BIO-BASED MATERIALS FOR REPAIRING CRACKS IN CONCRETE: AN EXPERIMENTAL STUDY

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PREFACE

Recently, liquid-based repair techniques in the field of self-healing through the use of microbial induced calcium carbonate precipitation (MICP) have been intensively investigated. The mixtures are typically comprised of a microorganism, an organic carbon source and a calcium source which is readily available in concrete. When dry yeast is selected as the microorganism, carbon dioxide produced through the microbial metabolic processes consuming an organic carbon source such as glucose provides carbonate ions. The carbonate ions lead to react with calcium ions present in the mixture, leading to the precipitation of calcium carbonate depends on the pH levels in the alkaline environment. It should be noted that the material produced through the reactions is not harmful to concrete materials because the precipitates are mainly comprised of calcium carbonate which is one of the reaction products formed by carbonation of hydration products. Besides the material properties, the mixture is a less viscous material compared to conventional materials such as epoxy resin. This may overcome shortcomings associated with the conventional repair materials as mentioned previously. Thus, it would be beneficial if the mixtures penetrate into deeper zones of gaps formed between concrete members and could effectively improve the water tightness of concrete with defects.

In this research, the applicability of yeast-based mixtures for concrete repair has been studied. This technique employs yeast, glucose and calcium acetate mixed in Tris buffer solution. The microbial metabolic process leads to precipitation of calcium carbonate. Influencing factors on the precipitation rate depending on the constituents of yeast-based mixtures involving types of dry yeast and the concentration of yeast-based mixtures were studied. The influence of temperature on the precipitation rate was also examined for practical application.

Initially, tube precipitation test method was conducted for investigating the pH and the precipitation rate of calcium carbonate by measuring filtered paper. Then, calcium ions produced in the mixture were measured by commercially available meter (calcium ion electrode model

CA-2031) to calculate the decreasing rate of calcium ions in test tubes. The result showed that the decreasing rate of calcium ions has a good correlation with the precipitation rate of calcium carbonate measured using the filtered paper. The decreasing rate of calcium ions was almost equal to the amount of precipitation of calcium carbonate. Therefore, larger decreasing rate of calcium ions at early stages of the tests means that the precipitation rate of calcium carbonate is higher. In this thesis, the decreasing rate of calcium ions is used as an index of the precipitation rate of calcium carbonate under the test tube conditions.

Whole series of the experiment in this study were studied based on specific objectives of each experiment. The several factors affecting the precipitation rate of calcium carbonate were examined in each chapter. First, this study investigated the effects of changes of temperature to increase the precipitation rate of calcium carbonate in mixtures tested. The second series of experiments was performed to examine the effects of dry yeast's type to enhance the precipitation rate of the calcium carbonate. In order to assess the influence of types of dry yeast, Fourier-Transformed Infra-Red spectroscopy (FT-IR) analysis was used. The third series of experiment was conducted to evaluate the influence of different concentration of yeast-based mixtures on calcium carbonate precipitation. Finally, this study observed the applicability of the yeast-based mixtures for repairing water leakage in concrete specimens. Based on the result of series experiments carried out, the best composition of yeast-based mixtures with the highest precipitation rate of calcium carbonate was selected. Furthermore, water permeability test was conducted on the concrete leakage to determine the flow rate and effectiveness of the yeast-based mixtures application.

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CHAPTER 1
INTRODUCTION

A. General

Concrete structures have been commonly used in infrastructure facilities. Concrete has a great variety of applications because it not only meets structural demands but also lends itself readily to architectural treatment. Although quality control of concrete used in these structures is strictly required, initial defects are likely to occur owing to structural constraints and poor workmanship. For example, construction-joint or gap especially at joints forms between structural members used in bridges structures. When it rains, it may cause leakage from the gap leading to a decrease in serviceability and should be aesthetically unacceptable.

Most of the codes of practice consider cracks smaller than 0.3 mm acceptable for aggressive environmental conditions [1], [2]. Some of cracks are not detectable and cannot be accessed [3]. The other factors such as location of the cracking in the damaged structure make repair difficult with conventional repair materials. Difficulties associated with repair techniques for such defects are that conventional repair materials are less effective in sealing the gap formed spatially distributed in large areas, especially deeper zones in concrete structures. For example, organic materials e.g. cement-based grout materials with higher viscosity are less practical in repairing deeper zones of cracks or gaps in large areas. In addition, they may cause adverse effect on the natural environment if they flow out through the gaps or cracks which are not completely repaired. Therefore, it is advantageous if the materials are not based on such materials. However, it should have similar materials properties of concrete.

The bio-based material technology is a relative recent advance for the improvement of durability and other concrete properties. In a self-healing approach, the application of isolated bacterial cultures and mixed cultures into the fractured concretes was found to seal cracks effectively [4]. This was achieved by the precipitation of calcium carbonate caused by metabolic activity of bacteria [5]. Bacteria culture can be injected into the concrete surface to trigger self-healing [6]. Furthermore, bacteria culture was sprayed onto the surface of cracked concrete in a parking garage [7]. Consequently, the water permeability of the cracked concrete was significantly reduced due to self-healing. Recently, many researchers have been studying bacteria-based approach, where bacteria is embedded in concrete in order to increase strength and improve durability [8].

In other investigations, bacteria was directly added to the concrete mix instead of spraying and injection approach [9]–[12]. Thus, cracks were plugged by the microbial precipitation due to ureolytic activity of bacteria. This proved to be a better approach compared to spraying and injection approach. However, severe environment within the concrete matrix tends to decrease the lifespan of the bacteria [9]. As a result, the efficiency of crack sealing decreased over time. Thus, it is necessary for the bacteria to be protected in order to increase its lifespan [13]. This protection was achieved by encapsulating the bacteria. Wang et al. [14], have encapsulated bacterial spores in a hydrogel before mixing them with concrete. Crack width of 0.5mm was completely filled by the calcium carbonate precipitation. The water absorption was also decreased to 68%. In other investigations, immobilization of bacteria in microcapsule led to a more enhanced performance in which the maximum crack width 0.97mm was filled [15]. More recently, bacteria was encapsulated in graphite nanoplates [16]. The result has shown that crack width of 0.81 mm was sealed for specimens pre-cracked at 3 and 7 days. Achal [17] and Mostavi [18] also evaluated self-healing efficiency based on the depth of crack plugged. They stated that the cracks were plugged up to crack depths of 27.2 mm and 32 mm, respectively. Achal [17] was demonstrated the bio-cementation ability of a bacterial strain Bacillus sp. CT-5 to seal cracks. On the other hand, Mostavi [18] used double-walled sodium silicate microcapsules. All the self-healing approaches for cracked concrete revealed that the encapsulation technique was more effective due to extension of the lifespan of bacteria for prolonged performance. And also larger size cracks were completely filled using the yeast-based mixtures.

On the other hand, liquid-based repair techniques in the field of self-healing through the use of microbial induced calcium carbonate precipitation (MICP) have been intensively investigated [19]–[24]. The mixtures are typically comprised of a microorganism, an organic carbon source and a calcium source which is readily available in concrete. Dry yeast commercially available was used as a microorganism due to its effectiveness and untreated prior to use. When dry yeast is selected as the microorganism, carbon dioxide produced through the microbial metabolic processes consuming an organic carbon source such as glucose provides carbonate ions. The carbonate ions lead to react with calcium ions present in the mixture, leading to the precipitation of calcium carbonate depending on the pH levels in the alkali environment. The $CaCO_3$ precipitates according to the following reactions:

$$C_6H_1\ O_6 \rightarrow 2C\ \ _2 + 2C_2H_5O \tag{1.1}$$

$$C\ \ _2 + H_2O \rightarrow C\ \ _3^{2-} + 2H^+ \tag{1.2}$$

$$C\ \ ^{2+} + C\ \ _3^{2-} \rightarrow C\ \ \ \ _3 \downarrow \tag{1.3}$$

It should be noted that the material produced through the reactions is not harmful to concrete materials because the precipitates are mainly comprised of calcium carbonate which is one of the reaction products formed by carbonation of hydration products. Besides the material properties, the mixture is a less viscous material compared to conventional materials such as epoxy resin. This may overcome shortcomings associated with the conventional repair materials as mentioned previously. Therefore, it would be beneficial if the mixtures penetrate into deeper zones of gaps formed between concrete members and could effectively improve the water tightness of concrete with defects. Also, it is a great interest for the repair of concrete in wet area and difficult to repair by injection.

Along this study, calcium carbonate precipitation was promoted by microbial metabolic process of bio-materials mixtures as shown in **Figure 1.1.** In addition to the reaction, it is essential to control the pH levels in mixtures to facilitate the precipitation of calcium carbonate.

Tris (Tris hydroxymethyl aminomethane) buffer solution with an alkali buffering function was used in this study. The initial pH in the Tris buffer solution was adjusted to 8.0 and/ or 9.0 using hydrochloric acid. The use of hydrochloric acid should be minimized in adjusting pH levels for ensuring no adverse effect on the integrity of hardened concrete.

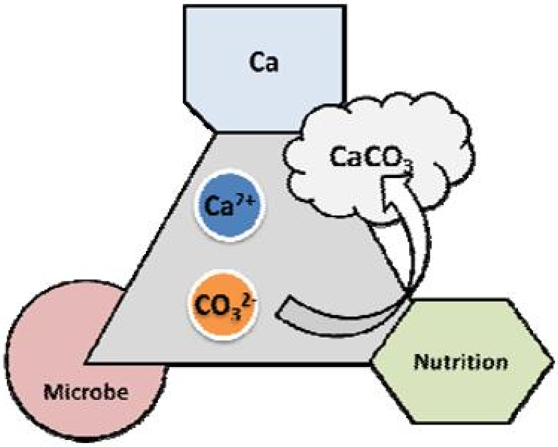

Figure 0.1 Basic constituents of bio-based repair materials

The source of calcium ions used for mixtures was chosen taking into consideration higher solubility and no adverse effect on concrete materials. The material should be also commercially available and cost-effective. Based on the considerations above mentioned, calcium acetate was chosen for this study. In addition, dry yeast commercially available was used as a microorganism, which is anaerobic and active in the oxygen-free environment. Organic carbon sources are necessary for the metabolism of the microorganism. Through the anaerobic fermentation, the yeast converts carbohydrates to carbon dioxide and alcohols in the presence of glucose ($C_6H_{12}O_6$) which was selected as an organic carbon source.

Calcium carbonate, as one of the most widely existing bio-minerals produced by organism, has three different crystal polymorph, such as calcite, aragonite, and vaterite. The phase diagram of $CaCO_3$ shows that calcite is the stable style under ambient conditions, aragonite is the high-pressure polymorph, and vaterite is thermodynamically unstable [25]. From the viewpoint of thermodynamics, calcite is more stable than aragonite. Vaterite is the least stable of these three phases and easily transforms into calcite in a chemistry laboratory. Although the amount of vaterite is far less than that of calcite and aragonite, it also plays key roles in biological life and health [26].

B. Objectives of the study

According to the previous research, Kubo et al. [21] developed yeast - based mixtures associated with microbial metabolic processes to repair concrete joint leakage. The results of this study showed that the application of the yeast-based mixture to a leakage point in cycle could not be repaired completely. And, Kawaai et al. [19] investigated some considerations on precipitation rate of calcium carbonate. This study concluded that the precipitation rate, particularly at early stages depends on the concentrations and initial pH levels. Also, calcium ions present in the mixtures were mostly converted into calcium carbonate which precipitated in the mixtures.

On the other hand, Yamamoto [27] explored the application of bio-based repair materials comprising dry yeast, glucose and calcium acetate to repair gap in concrete with 0.5mm crack width. This study showed the reduction effect on the water permeability was not clearly confirmed to seal leakage in concrete. Furthermore, there are not many cases studying the influence of temperature on calcium carbonate precipitation in yeast-based mixtures.

Based on the background as mentioned in Section 1.1, the main aim of this study is to examine the factors affecting the precipitation rate of calcium carbonate through the microbial metabolic process of yeast-based mixtures and applicability of bio-based material to repair water leakage in concrete.

Several factors affecting the precipitation rate of calcium carbonate were examined in each chapter. First, this study investigated the effects of changes of temperature to increase the precipitation rate of the calcium carbonate in mixtures tested. The mixtures were tested in different temperature conditions controlled at 10^{o}C, 20^{o}C and 30^{o}C up to 72 hours elapsed time.

The second series of experiments was performed to examine the effects of types of dry yeast to enhance the precipitation rate of calcium carbonate. In this study, three types of commercially available dry yeast were selected as microorganism. The third series of experiment was conducted to evaluate the influence of different concentration of yeast-based mixtures on calcium carbonate precipitation. The precipitation rate was observed up to 72 hours elapsed time.

Moreover, this study investigated the applicability of the yeast-based repair mixtures to concrete member. The best mixture with the highest precipitation rate of calcium carbonate based on the experiment was applied to the specimens with cracks. Correspondingly, water permeability test, flow rate analysis and observation by microscope were carried out to evaluate the effectiveness of the yeast-based mixtures for sealing cracks in concrete specimens.

Also, testing at microscale was conducted to identify and characterize the precipitated materials within concrete cracks after sealing. The precipitates were tested for Fourier-Transformed Infra-Red spectroscopy (FT-IR), Field Emission Scanning Electron Microscopy (FE–SEM) and X-Ray Diffraction (XRD) analysis for mineral identification formed through the microbial metabolic process of yeast-based mixtures in mixtures.

C. Contents of the book

Therefore, the book contains seven chapters. The contents of each chapter are briefly explained as follows. In chapter 1, the general introduction and background of yeast-based mixtures for concrete repair. Subsequently, the objective and methodology of this study are shortly described. Chapter 2 is related to experimental outline of the research. It is composed of materials and methods, testing procedure and evaluation technique of yeast-based mixtures precipitation.

Chapter 3 explains the effect of temperature on precipitation rate of calcium carbonate produced through microbial metabolic process of the yeast-based mixture. In order to evaluate the effect of changes of temperature, XRD analysis was conducted to examine the polymorph of calcium carbonate.

Chapter 4 presents the influence of the types of dry yeast on precipitation rate of calcium carbonate in yeast-based mixtures comprising yeast, glucose and calcium acetate. In order to assess the influence of types of dry yeast, Fourier-Transformed Infra-Red spectroscopy (FT-IR) analysis was used.

Chapter 5 describes the influence of the different concentration of yeast-based mixtures to increase the precipitation rate of calcium carbonate up to 72 hours of elapsed time. The precipitates were also tested for Fourier-Transformed Infra-Red spectroscopy (FT-IR) and X-Ray Diffraction (XRD) analysis for mineral identification formed through the microbial metabolic process of yeast-based mixtures in mixtures.

Chapter 6 presents applicability of the yeast-based mixtures to repair water leakage of concrete specimens. Also, this chapter describes the application of the yeast-based mixtures for concrete repair as a case study on cracked concrete specimens.

Finally, the conclusions and the recommendations for the further research of this study are given in chapter 7.