

EFFECT OF TEMPERATURE AND SOLUTION pH ON CALCIUM CARBONATE PRECIPITATION AS BIO-BASED REPAIR MATERIAL IN CONCRETE

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ABSTRACT: In the present work, the effect of temperature and solution pH on calcium carbonate precipitation from bio-based repair materials is investigated. A microbial metabolic process precipitates calcium carbonate. Mixtures of bio-based materials typically comprise a microorganism, an organic carbon source, and a calcium source. When dry yeast is selected as the microorganism, carbon dioxide produced through the microbial metabolic process of consuming an organic carbon source, such as glucose, provides carbonate ions. The carbonate ions lead to reactions with the calcium ions present in the mixture, leading to the precipitation of calcium carbonate. Mixtures were tested at temperature conditions of 10, 20, and 30 °C, and the pH of the solution was initially adjusted as 8.0 or 9.0, for up to 72 hours of elapsed time. In this research, temperature conditions of 20-30 °C is the optimum temperature range for increasing the precipitation of calcium carbonate. The results confirmed that the maturity index has a linear correlation with the decreasing rate of calcium ions. Large maturity indices mean that the decreasing rate of calcium ions is high. Maturity index was used as an indicator of the precipitation rate of calcium carbonate in the temperature range 10-30 °C; the datum temperature was 0 °C. The adequate concentration of the Tris buffer solution was found in the range 0.50-0.75 (mol/L), at an initial pH of 9.0, and 0.75-1.00 (mol/L), at an initial pH of 8.0. The rate of the initial reaction tended to be faster with the initial pH of 9.0.

Keywords: *Bio-based repair material, Precipitation, Calcium carbonate, Temperature, Solution pH*

1. INTRODUCTION

Concrete structures are commonly used in infrastructure facilities. Concrete has a great variety of applications because it not only meets structural demands but also readily lends itself to architectural treatment. Although quality control of the concrete used in these structures is strictly applied, initial defects are likely to occur owing to structural constraints and poor workmanship.

Most codes of practice consider cracks smaller than 0.3 mm to be acceptable for aggressive environmental conditions [1,2]. Some cracks are not detectable and cannot be accessed [3]. Other factors, such as the location of the cracking in the damaged structure, make repairs difficult when using conventional repair materials. Difficulties associated with the repair techniques for such defects include the fact that conventional repair materials are less effective in sealing the gaps which form and are spatially distributed over large areas, especially deeper zones in concrete structures. For example, organic materials (e.g. cement-based grout materials with higher viscosity) are less practical for repairing deeper zones of cracking or gaps in large areas. They may also harm 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 but have similar material properties to concrete.

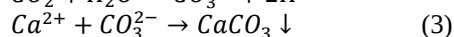
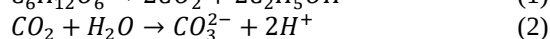
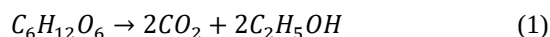
Bio-based material technology is a relatively recent advance for the improvement of durability and other concrete properties, especially in Indonesia. The Netherlands and Japan are other countries focusing on researching the field of bio-based materials.

In the self-healing approach, the application of isolated bacterial cultures and mixed cultures into fractured concrete was found to seal cracks effectively (by Jonkers et al. [4]). This was achieved by the precipitation of calcium carbonate from the metabolic activity of bacteria [5]. Bacteria cultures can be injected into the concrete surface to trigger self-healing [6]. Furthermore, bacteria cultures were sprayed onto the surface of cracked concrete in a parking garage in [7].

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 [8–13]. The mixtures typically comprise a microorganism, an organic carbon source and a calcium source, which is readily available in concrete. Commercially available dry yeast was used as a microorganism, due to its effectiveness and lack of treatment prior to use.

Putri et al. [14] also investigated the effect of

dry yeast types on calcium carbonate precipitation rates in Japan. When dry yeast is selected as the microorganism, carbon dioxide, produced through microbial metabolic processes (by consuming an organic carbon source such as glucose), provides carbonate ions. The carbonate ions 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:



It should be noted that the material produced by these reactions is not harmful to concrete materials because the precipitates mainly comprise calcium carbonate, and the carbonation of hydration products form one of the reaction products. Besides the material properties, a less viscous mixture is obtained when compared to conventional material, such as epoxy resin.

2. RESEARCH SIGNIFICANCE

In Japan, Kubo [10], Yamamoto [15], and Putri [16-19] explored the development of the microbial metabolic process in yeast-based mixtures at a constant temperature of 20 °C. Sakakihara [20,21] studied microbial-based grouting material for crack repairs. Avramenko [22] also reviewed the MICP technique. In real conditions, the temperature always changes. Therefore, in this research, the effects of temperature and pH solutions were examined to enhance the precipitation rate of the calcium carbonate in bio-based repair materials consisting of yeast, glucose, and calcium acetate. In this study, *fermipan* (commercially available dry yeast in Indonesia) was used as a microorganism.

3. METHODOLOGY

3.1 Basic Element of Bio-based Materials

In this research, calcium carbonate precipitation was promoted by the microbial metabolic processes in bio-material mixtures, as shown in Fig. 1. A Tris buffer solution with an alkali buffering function was used in this study. The initial pH solution was adjusted to 8.0 or 9.0 using hydrochloric acid.

The source of calcium ions used for the mixtures was chosen to take into consideration higher solubility and no adverse effect on concrete materials. The material also needed to be commercially available and cost-effective. Based

on these considerations, calcium acetate monohydrate was chosen for this study.

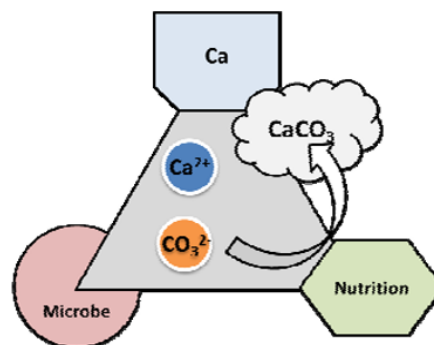


Fig. 1 Basic elements of bio-based materials

The commercial dry yeast was used as a microorganism, which is anaerobic and active in an oxygen-free environment. The metabolism of the microorganism used an organic carbon source, in which glucose ($\text{C}_6\text{H}_{12}\text{O}_6$) was selected. The yeast converted carbohydrates to carbon dioxide and alcohol in the presence of $\text{C}_6\text{H}_{12}\text{O}_6$ through anaerobic fermentation. Furthermore, the specifications of the basic constituent materials are described in Table 1.

Table 1 Specification of basic materials

Name of material	Chemical Formula	Molar mass	Solubility
Glucose	$\text{C}_6\text{H}_{12}\text{O}_6$	180.16	49.0 g/100 ml (20 °C)
Calcium acetate mono-hydrate	$(\text{CH}_3\text{COO})_2\text{Ca} \cdot \text{H}_2\text{O}$	176.18	34.7 g/100 ml (20 °C)
Dry yeast	-	-	-

Based on the basic composition provided by Yamamoto [15], a series of cases were set in controlled temperature conditions and measured for up to 72 hours elapsed time. The mix proportions in each case were specified as shown in Table 2.

Each mixture was prepared in test tubes. These mixtures were subsequently tested to measure the concentration of calcium ions and pH after the reaction was started. The test tubes were placed in conditions controlled at 10, 20, and 30 °C for the measurements. The concentration of calcium ions and pH was measured in filtered mixtures using commercially available meters and a calcium ion electrode meter. Each test was carried out using two test tubes, to confirm the consistency of the results obtained.

Table 2 Basic composition of yeast-based mixtures

Case	Dry yeast (g/L)	Glucose (mol/L)	Calcium acetate (mol/L)	Tris buffer solution pH	Mol/L	Temperature	Elapsed time (hours)
A	9.0; 18.0; 27.0; 36.0	0.1; 0.2; 0.3; 0.4	0.05; 0.10; 0.15; 0.20	8.0 9.0	0.25; 0.5; 0.75; 1.0	30 °C	12, 24, 36, 48, 60, 72
B	9.0; 18.0; 27.0; 36.0	0.1; 0.2; 0.3; 0.4	0.05; 0.10; 0.15; 0.20	8.0 9.0	0.25; 0.5; 0.75; 1.0	20 °C	12, 24, 36, 48, 60, 72
C	9.0; 18.0; 27.0; 36.0	0.1; 0.2; 0.3; 0.4	0.05; 0.10; 0.15; 0.20	8.0 9.0	0.25; 0.5; 0.75; 1.0	10 °C	12, 24, 36, 48, 60, 72

Correspondingly, other test tubes were placed at changing temperatures. The temperature was controlled ranging from 10-30 °C for up to 72 hours elapsed time. Temperature patterns for each case (cases AA, BB, CC, and DD) are shown in Fig. 6 to Fig. 9. Also, another bio-based mixture was prepared for different concentrations and pH solution measurements. For each group, the concentration of the Tris buffer solution was adjusted to 0.25, 0.50, 0.75, and 1.00 mol/L, respectively.

3.2 Tube Precipitation Test Method

In the tube precipitation test method, once the reaction began, each mixture in the test tube was measured to obtain the concentration of calcium ions and pH value. To measure the concentration of calcium ions and pH in mixtures after the reaction was started, each mixture was prepared in a test tube. First, each material, based on the mix proportion specified (shown in Table 2), was prepared and mixed with Tris buffer solution in a beaker. The mixture in the beaker was then stirred at 500 rpm for 5 minutes, or until each material was dissolved into the solution. The Tris buffer solution was added, to make a 120 mL solution. After separating the sample into four test tubes (each test tube held 30 mL, as shown in Fig. 2), the test tubes were placed in a temperature-controlled room for the measurements to be carried out.

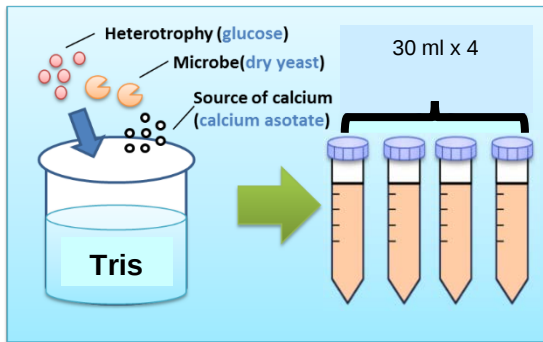


Fig. 2 Mixing process of bio-based material

After several elapsed time periods, filtration was carried out, to measure the precipitation rate of calcium carbonate (by the weight method). On the other hand, the concentration of calcium ions and pH were measured in mixtures using a commercially available calcium ion electrode meter. Each test was carried out using two test tubes to confirm the consistency of the results obtained.

The decreasing rate of calcium ions and precipitated calcium carbonate was determined from the equations below, based on the results of the calcium ions measured:

$$\text{Decreasing rate of calcium ion} = \frac{[C_0] - [C_a]}{[C_0]} \quad (4)$$

$$\text{CaCO}_3 = [Q] \times m \times M \times \frac{[C_0] - [C_a]}{[C_0]} \quad (5)$$

On the other hand, based on the concentration of calcium ions and pH measured, the concentration of carbonate ions was estimated using the following equations:

$$[\text{CO}_3^{2-}] = \frac{[C_0] - [Ca]}{M} \quad (6)$$

$$\text{pH} = \text{pKa} + \log \frac{[B] - [H^+]}{[BH^+] + [H^+]} \quad (7)$$

$$[\text{CO}_3^{2-}] = \frac{1}{2} [H^+] \quad (8)$$

where CaCO_3 is the amount of precipitates (g), $[Q]$ is the concentration of calcium acetate (mol/L), m is the amount of solution (L), M is the molar mass of calcium carbonate (100.09), $[C_0]$ is the initial concentration of calcium ions (g/L), $[Ca]$ is the concentration of calcium ions measured (g/L), pKa is the acid dissociation constant (8.2, 20 °C), $[B]$ is the concentration of the base (mol/L), $[BH^+]$ is the concentration of acid (mol/L), $[H^+]$ is the concentration of hydrogen ions (mol/L), $[\text{CO}_3^{2-}]$ is the estimated concentration of carbonate ions (mol/L) and M is the Molar mass of calcium (40.08).

4. RESULTS AND DISCUSSION

4.1 Effect of Different Temperatures

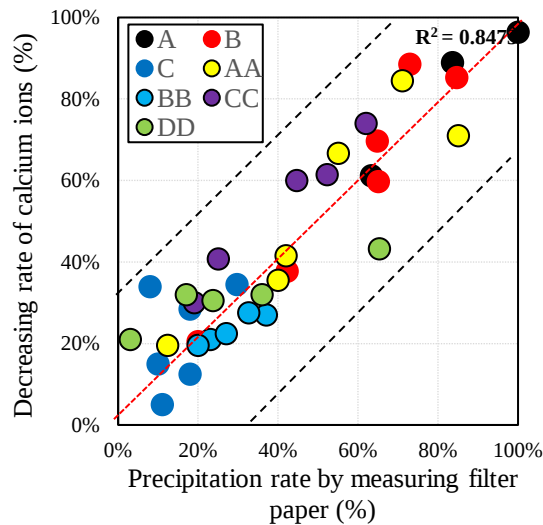


Fig. 3 The relation between the decreasing rate of calcium ions and precipitation rate (measured by filter paper)

The effect of temperature and different pH solutions was examined, concerning precipitation rates up to 72 hours after mixing. Figure 3 shows the relationship between the decreasing rate of calcium ions and the precipitation rate of calcium carbonate by measurements using filter paper and based on Eq. (4). The results confirmed that the decreasing rate of calcium ions had good correlation with precipitation rates measured by the filter paper.

The average difference between the decreasing rate of calcium ions measured and the precipitation rates calculated by the filter paper was less than 30%. The maximum precipitation of calcium carbonate was reached at 30 °C and the lowest at 10 °C. The decreasing rate of calcium ions was almost equal to the amount of precipitation of calcium carbonate. Therefore, the decreasing rate of calcium ions is used as an index of the precipitation rate of calcium carbonate under the test tube conditions.

Correspondingly, Fig. 4 shows a decreasing rate of calcium ions and Figs. 5-8 show the pH of the bio-based mixtures placed in several temperature conditions for up to 72 hours. This result confirmed that temperature rises to promote the formation of carbonate ions and the precipitation amount of calcium carbonate. As a result, the pH decreases due to the metabolic process in the mixtures. Otherwise, the precipitation rate of calcium carbonate was slower when the temperature became low. According to this research, 20-30 °C is the optimum temperature range for increasing the precipitation of calcium

carbonate.

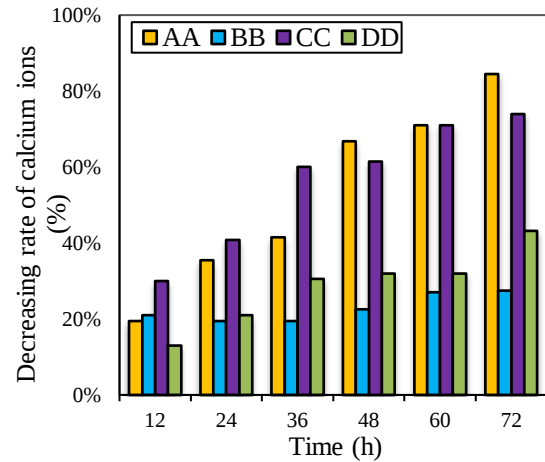


Fig. 4 Decreasing rate of calcium ions with changing temperature

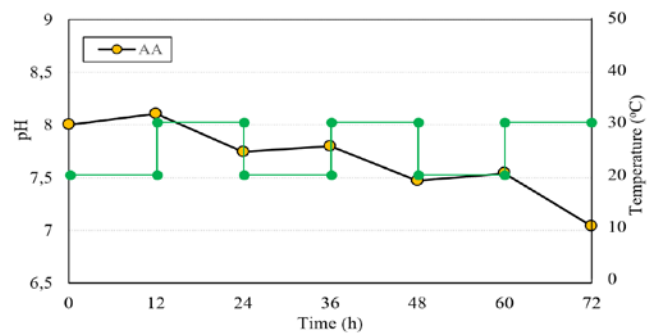


Fig. 5 pH of mixtures at temperatures of 20-30 °C

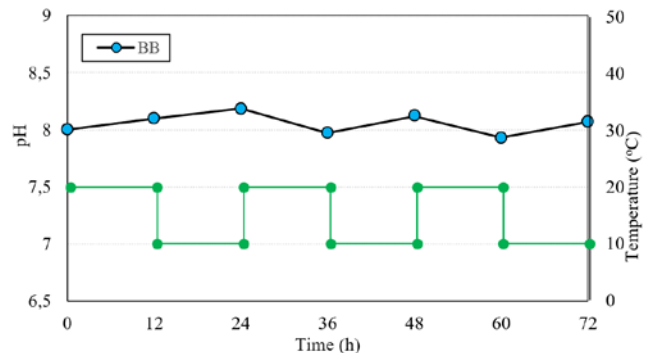


Fig. 6 pH of mixtures at temperatures of 10-20 °C

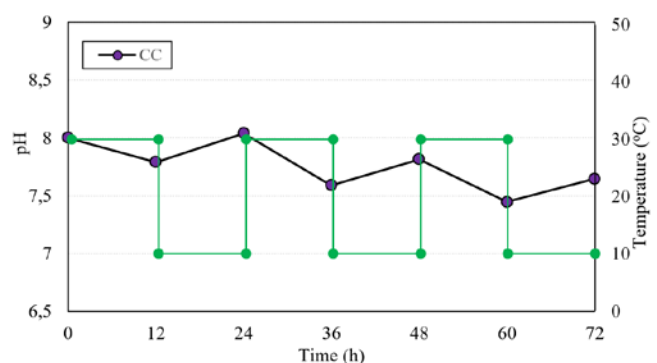


Fig. 7 pH of mixtures at temperatures of 10-30 °C

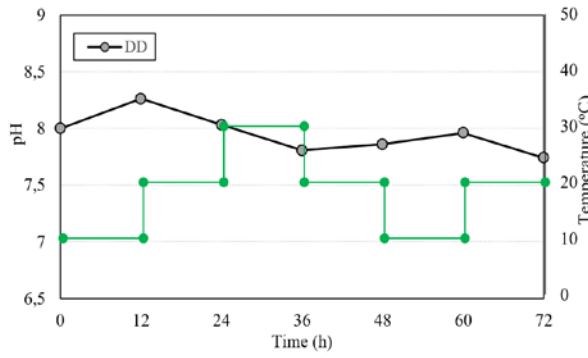


Fig. 8 pH of mixtures at temperatures of 10-20 °C

Furthermore, Fig. 9 displays the correlation between maturity index and the decreasing rate of calcium ions. The maturity index computed by the Nurse-Saul maturity function is described by Eq. (6) and is the temperature-time factor. Schematic temperature history and the temperature-time factor were computed according to the scheme for cases AA to DD. The Nurse-Saul maturity function is defined as:

$$M = \sum_0^t (T - T_0) \Delta t \quad (8)$$

where M is the maturity index (°C-hours or °C-days), T is the average temperature during the time interval Δt (°C), T_0 is the datum temperature (°C), t is the elapsed time (hours or days), and Δt is the time interval (hours or days).

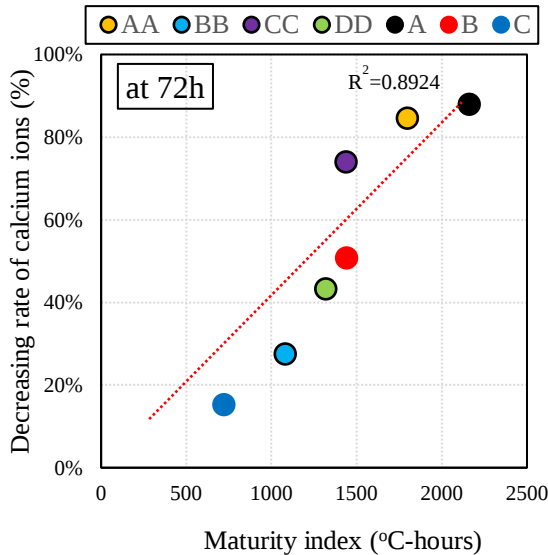


Fig. 9 Correlation between decreasing rate of calcium ions and the area of temperature pattern

Therefore, the results confirmed that the maturity index has a linear correlation with the decreasing rate of calcium ions. A large maturity index means that the decreasing rate of calcium ions is high. Therefore, the maturity index was used as an index of the precipitation rate of calcium carbonate in the temperature range 10-30 °C, and the datum temperature was 0 °C.

Based on these results, it is recommended that the concentration of the Tris buffer solution should be adjusted, depending on the initial pH, for facilitating the precipitation processes. Therefore, the utilisation of a lower pH (8.0) required a relatively higher concentration of Tris buffer solution, especially for mixtures comprising more yeast.

4.2 Effect of Different pH Solution

The mix proportion was either set as a basic composition (series 1), twofold of basic composition (series 2), threefold of basic composition (series 3), or fourfold of basic composition (series 4), to investigate the effects of concentration and pH solution. In Figs. 10-17, the black line refers to series 1, the red line refers to series 2, and the blue and yellow lines refer to series 3 and 4, respectively.

Figure 10 and Fig. 11 show the results of the decreasing rate of calcium ions and pH, up to 72 hours of elapsed time for all series. Based on Fig. 10, two groups are confirmed. The mixture with the lowest concentration of Tris buffer solution (0.25 mol/L) caused significant changes in pH in the mixture after 24 hours. The pH was kept lower than 6.5 and the decreasing rate of calcium ions reached less than 20%. Similarly, pH for the mixtures with concentrations of Tris buffer solutions of 0.50 and 0.75 mol/L, also fell below 6.5, except for the A1 mixture: this reached a pH of about 7.5 and 7.0 for the concentrations of Tris buffer of 0.50 and 0.75 mol/L, respectively.

On the other hand, the higher concentrations of mixtures tested and mixed with a higher concentration of Tris buffer solution (1.0 mol/L) also lead to lower. The pH decreases to 6.5 or less. However, the decreasing rate of calcium ions reached a higher amount, i.e. about 60% of the decreasing rate of calcium ions. Except for the A1 mixture, the pH was kept at about 7.5. It showed that a low concentration of mixture leads to higher pH. This is because the yeast's metabolism is not active and carbon dioxide generation is small.

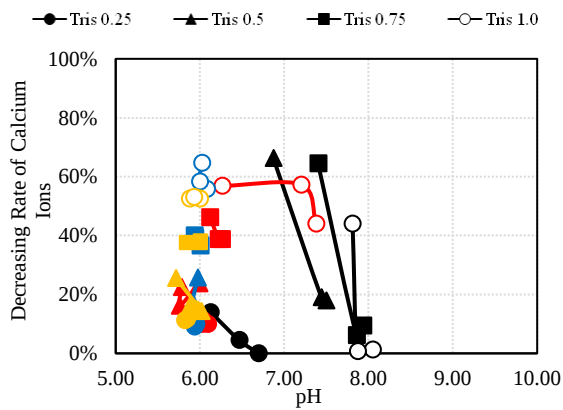


Fig. 10 Decreasing rate of calcium ions and pH (initial pH 8.0, T = 20 °C)

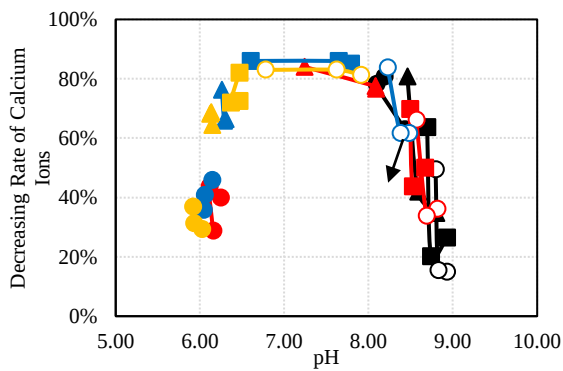


Fig. 11 Decreasing rate of calcium ions and pH (initial pH 9.0, T = 20 °C)

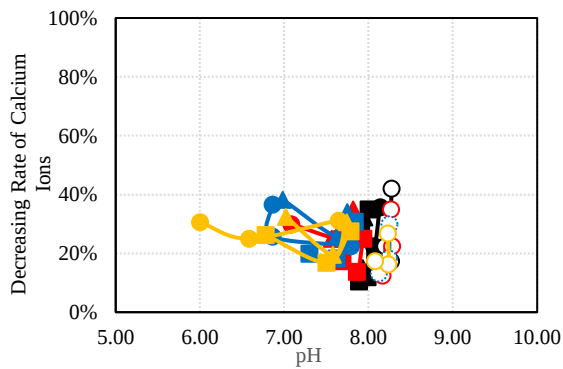


Fig.12 Decreasing rate of calcium ions and pH (initial pH 8.0, T = 10 °C)

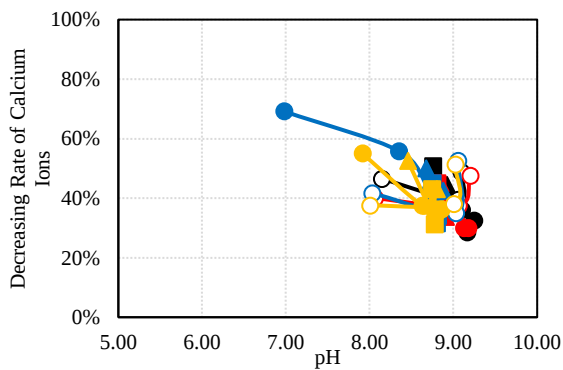


Fig. 13 Decreasing rate of calcium ions and pH (initial pH 9.0, T = 10 °C)

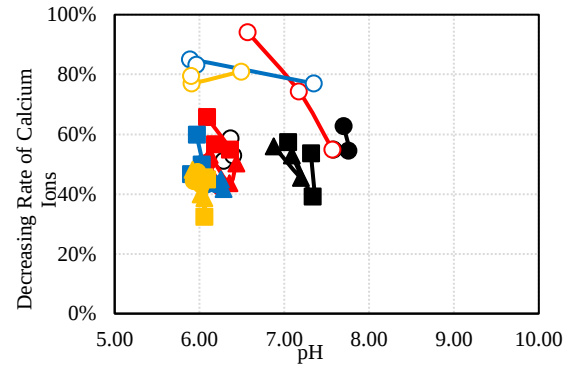


Fig. 14 Decreasing rate of calcium ions and pH (initial pH 8.0, T = 30 °C)

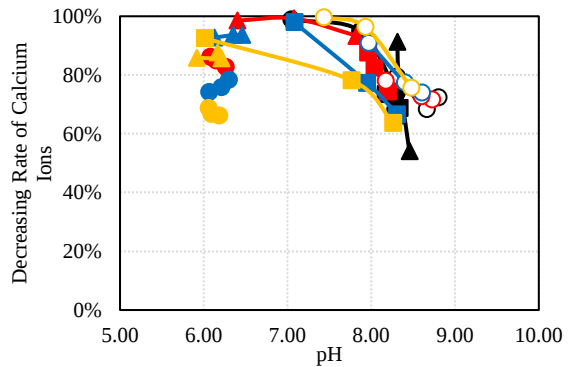


Fig. 15 Decreasing rate of calcium ions and pH (initial pH 9.0, T = 30 °C)

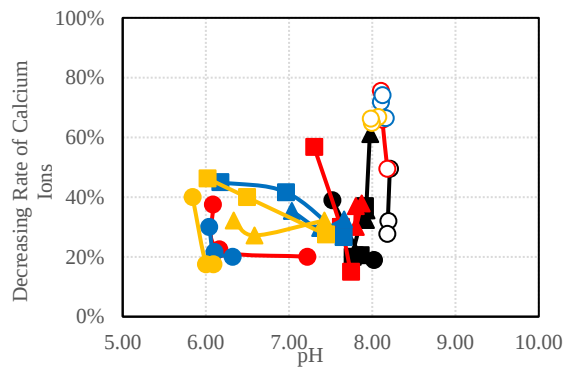


Fig. 16 Decreasing rate of calcium ions and pH (initial pH 8.0, temperature from 10-30 °C)

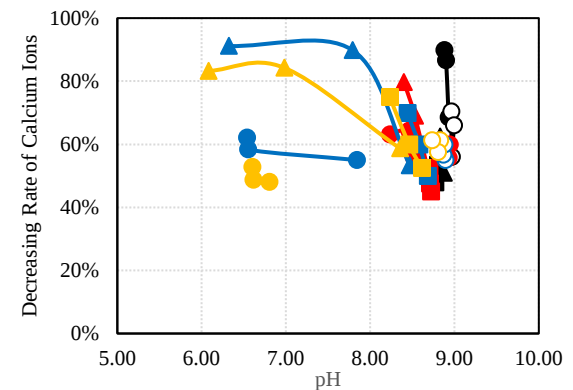


Fig. 17 Decreasing rate of calcium ions and pH (initial pH 9.0, temperature from 10-30 °C)

Otherwise, Fig. 11 shows the decreasing rate of calcium ions and pH for an initial pH adjusted to 9.0. It can be seen that, for the mixture with the highest concentration of Tris buffer solution (i.e. 1.0 mol/L) in each mixture, the changes of pH in the mixtures after 24 hours was marginal, except the mixture with a high concentration of yeast (B4 mixture). For the other concentrations of Tris buffer (0.50 and 0.75 mol/L), the pH drops rapidly for the B3 and B4 mixtures after 24 hours, reaching less than 6.0. The maximum precipitation of calcium carbonate was reached in the B2 and B3 mixtures, when adjusted to concentrations of 0.50-0.75 mol/L.

Similar experiments were conducted with controlled temperatures of 10 °C, 30 °C, and a fluctuating temperature range from 10-30 °C. Figure 12 to Fig. 17 show the decreasing rate of calcium ions and pH at different temperatures. These results confirm that the precipitation rate of calcium carbonate is affected by temperature. At low temperatures (10 °C), the decreasing rate of calcium ions reaches below 40% for up to 72 hours of elapsed time at an initial pH adjusted to 8.0. On the other hand, for the initial pH adjusted to 9.0, it was between 30 and 60% after 72 hours.

Moreover, at a controlled temperature of 30 °C, the decreasing rate of calcium ions was above 40% after 24 hours elapsed time, except for the A4 mixture. For the initial pH adjusted to 9.0, it was above 60% after 24 hours. Furthermore, at a fluctuating temperature (controlled from 30-10°C), the graphs show a similar trend, with a decreasing rate of calcium ions and pH in yeast-based mixtures placed at a temperature of 20 °C. In this study, the concentration of Tris buffer solution was found to be adequate in the ranges 0.50-0.75 mol/L, in the case of an initial pH adjusted to 9.0, but 0.75-1.00 mol/L, in the case of an initial pH adjusted to 8.0. This result was confirmed by the previous research [10]. It was found that the generation of carbonate ions is faster in the mixture with an initially adjusted pH of 9.0. The results show that calcium carbonate precipitates depend on the different composition of yeast-based mixtures within 72 hours after mixing. In this study, the concentrations of Tris buffer solution were found to be adequate in the range 0.50-0.75 mol/L, at an initial pH of 9.0, and 0.75-1.00 mol/L, at an initial pH of 8.0. In the case of mixtures with an initial pH of 9.0, the rate of initial reaction tends to be faster, which could be attributed to the lower concentration of the Tris buffer solution, adjusted to a relatively higher pH in the mixture. The highest precipitation was reached in the B3 mixture, at an initial pH adjusted to 9.0 and a Tris buffer solution adjusted to 0.75 mol/L. The concentration of dry yeast was specified as 27 g/L, which was mixed with glucose and calcium acetate with concentrations of 0.30 mol/L and 0.15 mol/L, respectively.

5. CONCLUSIONS

The results show the effects of changes in temperature on the precipitation rate of calcium carbonate. Based on the mass of precipitates left in the tubes after filtering, the maximum precipitation of CaCO₃ was observed at 30 °C and the lowest precipitation was observed at 10 °C. The results were confirmed by the calcium ion measurement in the mixtures. At 30 °C, after 24 hours of elapsed time, the concentration of calcium ions was decreased by more than 90%. On the other hand, the pH was gradually lowered below 7.0. In this research, 20–30 °C is the optimum temperature range for increasing the precipitation of calcium carbonate. The temperature rises to promote the formation of carbonate ions and the amount of calcium carbonate precipitated.

In addition, the results show that calcium carbonate precipitates depend on the different composition of yeast-based mixtures within 72 hours after mixing. In this study, the concentrations of Tris buffer solution were found to be adequate in the range of 0.50-0.75 mol/L, at an initial pH of 9.0, and 0.75-1.00 mol/L, at an initial pH of 8.0. In the case of mixtures mixed with an initial pH of 9.0, the rate of initial reaction tended to be faster, which could be attributed to the lower concentration of the Tris buffer solution, adjusted to a relatively higher pH in the mixture.

6. ACKNOWLEDGEMENTS

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