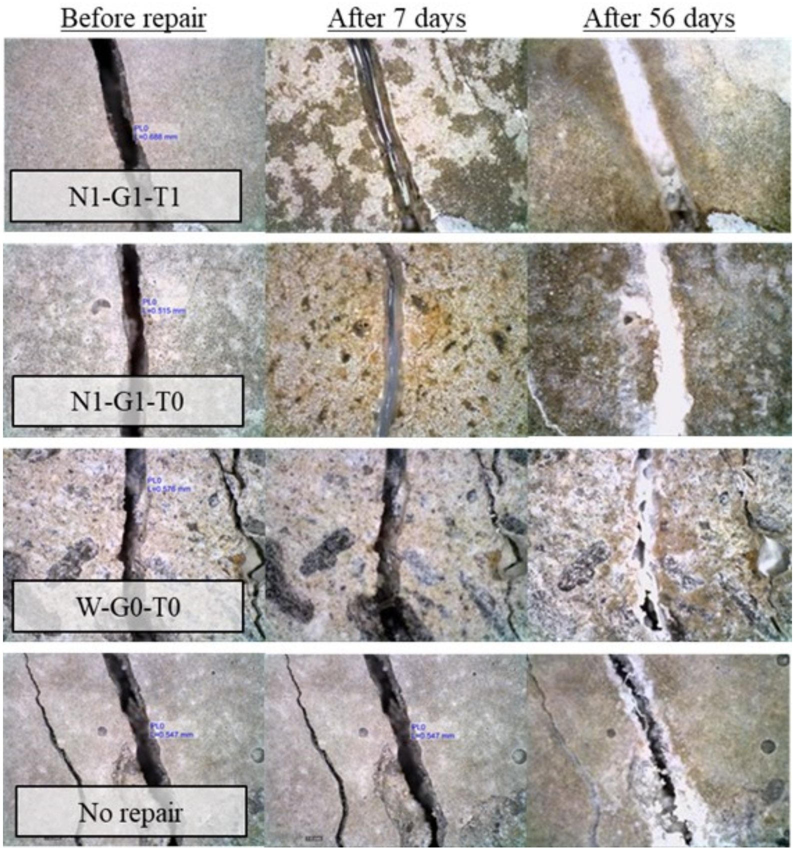

Figure 4. Observations by microscope on the repaired surface (wet condition).

3.3 Rate of Absorption of Water

Water absorption test was carried out based on modified procedure in accordance with ASTM C 1585. According to the standard, the primary gradient is obtained based on the changes of weight up to 24 hours, and the subsequent changes of weight are classified into secondary gradient. Figure 5 shows the primary gradients calculated based on the testing results for the all the mixtures including specimens without repair. As can be seen in the figure, the primary gradients are decreased with increasing rate of sealing effect of crack repair. In particular, the smallest gradient was obtained in the case of N1-G1-T0 mixture. This is consistently observed in each exposure condition, thus suggesting that the crack repair using the N1-G1-T0 mixture contributed to higher resistance against the ingress of water through the cracks. Although the sealing rate is slightly lower in the case of specimens exposed to wet and dry environment as reported in Section 3.1, the resistance against the water absorption could be enhanced using the crack repair though the MICCP. As discussed in Section 3.1 and 3.2, the presence of gel films are significant factors contributing to higher sealing effect in both exposure cases, which led to the formation of bio-composite through the calcium carbonate precipitation. The results suggest that higher sealing rate of crack repair associated with calcium carbonate precipitation leads to higher resistance against water absorption tested in accordance with ASTM C 1585 in this study.

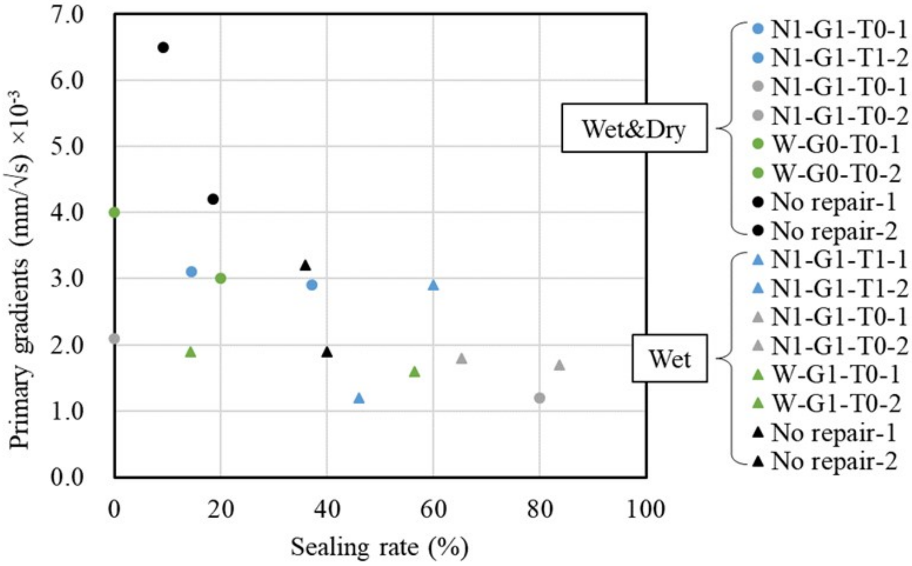

Figure 5. Primary gradient of water absorption test and sealing rate of crack repair.

4 Conclusions

- Based on the observations made on the sealing rate of crack repair, the presence of gel films remained intact on the cracked surface under exposure conditions could contribute to higher sealing effect in the cases of the N1 mixtures.
- The results suggest that higher sealing rate of crack repair associated with calcium carbonate precipitation leads to higher resistance against water absorption especially in the case of the N1-G1-T0 mixture under both exposure conditions (wet&dry and wet) tested in this study.

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