



Utilization of Soybeans as Bio-Catalyst in Calcite Precipitation Method for Repairing Cracks in Concrete

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Abstract. Concrete is a material that has high compressive strength. However, concrete has a lower tensile strength than its compressive strength. As a result, the concrete often cracks and allows the entry of harmful substances such as Cl^- dan SO_4^{2-} causing corrosion of the reinforcement. Therefore, the repair method began to shift from the conventional way to the concept of self-healing concrete which involves the deposition of $CaCO_3$. Precipitation can be done by the enzymatically – induced carbonate precipitation (EICP) method through a combination of urease, urea, and $CaCl_2$ into a solution. This research used soybean extract as a substitute for pure urease enzyme. Variations in the concentration of soybean flour used as injection solution were variations in the content of soybean flour 15 g/L because it produced an optimum calcite mass of 2.62 grams. As a result, there was an increase in the compressive strength of BI against BR. In addition, there was a decreased value of permeability and porosity and the number of injections carried out. The increase in compressive strength, decrease in permeability, and decrease in porosity in concrete is due to $CaCO_3$ deposition in the concrete which can cover the pores and cracks in the concrete.

Keywords: Concrete; $CaCO_3$; Compressive strength; Permeability; Porosity

INTRODUCTION

Concrete is a building material that has a high compressive strength [1]. However, concrete also has limitations such as tensile strength, which is lower than its compressive strength, which is only around 9%-15% of compressive force, so concrete is brittle. Therefore, cracks in the concrete structure cannot be avoided [2,3]. This matter can accelerate the corrosion of steel reinforcement and damage to the concrete, thereby shortening the life of the concrete [4]. In addition, hazardous substances such as Cl^- and SO_4^{2-} can enter through the cracks even if the damages are minor [5]. Cracks in concrete result in decreased strength and durability, and the cost of repairs is quite expensive. Even in the UK, maintenance, and repairs in the construction sector cost £40 billion [6].

Conventional repair methods began to shift along with the development of concrete technology with a self-healing function, which is expected to solve these problems so that the topic of self-healing has become popular in recent years [7]. "Self-healing concrete" refers to a material that can recover its original properties after being degraded. Concrete by itself has autogenous healing properties due to continuous mineral hydration and deposition [8].

Precipitation of calcium carbonate ($CaCO_3$) can be done by the enzyme – induced carbonate precipitation (EICP). The precipitation occurs due to the influence of enzyme induction so that carbonate ions can be formed. Yasuhara et al. combined urease, urea, and calcium chloride ($CaCl_2$) into a solution and proved to be able to precipitate $CaCO_3$ [9]. However, the use of urease in the EICP method turned out to be less economical due to the high price of urease, especially if its use on a large scale can reach >90% of

material costs [10]. Therefore, an alternative material that is environmentally friendly and inexpensive is needed to replace the urease enzyme.

Several plant families, including some varieties of legumes, melons, pumpkins, and pine, are affluent in the enzyme urease [11]. Zulfikar et al. proved that soybeans could be used as an additive in the catalyst process with urease. The use of soybeans is proven to have the capacity to reduce the permeability of concrete by 95.43% [12]. Soybean powder has the potential to replace the urease enzyme when viewed from previous studies so that based on these considerations, this study aims to determine the effect of soybean powder as a substitute for urease on compressive strength, permeability, and porosity in cracked concrete.

METHODOLOGY

Materials

The primary materials used in this study include gravel, cimangkok sand, portland composite cement (PCC), urea, calcium chloride dihydrate, and Gasol soybean flour. Aggregate property testing is carried out first to determine the characteristics of the aggregates used in the manufacture of concrete. Aggregate property tests include sieve analysis of fine aggregate and coarse aggregate, testing specific gravity and absorption of fine aggregate and coarse aggregate, loss Angeles testing, testing the moisture content of fine aggregate and coarse aggregate, and slurry content of fine aggregate and coarse aggregate.

Calcite Precipitation Test (test-tube)

The composition of the calcite precipitation solution was varied based on the concentration of soy flour and reagents, as shown in Table 1. The first step is the reagents (urea and CaCl_2) and soybean flour are prepared, then the soybean flour is mixed with distilled water while stirring with a magnetic stirrer for 5 minutes. The resulting mixture is then filtered through a sieve to separate the solution and soybean precipitate. Meanwhile, the reagent is mixed with distilled water until all the particles are dissolved. The reagent solution and soybean solution were mixed into a precipitation test tube with a volume of 30 mL and then left for seven days. After curing time, the solution was filtered through 20-25 m filter paper. The calcite mass formed can be measured and weighed after being dried in the oven for 24 hours at a temperature around 60 °C. The procedure detail can be seen in Figure 1.

TABLE. 1 Variations in the composition of calcite precipitation

Sample	Concentration		
	CaCl_2 (mol/L)	Urea (mol/L)	Soybean Flour (g/L)
A	1	1	1
B	1	1	5
C	1	1	10
D	1	1	15
E	1	1	20

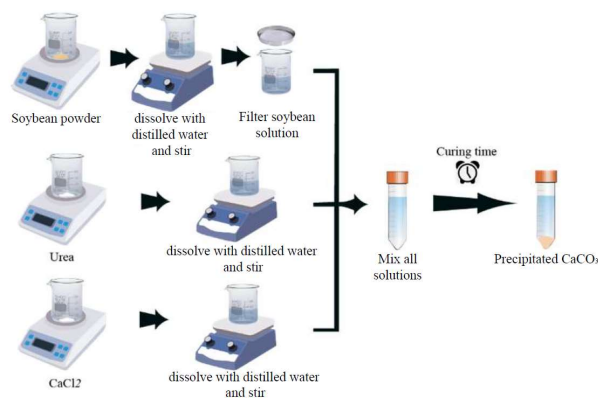


FIGURE. 1 Test-tube procedure

Concrete Sample Making

The manufacture of concrete samples is based on the circular letter of the minister of public works and public housing number: 07/SE/M/2016 concerning guidelines for determining standard concrete mixtures with OPC, PPC, and PCC cement. The mix design calculation is carried out before the concrete mixing process. The quality of the concrete planned in this study is K-250 concrete (20.75 MPa). The size of the mold is 5 cm x 15 cm with a shape like a cylindrical formwork, while the size of the concrete sample to be made 5 cm x 10 cm with a hole with a diameter of ± 0.5 cm is precisely 1 cm above the sample which serves as the outlet for water during the permeability test. The proportion of concrete mix from the mix design is shown in Table 2.

TABLE. 2 The proportion of the concrete mix

Volume	Mixed Proportion			
	Cement (kg)	Water (kg)	Fine aggregate (kg)	Coarse aggregate (kg)
Per m ³	376	233	543	1096
Per mold (0.0002 m ³)	0.075	0.047	0.109	0.219

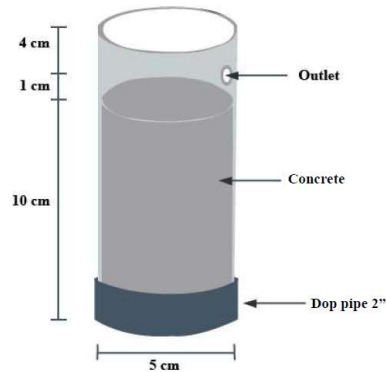


FIGURE. 2 Concrete sample mold 5 x 10 cm

Cracking and Curing of Concrete

Cracks in the concrete are made when the age of the concrete reaches 2-3 days. Cracks were made using a universal testing machine (UTM). The procedure for making cracks can be seen in Figure 3. The test objects are divided into 6 types, including intact concrete (BU), cracked concrete without injection (BR), and cracked concrete injected with calcite precipitation solution with n injections (BI1, BI2, BI3, BI4). The BU and BR samples were then cured for 28 days by immersing them in a tub filled with water. The treatment for injected cracked concrete (BI) is slightly different. That is, it is not fully immersed for 28 days because the sample needs to be injected.

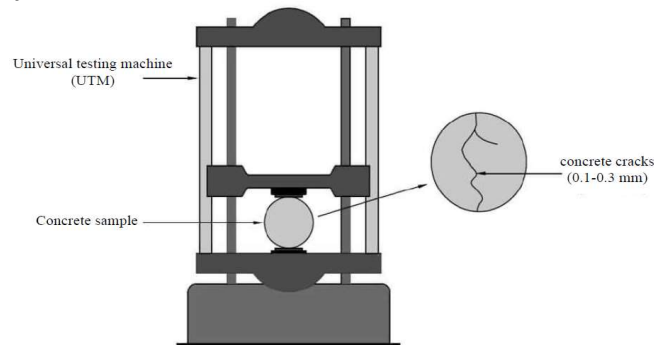


FIGURE 3. Illustration of cracking in concrete samples using UTM

Concrete Injection

Injection of calcite precipitation solution was carried out on injected cracked concrete samples (BI). The injection time was carried out for three days, referring to a similar study [12], then the concrete was again immersed in an immersion bath. The injections made at BI1, BI2, BI3, and BI4 are undoubtedly different. The injection plan in more detail can be seen in Table 3.

TABLE 3. Experimental conditions for injection of calcite precipitation

Sample Type	Injection amount	Injection order	Injection Scenario
BI1	1x	1	at the age of concrete 5 days
BI2	2x	1	at the age of concrete 19 days
		2	at the age of concrete 25 days
BI3	3x	1	at the age of concrete 12 days
		2	at the age of concrete 19 days
		3	at the age of concrete 25 days
BI4	4x	1	at the age of concrete 5 days
		2	at the age of concrete 12 days
		3	at the age of concrete 19 days
		4	at the age of concrete 25 days

Concrete Compressive Strength Test

The compressive strength test refers to SNI-03-1974-2011 on how to test the compressive strength of concrete with cylindrical specimens. Tests were carried out with the Universal Testing Machine (UTM) on 28 days old concrete samples with a total of 12 samples consisting of intact concrete (BU), cracked concrete without injection (BR), and cracked concrete with a certain number of injections (BI1, BI2, BI3, BI4).

Concrete Porosity and Permeability Testing

Porosity testing refers to ASTM C-642-97. Porosity can describe the density of concrete and is defined as the pore content contained in concrete [25]. The number of samples consisted of 12 samples and was measured at 28 days of concrete age. Porosity and permeability tests are interconnected because the tests are carried out at the same time. Permeability testing was carried out on 28 days old concrete samples using the flow test method, and the goal is to determine the percentage of water passing in the concrete. A total of 12 samples were tested for permeability using a simple tool. Then, the permeability test procedure is carried out by utilizing the force of gravity. A more detailed illustration of the permeability test can be found in Figure 4.



FIGURE 4. Schematic of testing the permeability of concrete.

RESULT AND DISCUSSION

Aggregate Properties

Concrete is planned with the quality of K-250, which is included in class III concrete and is intended for structural activities [26]. Determination of the K-250's quality is based on its use for reinforced concrete which is medium quality with K250-<K400's quality [27]. The planned slump value is 10±2 cm, based on the 1971 Indonesian reinforced concrete regulations, this value meets the slump requirements for columns, foundations, beams, and slabs, which is 5-15 cm. The results of testing the aggregate properties can be seen in Table 4.

The specific gravity of the aggregate used is the bulk specific gravity in the SSD (saturated surface dry) state. The specific gravity of the coarse aggregate does not meet the standard specifications. Aggregates with a specific gravity between 2.5-2.7 g/cm³ produce a concrete density of about 2.3 g/cm³ [31]. Aggregates with high specific gravity will produce concrete with high specific gravity and high compressive strength. The absorption of coarse aggregate exceeds the standard specifications, it affects the water requirement for the concrete mixing process. The moisture content of the tested aggregates, both coarse and fine aggregates, did not meet the standard specifications, this was due to the effect of drying the aggregates for too long so that the water content in the aggregates was small.

TABLE 4. Results of testing aggregate properties

No	Type of examination	Result	Standard specification	Reference
1	The specific gravity of fine aggregate			
	a. Bulk specific gravity (JKP)	2.60	2.58-2.83	
	b. Absorption	2.22	2-7%	
	c. Fineness modulus	2.13	1.5-3.8	
2	d. Sludge levels	1.15	<5%	ASTM C 33 1986
	The specific gravity of coarse aggregate			SK SNI S-04-1989-F
	a. Bulk specific gravity (JKP)	2.48	2.58-2.83	SNI 1969 2008
	b. Absorption	3.15	<3	SNI 1970 2008
	c. Fineness modulus	6.65	6-7.1	
3	d. Sludge levels	0.13	<1%	
	Water content			
	a. Fine aggregate	2.67	3-5%	
	b. Coarse aggregate	0.87	3-5%	

Calcite Deposition Test (Test-tube)

The calcite precipitation method is used as an alternative in repairing concrete cracks. The use of enzymes in calcite precipitation comes from soybean flour. Soybean flour acts as a catalyst in the calcite precipitation method, which significantly affects the amount of calcite deposition. Therefore, calcite deposition was evaluated directly by the test-tube method. The test-tube test results can be seen in Figure 5.

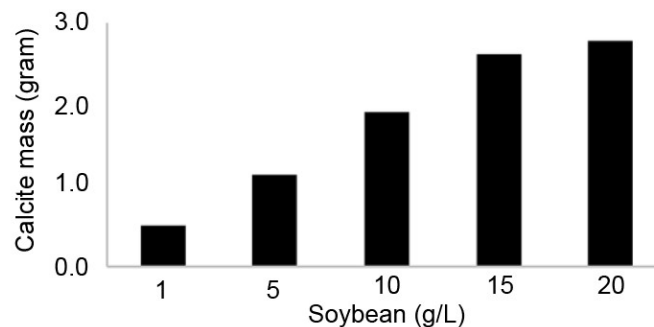


FIGURE 5. Graph of test-tube test results

Based on Figure 5 on the graph of the tube test results, it can be seen that the higher the soybean content, the higher the calcite mass formed. The precipitation ratio increased with the addition of the concentration of soy flour used. The addition of soy flour concentration from 1 g/L to 20 g/L showed an increase in the calcite mass deposition ratio of 4.56 times. This increase showed an increase in the mass of calcite formed and an increase in the content of undissolved soybeans. This undissolved soybean content can inhibit the injection of calcite precipitation solution in concrete because the sediment formed can inhibit the permeation of the solution in the concrete. The difference in the mass of calcite produced between the levels of soybean flour 10 g/L and 15 g/L was 0.71 grams, while the difference in the mass of calcite produced between the levels of soybean flour 15 g/L and 20 g/L was 0.16 grams. However, increasing the concentration of soy flour from 15 g/L to 20 g/L showed the smallest increase in precipitation ratio of 6.1%, so that the concentration of 20 g/L soybean flour was not used for injection, unlike the case with the increase in the precipitation ratio from a concentration of 10 g/L to 15 g/L, which experienced a relatively large increase in the precipitation ratio of 71.2%. The mass of calcite formed from a concentration of 15 g/L is also large enough so that the concentration of 15 g/L is the optimum variety of calcite precipitation solutions.

Concrete Compressive Strength

The compressive strength test was carried out on a 28-day-old K-250 concrete sample. BU and BR behaved as controls, while BI was the test result parameter due to the influence of the test tube solution. The results of the 28-day-old concrete compressive strength test for all samples can be seen in Figure 6.

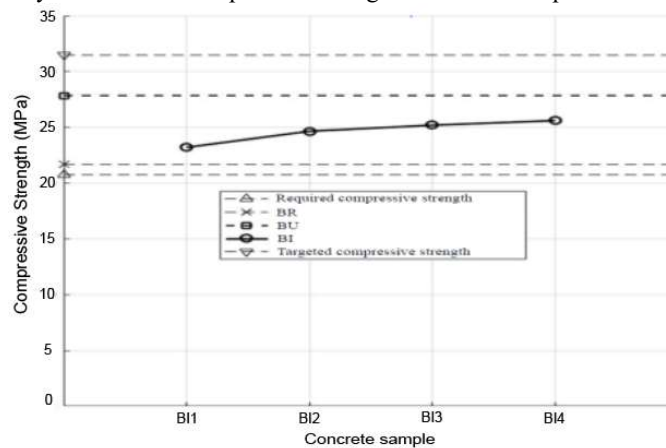


FIGURE 6. Compressive strength test results

Based on the graph of the results of the compressive strength test above, all concrete samples meet the required compressive strength of 20.75 MPa based on the mix design with the circular letter of the minister of public works and public housing number: 07/SE/M/2016, even BR which is the lowest value which is 21.66 MPa while BU 27.85 MPa acts as a control and is the sample with the highest compressive strength value. The compressive strength of cracked concrete decreased by 28.54% from that of intact concrete. This shows the effect of cracking on the compressive strength of concrete. The width of the crack affects the decrease in compressive strength that occurs, the reduction in compressive strength can reach 80% with the increase in the width of the damage that occurs in the concrete [32]. The compressive strength test was carried out at the age of 28 days of concrete. The compressive strength that occurs after the age of 28 days of concrete has not experienced a significant increase so that the age of 28 days of concrete is often used as a standard in testing concrete [33].

Repair of cracks in concrete using calcite precipitation solution is proven to increase the compressive strength of concrete. The compressive strength values at BI1, BI2, BI3, and BI4 are 23.20 MPa, 24.65 MPa, 25.18 MPa, 25.60 MPa, respectively. This value is above the compressive strength of BR. Based on the graph with the addition of the solution once, the compressive strength of the concrete increased by 7.07% of the BR strength. The compressive strength of BI2, BI3, and BI4 also increased after adding the test tube result solution with an increase in the compressive strength of BR, which were 13.76%, 16.24%, and 18.15%, respectively. Calcite precipitation solution has a viscosity that is almost like water, this solution will enter through the cracks and begin to occupy the pores in the concrete matrix. The formation of calcite in the pores then makes the pores filled with calcite, thereby reducing the pores in the concrete and increasing its compressive strength [34]. Another study also reported that the increase in strength that occurs is due to the deposition of CaCO₃ in the pores and cracks that occur in the concrete [35,36]. Deposit in the pores of the concrete causes the concrete to become denser to increase its compressive strength [37].

Permeability and Porosity Testing of Concrete

Permeability is one of the main factors causing damage and decreased resistance [38]. Permeability testing is carried out in conjunction with porosity. The porosity test only uses mass parameters, including oven-dry mass, surface saturated mass, and mass in water. The permeability value can show the density of a material, while the porosity value describes concrete density. Permeability and porosity have a direct relationship with the compressive strength of concrete. Increased permeability and pore size in concrete can cause a decrease in compressive strength and vice versa. The data from the permeability and porosity test results can be seen in Figure 7.

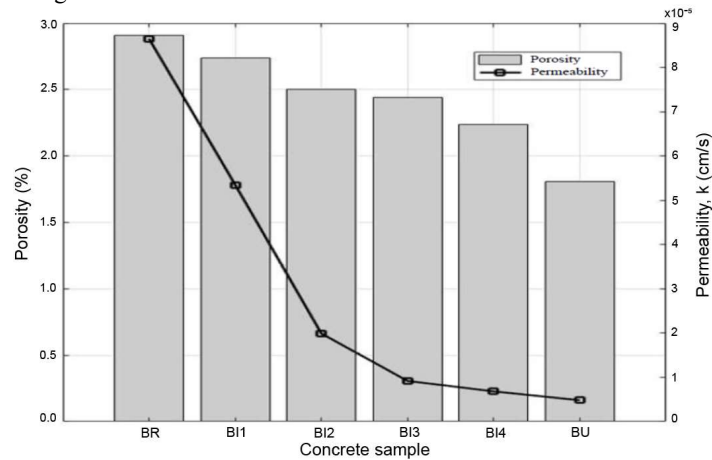


FIGURE 7. The relationship between permeability and porosity

Based on Figure 7 regarding the relationship between permeability and porosity, the permeability value is depicted by a line graph while a bar graph represents the porosity value. The greatest permeability value is found in BR of 8.65×10^{-5} cm/s. This means that the BR is easier for water to pass because of the cracks in the concrete. The presence of cracks that occur in the concrete causes the density of the concrete to decrease, this can allow harmful substances to flow in the concrete matrix and reduce the resistance of the concrete [4]. Cracked concrete (BR) has increased permeability up to 94.4% of intact concrete, which is 4.81×10^{-6} cm/s. However, concrete consists of aggregates that have a lower permeability value than other materials in the mixture, due to the micro-cracks that occur in the zone between the cement paste and the aggregate, the permeability increases. These microcracks are larger than the capillary pores and microcrack propagation will develop interconnections that increase the permeability of the concrete [39]. There was a decrease from BI1 to BI4 successively by 5.35×10^{-5} cm/s, 1.99×10^{-5} cm/s, 9.17×10^{-6} cm/s, 6.82×10^{-6} cm/s. The decrease in permeability occurs along with the increase in the number of injections made in the concrete. It happens because the injection of calcite precipitation solution can precipitate calcium carbonate or calcite crystals that cover the cracks and make the pores in the concrete matrix tighter [34]. Pore size also affects permeability in the presence of calcite precipitation solution, the pore size in concrete becomes smaller than in BR, which is not injected with calcite precipitation solution. Along with the increase in the number of injections, the permeability decreases, this is because the pores of the concrete are getting clogged so that the fluid is increasingly difficult to flow in the concrete matrix [40].

The results of the porosity test can also support the results of the permeability. The porosity value in BR is 2.91%, while the BU is 1.81%. The greatest porosity value is found in BR. The cracks that occur cause the compressive strength of the concrete to decrease and increase the porosity or pore size of the concrete [41]. A higher porosity value in BR indicates that BR easily absorbs water than BU, which has a small porosity value. The value of porosity and permeability can measure concrete durability. BU with no cracks in the concrete matrix makes BU more impermeable and its water resistance is higher than BR. Based on Figure 7, there is a decrease in the BR porosity value when injected with calcite precipitation solution. The porosity values in BI1, BI2, BI3, and BI4 were 2.74%, 2.50%, 2.44%, and 2.24%, respectively. The decreasing porosity of the concrete is in line with the decreasing permeability of the concrete. This is due to the deposition of CaCO_3 in the concrete so that it forces pores of the concrete to become denser [42]. The porosity and permeability values formed after injection with calcite precipitation solution decreased and approached the BU value. Therefore, BU indicates that the concrete is impermeable and tight. Likewise, BI is getting tighter and tighter after injection. This shows that the calcite precipitation solution is proven to deposit calcite which functions to close the cracks and pores in the concrete.

CONCLUSION

Based on the research that has been done, it can be concluded that:

1. Variations in soybean flour concentration of 15 g/L produced optimum calcite mass, which was then used in calcite precipitation solution to be injected into concrete.
2. The increase in compressive strength occurs in cracked concrete injected with calcite precipitation solution. As the number of injections increases, the compressive strength also increases, but the increase will be smaller because the calcite formed in the previous injection will inhibit the solution from entering through the crack gap.
3. There is a decrease in the value of permeability and the addition of the amount of solution injected. The reduction in permeability in the injected concrete indicates that the calcite precipitation solution can precipitate CaCO_3 to close the cracks that occur
4. The porosity value is reduced in the injected cracked concrete. This shows that the pore size of the concrete is getting smaller because the calcite deposits are increasingly pressing the pores and causing the concrete to be denser and more impenetrable.

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