The Effect of Sulfate Exposure on The Mechanical Properties of Conventional Portland Composite Cement Concrete

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Abstract

The durability of concrete is an essential factor in construction as a means to enhance the quality of public facilities planning. The durability of concrete structures can be degraded due to several factors, one of the factors is sulfate attack concrete. Expose to magnesium sulfate (MgSO4) causes more aggressive damage compared to sodium sulfate. The negative effect of sulfate exposure is the accelerated formation of microcracks that lead to concrete deterioration. This study aims to investigate the influence of expose to a 5% magnesium sulfate solution on the mechanical properties of Portland composite cement concrete (PCC). Sulfate exposure was conducted after the PCC concrete was cured in water for 28 days. Observations and testing were carried out in four stages: before exposure, after 28, 56, and 90 days. The compressive strength of the concrete increased by 8.78% after 56 days of exposure but decreased by 7.2% from the 56-day strength during the 90-day exposure. The modulus of elasticity, Poisson's ratio, tensile strength, and fracture energy were directly proportional to the compressive strength values generated during sulfate exposure. Additionally, the mass of the concrete increased by 0.87% after 56 days of exposure and then decreased to 0.56% during the 90-day exposure..

Keywords

Portland composite cement, concrete, magnesium sulfate attack, mechanical properties, durability

INTRODUCTION

Concrete is widely used as a construction material for infrastructure [1, 2]. The constituents of concrete generally include cement, water, coarse aggregates, and fine aggregates, involving chemical and mechanical properties [3]. Currently, most infrastructure development employs concrete based on Portland Composite Cement (PCC) due to the availability of easily obtainable materials. Concrete plays a crucial role in determining the lifespan and strength of a building. The lifespan and strength of a structure are influenced by various factors, one of which is the durability of the materials [4]. However, the durability of materials can experience degradation due to several factors, including sulfate attack. A sulfate attack is a chemical deterioration process that occurs between hydrated cement compounds (alumina and calcium hydroxide) and sulfate ions (SO) [5]. SNI-2847:2013 [6] require a minimum concrete quality for environments with high sulfate exposure is 31 MPa. This is intended to mitigate concrete damage caused by sulfate exposure.

Research conducted by Maes [7, 8] on the resistance of concrete to sulfate exposure using ordinary portland cement, High Sulfate Resistant (HSR) cement, and Blast Furnace Slag indicated that chloride penetration increases with the increasing sulfate content in the concrete environment during short immersion periods. Another study by [1] stated that concrete made with Portland cement immersed in magnesium and sodium sulfate for three months forms a softening layer on the concrete surface. A sulfate attack is a reaction between sulfates and hydrated calcium hydroxide in cement, forming calcium sulfate and calcium aluminate hydrate, which then becomes calcium sulfoaluminate [9]. Exposure to sulfate solutions has a drawback, as it leads to the formation of microcracks in concrete accompanied by the formation of ettringite due to magnesium sulfate, which accelerates the process of concrete deterioration [10]. The formation of ettringite and gypsum is triggered by a high level of concrete porosity; high porosity has significant potential for the diffusion process in concrete exposed to magnesium sulfate solution. Diffusion is one of the mechanisms through which magnesium sulfate solution enters the concrete [11]. Ordinary portland cement, commonly used as a binder in conventional concrete, has a potential global warming impact of 0.66-0.82 kg CO2 emitted per production [12]. This research aims to investigate the influence of expose to a 5% magnesium sulfate solution on the mechanical properties of PCC concrete.

RESEARCH SIGNIFICANCE

This study examined the effect of expose to a 5% magnesium sulfate solution on the mechanical properties of PCC concrete, including compressive strength, elastic modulus, Poisson's ratio, split tensile strength, and fracture energy. In addition, changes in weight, solution pH, and chemical composition before and after exposure were also observed in this study.

METHODOLOGY

A. MATERIALS

The materials used in this research include cement, water, coarse aggregates, and fine aggregates. The cement used is Portland Composite Cement Type V, because it is a type of cement that is suitable for various concrete applications where good resistance to high sulfate levels is required. The fine aggregate used is natural sand containing no more than 5% silt, which does not harm the concrete. The sand used in this study is local sand, classified as Zone 3 based on sieving analysis. The coarse aggregate is hard and non-porous natural crushed stone particles containing less than 1% silt. The crushed stone used has a maximum size of 20 mm. Concrete with a maximum aggregate size of 20 mm exhibits better compressive strength compared to concrete utilizing maximum aggregate sizes of 40 mm and 10 mm [13].

B. PREPARATION OF SPECIMENS

Specimens used in this study consist of two types, which is 100 mm x 200 mm cylinder and 150 mm x 150 mm x 550 mm notched beam. Cylinders specimens are used for compressive strength, elastic modulus, Poisson's ratio, and split tensile strength test while, the notched beam are used to specimen fracture energy. The composition of PCC concrete shown in Table 1.

Table 1 Mixture Proportions of PCC Concrete

w/c	Mixture proportion (kg/m^3)			
	Water	Cement	Sand	Aggregate
0,39	174,15	520,34	577,16	1156,51

After casting, the specimens were initially cured in water immersion for 28 days. Subsequently, the concrete was immersed in a magnesium sulfate solution with a 5% [14] for 28, 56, and 90 days. During the exposure period, the acidity level of the solution was periodically monitored every seven days through pH measurements. The initial pH value of the solution should fall within the range of 6-8 [14]. This research investigated the pH values of the solution, changes in specimen mass, chemical composition within the specimens, compressive strength, modulus of elasticity, Poisson's ratio, tensile splitting strength, and fracture energy.

C. CONCRETE TESTING

1. Compressive Strength, Elastic Modulus, and Poisson’s Rasio

Compressive strength testing is based on ASTM C 39 [15], The calculation formula for the compressive strength of concrete is as follows.

$$fc' = \frac{P}{A} \quad (1)$$

Where:
f’c = Compressive Strength (MPa)
P = Axial Compressive Force (N)
A = The cross-sectional area of the concrete (mm^2)

Standard deviation was also calculated in this study to assess the level of consistency in concrete quality. The calculation use formula is as follows.

$$S = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}} \quad (2)$$

Where:
x_i = value data i-th
$\bar{x}$ = mean
n = number of specimens

The modulus of elasticity test was performed acording to ASTM C469 M-14 [16]. The calculation formula for the elastic modulus of concrete is as follows.

$$E = \frac{(S_2 - s_1)}{(\varepsilon_2 - \varepsilon_1)} \quad (3)$$

Where:
E = Elastic modulus (MPa)
S_1 = Initial stress equivalent to 0,5 MPa
S_2 = Stress when 40% ultimate load (MPa)
ε_1 = Strain when initial stress (0,5 MPa)
ε_2 = Strain when stress reach 40% ultimate load

The poisson’s ratio test was performed based on ASTM C469 M-14 [16]. The calculation formula for the poisson’s ratio of concrete is as follows.

$$\mu = \frac{(\varepsilon_{t2} - \varepsilon_{t1})}{(\varepsilon_2 - \varepsilon_1)} \quad (4)$$

Where:
μ = Poisson’s Ratio
ε_{t1} = Transversal strain when initian stress
ε_{t2} = Transversal strain when stress reach 40% ultimate load
ε_1 = Strain when initial stress (0,5 MPa)
ε_2 = Strain when stress reach 40% ultimate load

Figure 1 Testing setup of compressive strength, elastic modulus, and poisson’s rasio

2. Split Tensile Strength

Compressive strength testing is based on ASTM C469-11 [17]. The calculation formula for the split tensile strength of concrete is as follows.

$$T = \frac{2P}{\pi l d} \quad (5)$$

Where:
T = Split tensile strength (MPa)

P = Axial force (N)
L = High of specimen (mm)
d = Diameter of specimen (mm)

Figure 2 Testing setup of split tensile strength

3. Fracture Energy

This testing was conducted using the three-point bending method on a beam specimen measuring 150x150x550 mm with a 25 mm notch, following the standard procedure. Based on JCIS [18]. The calculation formula for the fracture energy of concrete is as follows.

$$GF = \frac{0.75W0 + W_1}{A_{lig}} \quad (6)$$

$$W1 = 0.75\left(\frac{S}{L}\, m1 + 2m_2\right) g.\, CMOD_c \quad (7)$$

Where:
G_F = Fracture energy (N/mm)
W_1 = work done by deadweight of specimen and loading jig (Nmm)
A_{lig} = Area of broken ligament (b x h) (mm^2)
m_1 = Mass of specimen (kg)
S = Loading span (mm)
L = Total length of specimen (mm)
M_2 = Mass of jig not attached to testing machine but placed on specimen until rupture
g = Gravitational acceleration (9,807 m/s^2)
$CMOD_c$ = crack mouth opening displacement at the time of rupture (mm)
W_0 = Area below CMOD curve up to rupture of specimen (Nmm)

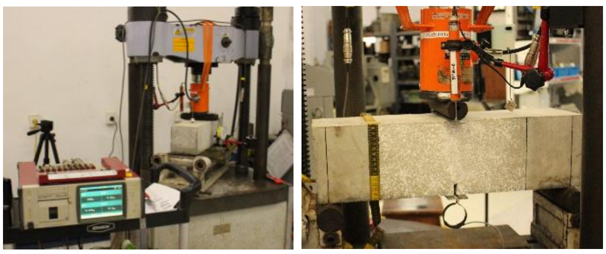


Figure 3 Testing setup of fracture energy

RESULTS AND DISCUSSIONS

A. VISUAL INVESTIGATION

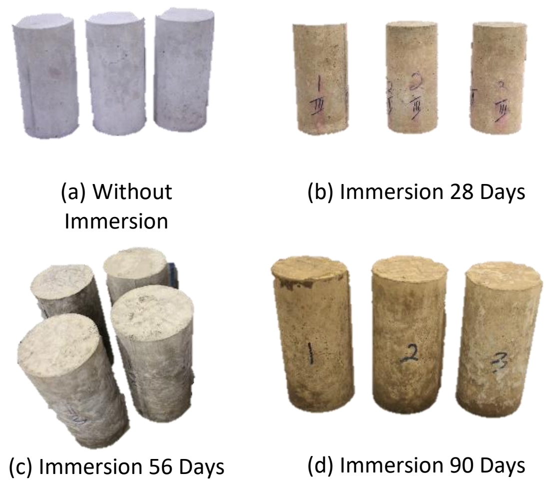


Figure 4 Appearance of concrete specimens immersed in solution of 5% MgSO4 for 90 days

Figure 4 shows the formation of white crystals on the concrete after immersion in the magnesium sulfate solution for 28, 56, and 90 days. The color change is influenced by the reaction between the concrete and oxygen, causing the magnesium sulfate solution to become saturated and form crystals. This condition in line with the result conducted by Maes [7]. Visual signs of concrete damage are not apparent during the 28–56-day exposure period, but surface cracking begins to occur after 90 days of exposure. This is influenced by the formation of gypsum and ettringite within the concrete pores, which have expansive properties and result in cracking on the concrete surface [19, 20].

B. MASS CHANGE

Figure 5 shows an increase in the weight of the test specimens after sulfate exposure. The most significant increase in weight occurs at a soaking age of 56 days, reaching 0.87%. This weight increase is influenced by the migration and diffusion processes between the concrete and sulfate, resulting in the formation of gypsum and ettringite reactions that fill the voids in the concrete. [21–23]. Gypsum and ettringite are expansive products, and with further development, cracks leading to the spalling of the concrete surface that can result in a decrease in mass, as indicated after concrete specimens are immersed in 90 days (Figure 5).

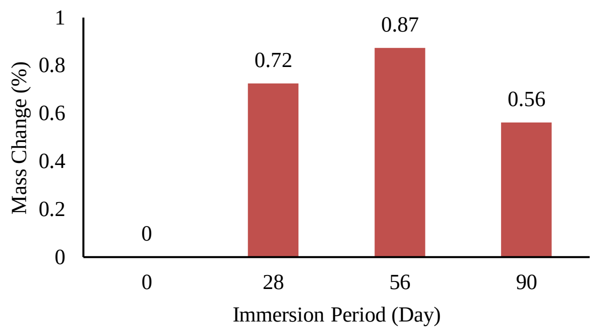


Figure 5 Mass change of concrete specimens immersed in solution of 50-gram $MgSO_4$ in 900 mL water for 90 days

C. pH OF IMMERSION SOLUTION

The hydrogen potential (pH) measurements aimed to determine the changes in the acidity level of the solution. The initial pH value before immersing the test specimens was 6.3. This result complies with the required range [14], as the pH value of the magnesium sulfate solution with 50 grams in 900 mL of water is considered acceptable when within the range of 6-8.

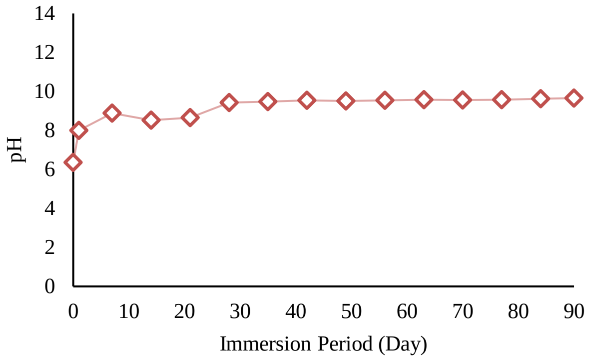


Figure 6 Change in pH of the solution versus time

Figure 6 illustrates the increase in the pH values of the solution during the 28, 56, and 90-day exposures, measuring 9.42, 9.55, and 9.66, respectively. This is influenced by the presence of silica (Si) in the concrete, which undergoes leaching when immersed in the sulfate solution, resulting in an alkaline nature, especially during the initial stages of immersion [24, 25].

D. CONCENTRATION OF SOLUTION

Testing the sulfate content in a 5% $MgSO_4$ solution was conducted by collecting samples of the solution that had been in contact with PCC concrete for 1, 28, 56, and 90 days. As a control, samples of the solution were also taken before being in contact with the concrete. Subsequently, the samples were analyzed using the gravimetric method. Figure 7 shows that the solution's sulfate content decreased during exposure. Initially, before immersion, the sulfate content was 2.64%. After 1 day of immersion, the sulfate content decreased by 3%. After 28 days of immersion, the sulfate content decreased by 69%. This indicates a significant reduction in sulfate content from 1-day exposure to 28-day exposure. However, there was almost no further decrease in sulfate content from the 28-day exposure to the 90-day exposure.

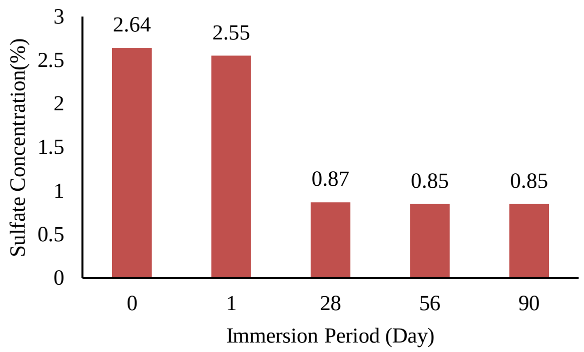


Figure 7 Sulfate concentration of solution

E. SCANNING ELECTRON MICROSCOPY-ENERGY DISPERSIVE X-RAY (SEM-EDX)

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) analyses were conducted to identify ettringite and gypsum through microstructural analysis. The SEM analysis showed that the PCC concrete exhibited relatively large but unevenly distributed pores. Based on the porosity test acording to [26], porosity value of the PCC concrete was measured at 10%. Figure 8 also illustrates the presence of formed gypsum and ettringite and the occurrence of cracks in the PCC concrete after 90 days of sulfate exposure. This indicates that the expansive gypsum and ettringite can exert pressure on the concrete, leading to crack formation.

Additionally, the microstructural observations through EDX in Table 2 revealed the absence of the sulfur (S) element after a 90-day sulfate exposure. This finding aligns with previous research conducted by [22], which also reported the absence of the sulfur element within a 3-month sulfate exposure. Another study demonstrated that the sulfur element began to appear in OPC concrete at a rate of 6.81% after 18 months of exposure [27].

Table 2 EDX result test (Weght %)

Immersion Period (Day)	Calcium (Ca)	Magnesium (Mg)
0	23.73	10.19
90	9.22	0.52

Table 2 shows the Calcium (Ca) element reduction after 90 days of sulfate exposure. This result is influenced by breaking the C-S-H bonds when reacting with the sulfate element, resulting in the loss of the Calcium (Ca) element. Consequently, this can potentially lead to a decrease in the mechanical characteristics of PCC concrete as the sulfate exposure time increases.

F. X-RAY FLUORESCENCE (XRF)

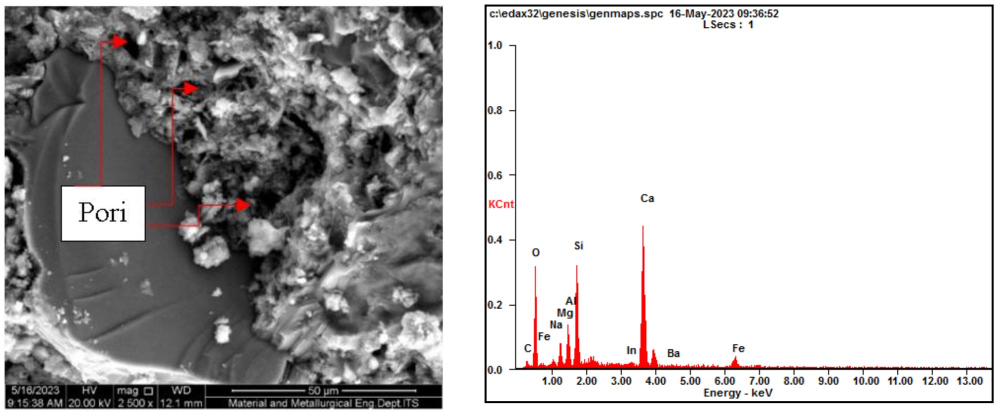
Table 3 Sulfate composition result test by XRF Analyses

Immersion Period (Day)	Compound	Oxide 1 (%)	Oxide 2 (%)	Mean (%)
0	SO_3	0.97	1	0.99
90	SO_3	1.3	1.2	1.25

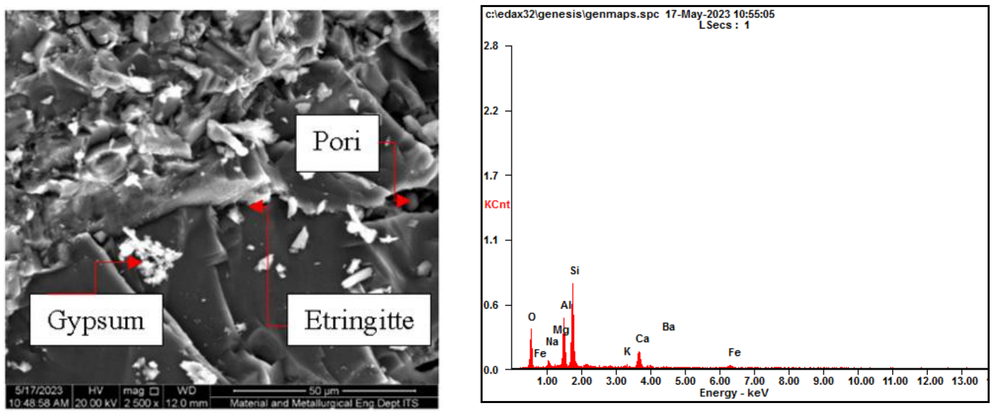
Table 3 illustrates a 27% increase in the SO3 compound in the concrete after a 90-day sulfate exposure. This is attributed to pores in the concrete, allowing the sulfate from the 50 grams of MgSO4 in 900 mL of water solution to infiltrate the concrete during the exposure period. The infiltrated sulfate reacts and can form ettringite and gypsum.

G. COMPRESSIVE STRENGHT

This test aims to determine the characteristics of concrete before and after sulfate exposure. The compressive strength at 28 days reaches 32.57 MPa, which meets the SNI 2847-2019 standard for sulfate exposure classification of class S3, which requires a minimum of 31 MPa.



(a) Before Immersion



(b) After Immersion 90 Days

Figure 8 SEM-EDX test result before and after immersion

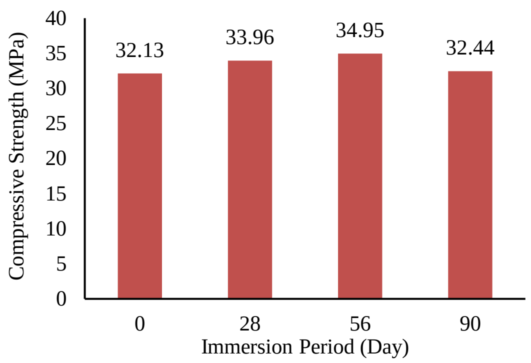
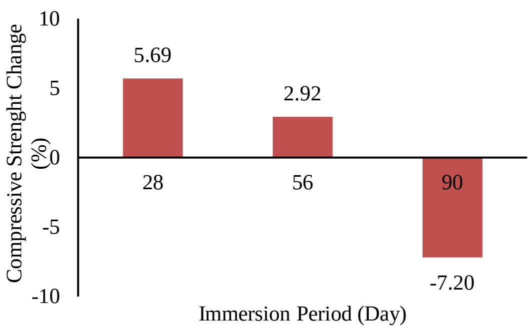
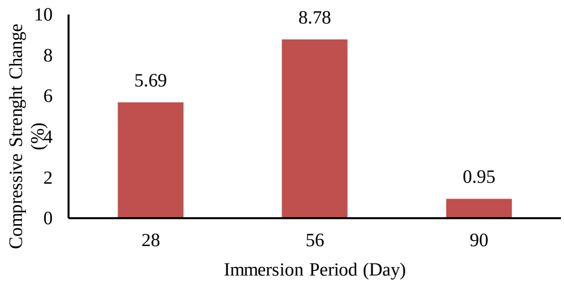


Figure 9 Compressive strenght test result



(a)



(b)

Figure 10 (a) Compressive Strength Change compared to Previous Age; (b) Compressive Strength Change compared to Initial Age

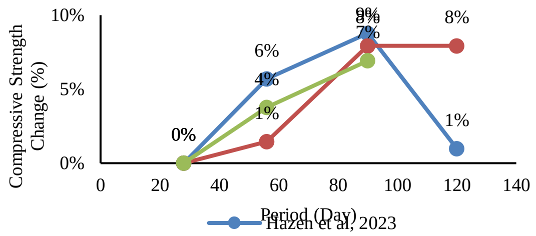


Figure 11 Compressive Strength Change compared to Previous Study with Non-immersed Samples

Figure 10 shows that the compressive strength of concrete increases by 5.69% after 28 days of exposure, then after 56 days of exposure, it only increases by 2.92% compared to the compressive strength after 28 days, which is an overall increase of 8.78% from the compressive strength of concrete before sulfate exposure. After 90 days of exposure, the compressive strength of concrete decreases by 7.20% compared to the compressive strength after 56 days, which is a decrease of 0.95% from the compressive strength of concrete before sulfate exposure. The results of this study are similar to a previous study conducted by [20], where the compressive strength of OPC concrete increased by 4.22% after 56 days of sulfate exposure and decreased by 2.36% after 90 days. However, when compared to concrete without sulfate exposure at the same concrete age, differences are evident, as presented in Figure 11. Figure 11 illustrates the changes in compressive strength of concrete at the ages of 56, 90, and 120 days compare to the concrete age of 28 days. The study by [28] indicates just 1% increase in compressive strength at the age of 56 days, followed by an 8% increase at the age of 90 days, with no change in compressive strength at the age of 120 days. Besides that, the study by [29] also demonstrates similar findings, where the compressive strength of concrete increases by 4% at the age of 56 days and 7% at the age of 90 days. This suggests that the formation of ettringite and gypsum in the concrete pores when exposed to magnesium sulfate can increase the density and mechanical strength of concrete in the initial stages of exposure [30]. In addition, as the exposure time increases, the pressure from the pore walls exceeds the tensile strength of the concrete, resulting in the formation of microcracks that can reduce the compressive strength, this is indicated by the slowing increase in the compressive strength of concrete at the age of 90 days (56 days of sulfate exposure) and the decrease in compressive strength at the age of 120 days (90 days of sulfate exposure) in this study, as shown in Figure 11. The compressive strength test results indicate a standard deviation of 1.19 to 1.30. These results are below the maximum limit of 1.4, categorized as excellent for concrete with a compressive strength ≤ 35 MPa [31].

H. ELASTIC MODULUS

The modulus of elasticity testing aims to determine the resistance of concrete to elastic deformation when a force is applied to it.

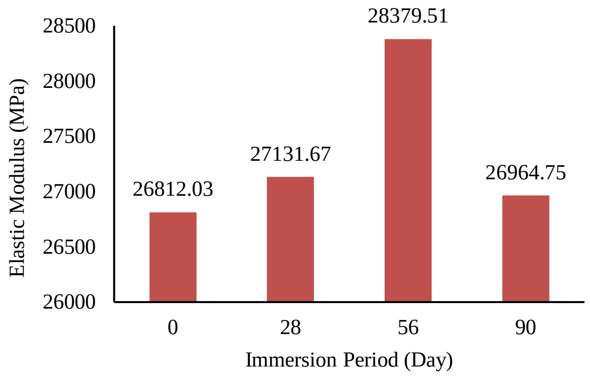


Figure 12 Elastic modulus test result

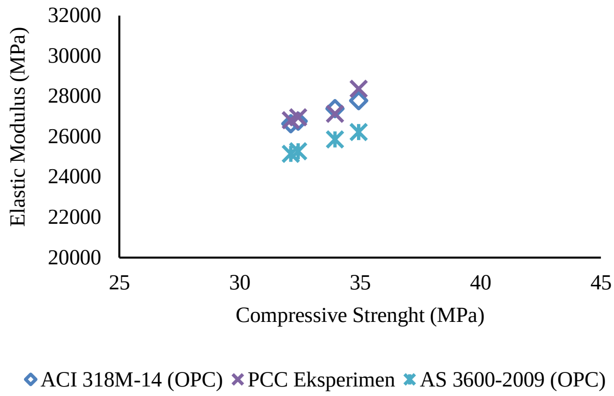


Figure 13 The relationship between compressive Strength and elastic modulus of PCC Concrete

Figure shows the values of the PCC concrete's elasticity modulus increasing until the 56-day exposure, then decreasing at the 90-day exposure. This trend is consistent with the changes in compressive strength during exposure to magnesium sulfate. Although exposure to magnesium sulfate influences the compressive strength and modulus of elasticity of PCC concrete, the relationship between compressive strength and modulus of elasticity of concrete (Figure) follows a similar trend as described by the formula [32].

I. POISSON'S RATIO

Table 4 Poisson's ratio result test

Without immersion	Immersion 28 days	Immersion 56 days	Immersion 90 days
0.2	0.22	0.2	0.19

The values of Poisson's ratio were determined by comparing the horizontal and vertical strains. Table 4 presents the experimental values of Poisson's ratio for PCC concrete, which fall within the range of 0.19-0.22. The Poisson's ratio values obtained in this study are in close agreement with the standard Poisson's ratio value of 0.2 for OPC concrete, as specified in AS 3600-2009 [33].

J. SPLIT TENSILE STRENGHT

The purpose of this test is to determine the ability of concrete to resist cracking due to external loads before and after sulfae exposure.

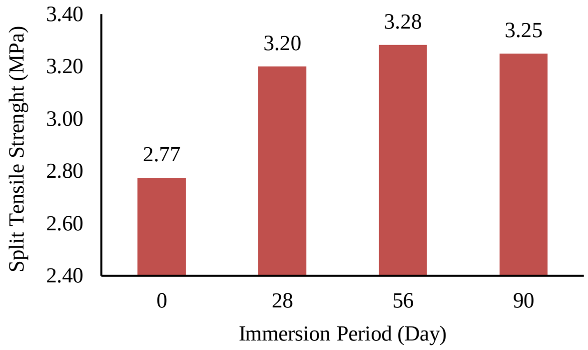


Figure 9 Split tensile strenght test result

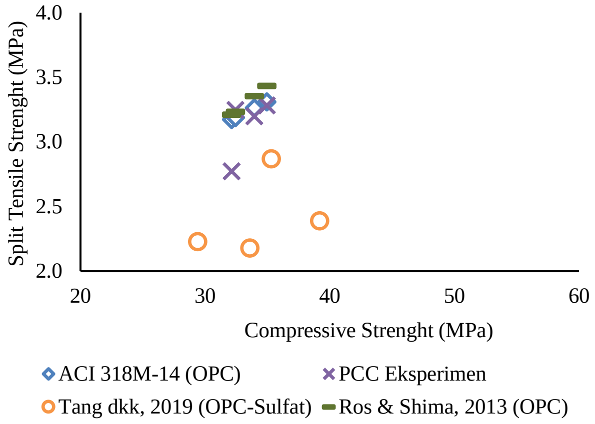


Figure 10 The relationship between compressive strength and split tensile strenght of PCC Concrete

Figure 9 demonstrates the increase in the splitting tensile strength of PCC concrete until 56 days of exposure, followed by a decrease at 90 days of exposure. This behavior corresponds to the changes in compressive strength during exposure to magnesium sulfate. These findings are consistent with previous studies by Bashandy dan Ros [20, 34] that investigated the effect of sulfate solution exposure on the relationship between splitting tensile strength and compressive strength of concrete, revealing a direct proportionality between these two properties. Despite the changes in compressive strength and splitting tensile strength of PCC concrete caused by magnesium sulfate exposure, an analysis of the relationship between compressive strength and splitting tensile strength (Figure 10) reveals a similar trend as described by the formula [32] and the research conducted by Ros [34].Figure 11 visually represents the damage observed during the splitting tensile strength test. The concrete specimen exhibits a central split, with some cracks crossing the coarse aggregates and others forming around them, resulting in winding crack paths. According to [35], cracks in normal-grade concrete typically occur around the coarse aggregates, resulting in meandering crack paths. In high-grade concrete, cracks traverse the aggregates. Therefore, the visual observations from the splitting tensile strength test indicate that the concrete falls within the normal-grade category. This observation is also supported by the experimental results, as the compressive strength of PCC concrete is < 40 MPa (normal-grade concrete).

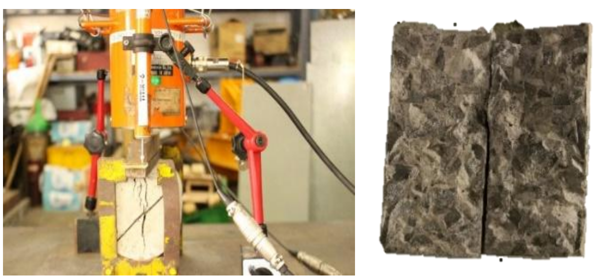


Figure 11 Damage visualization on splitting tensile strength test

K. FRACTURE ENERGY

This test aims to understand the behavior of concrete regarding the formation, propagation, and spread of cracks. [36].

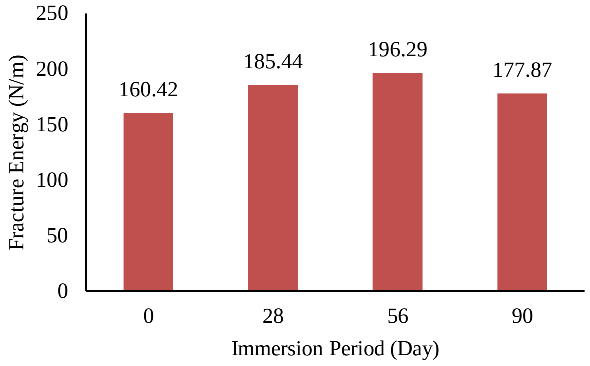


Figure 12 Fracture energy test result

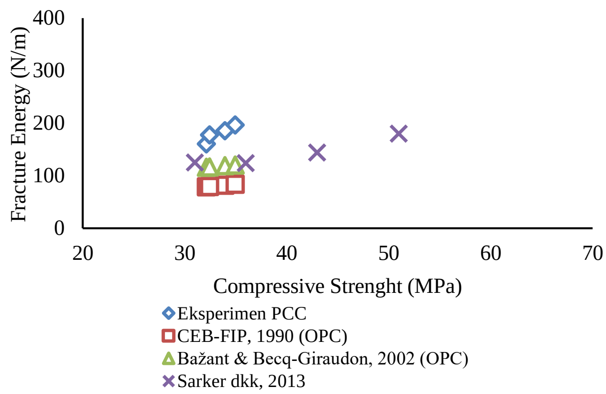


Figure 13 The relationship between compressive strength and fracture energy of PCC Concrete

Fracture energy is required to initiate cracks per unit surface area in a plane parallel to the crack direction [37–39]. he value of fracture energy is influenced by the mechanical interaction between aggregates and the binding matrix [40]. Fracture energy can be calculated by measuring the Load-CMOD curve using equation (6). The experimental Load-CMOD curve for PCC concrete is shown in Figure 14.

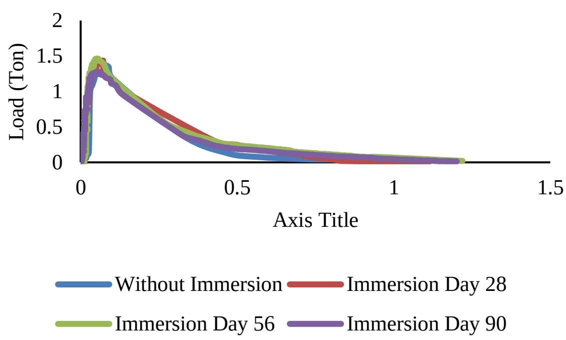


Figure 14 Curve of Load-CMOD

The test results indicate that the fracture energy value of PCC concrete is positively correlated with the compressive strength and splitting tensile strength, which is influenced by the duration of sulfate exposure. The compressive strength influences the fracture energy value. Prolonged sulfate exposure initially increases compressive strength, splitting tensile strength and fracture energy until a specific exposure time, after which they decrease [41]. This study's fracture energy value is higher than OPC concrete in CEB-FIP 1990 and the research conducted by [40, 42]. This is attributed to the crack resistance ability demonstrated by the splitting tensile strength test of PCC concrete, which is higher than OPC concrete's splitting tensile strength value in the research [43].

Figure 20 Damage visualization on fracture energy test

CONCLUSIONS

This study demonstrates that a 90-day magnesium sulfate exposure of PCC concrete. The detailed conclusions can be summarized as follows:

1. The pH value of the solution increased during the exposure period, indicating the alkaline nature of the leaching sulfates from the concrete.
2. The sulfate content in the solution decreased significantly after 28 days of exposure, indicating a reaction between sulfates and the concrete.
3. The compressive strength of PCC concrete initially increased up to 56 days of exposure but decreased after 90 days. This can be attributed to the formation of ettringite and gypsum, which initially enhanced the strength but eventually led to the development of microcracks at longer exposure times.
4. The modulus of elasticity exhibited a similar trend to the compressive strength, increasing up to 56 days and decreasing after 90 days of exposure.
5. The Poisson's ratio of PCC concrete fell within the range of 0.19-0.22, comparable to the standard values for OPC concrete.
6. The tensile strength and fracture energy showed an initial increase up to 56 days of exposure, followed by a decrease after 90 days. The compressive strength of the concrete influenced the fracture energy.

Overall, the 90-day exposure to magnesium sulfate had a notable influence on the mechanical properties of PCC concrete, leading to changes in strength, modulus of elasticity, and crack resistance.

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