The Durability of Composite Cement Paste Using Diatomaceous Earth against Sodium Sulfate Attack

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Abstract. In this study, the durability against sodium sulfate attack test of composite cement paste using diatomaceous earth was carried out. Diatomaceous earth was calcined in several processes, in order to obtain micro-particles with a more dominant silica content which was used as a mineral material to produce composite cement. The proportion of diatomaceous earth in composite cement was 0%, 10%, 20% and 30% of total cement weight. The amount of water was determined, so that a composite cement paste with normal consistency was obtained. Composite cement paste was made by mixing all the constituent materials in a mixer. The specimens were made in accordance with ASTM standards, namely cubes with 50 mm sides with 9 specimens for each mixtures. The test was carried out by immersing the specimens in 10% sodium sulfate solution for one and two months then the visual changes, loss of mass and changes in compressive strength were observed. Composite cement paste that experienced smallest damage was composite cement paste with 10% and 20% diatomaceous earth. Mass loss was occurred only in the original Portland cement paste after two months immersion. The compressive strength of the composite cement paste experienced the least degradation at 20% diatomaceous earth content.

1. Introduction
Cement paste has functions as an adhesive in the hardening process of the concrete mixture so that the aggregate grains are firmly bonded to each other and form a compact mass [1]–[3]. Therefore cement paste is one of the important components of concrete which is the most important element in a construction. This research was conducted in the form of testing the durability of a composite cement paste with diatomaceous earth minerals to replace some of the cement against sodium sulfate attack. There were several studies on the strength and durability of concrete under sulphate attack [4]–[8]. A common sign of sulfate attack is the formation of ettringite or wax salt that fills the pore space, after being filled over time the sulfate will induce an expansive internal pressure from the inside which will cause cracks along with the appearance of crystals or white spots in the area affected by sulfate attacks. This causes the risk of porous concrete become very high.

This research is important because currently infrastructure development is not only carried out in general residential areas that do not require special resistance requirements, but many constructions have been built in aggressive area to fulling the needs of areas with high sodium sulfate content such as industrial and mining areas which are vulnerable to attack these compounds.

Diatomaceous earth is classified as Class N SCM (supplementary cementitious materials). Diatomaceous earth was abundant in Aceh Besar, Seulimum district with an estimated 40,353,700.00 tons. The main chemical composition of calcined diatomaceous earth is SiO$_2$ of 56.0%, Fe$_2$O$_3$ of 26.4%, ZrO$_2$ of 7.2%, and Al$_2$O$_3$ of 6.5%, so that diatomaceous earth from Aceh Besar area has fulfilled EN 197-1 requirements because it has reactive silica (RS) above 25% [9]. Based on the results of previous research, it was found that blended cement which has characteristics such as
Portland cement can be made using diatomaceous earth as much as 10% to 30% in the mixture [10]. Composite cement paste with the amount of cement of 2040 kg/m$^3$ and micro particles of diatomaceous earth of 510 kg/m$^3$ resulted compressive strength of 53.94 MPa [10]. Many studies have been conducted on the utilization of diatomaceous earth as cement replacement as well as for lightweight aggregate and brick production [9–16].

The aim of this study was to investigate mass changes, visual changes and changes in the compressive strength of composite cement paste before and after exposure to sodium sulfate (Na$_2$SO$_4$). Based on these data, the durability of composite cement paste to Na$_2$SO$_4$ attacks was analyzed according to the ASTM Standard. The significance of this research is to produce composite cement using diatomaceous earth which is more durable and can be applied in the aggressive area.

2. Materials and Method

2.1. Materials

Diatomaceous earth obtained directly from Lampanah Village in the form of large chunks, then crushed with a hammer into small chunks. After that, the diatomaceous earth lumps were burned directly in a brick burning furnace for 3 days with a burning temperature of 650°C [17]. After incineration, the diatomaceous earth lumps were ground using a Los Angeles Machine became fine powder. Then, the resulting fine powder was sieved with sieve No.200 (0.075 mm) and then calcined in laboratory furnace at the temperature of 650°C for 5 hours. The cement used to mix with diatomaceous earth powder to produce composite cement was Ordinary Portland Cement (OPC) from PT. Semen Andalas Indonesia.

2.2. Method

Mixing was done in a concrete mixer and the workability was tested by means of a flow test. The composite cement paste mixture was designed so that the mixture was obtained at a normal consistency. The proportion of water needs is obtained through a series of tests that have been carried out by previous researchers [10]. The proportion of water needs obtained by previous researchers could be seen in Table 1.

| Mixtures                                    | Water Quantity (%) |
|---------------------------------------------|--------------------|
| Ordinary Portland Cement (OPC)              | 24.63              |
| Composite cement with 10% diatomaceous earth| 26.22              |
| Composite cement with 20% diatomaceous earth| 29.32              |
| Composite cement with 30% diatomaceous earth| 32.43              |

As many as 9 specimens were made for each mixture. Mix designs were used based on normal consistency. The composite cement paste mixture was cast into a 5 cm side cube mold. After 24 hours, the mold was opened and then the sample was treated by immersing it in water for 28 days. After that, the specimens were dried and then tested against the sodium sulfate attack. Sulfate attack resistance testing will refer to ASTM C452 / C452M-10 and ASTM C1012 / C1012M-18a [18,19]. Prior to testing, observations were made by looking at the change in the color of the composite cement paste, damage such as edge cracks on the surface of the composite cement paste and changes in the shape of the specimens surface after being exposed to the sodium sulfate solution.

The composite cement paste mix proportion was designed based on normal consistency [10] and shown in Table 2.

| Materials                                      | DE = 0% | 10% | 20% | 30% |
|-----------------------------------------------|---------|-----|-----|-----|
| OPC (kg)                                      | 4.208   | 3.787 | 3.366 | 2.945 |
| Diatomaceous earth, DE (kg)                   | 0       | 0.421 | 0.842 | 1.262 |
| Water (kg)                                    | 1.036   | 1.103 | 1.234 | 1.364 |
To obtain the compressive strength-immersion time curve of composite cement paste, a compression load was applied on the 50 mm cube specimens according ASTM C 109/C 109M-02 [20] through an compression testing machine until the specimens failed. The test was carried out after the exposure time of 1 and 2 months and compared with those without exposure.

3. Results and Discussion

3.1. Visual Appearance

Visual observations were mad after immersing the samples in Na$_2$SO$_4$ solution for 1 month and 2 months and compared with composite cement paste before immersion. The view obtained is shown in Figures 1-4. The defect on the surface of the specimens were marked with red cycles in the figures.

![Figure 1](image1.png)

**Figure 1.** Visual appearance of cement paste DE-0 after immersion of: (a) 0; (b) 1; (c) 2 months

![Figure 2](image2.png)

**Figure 2.** Visual appearance of cement paste DE-10 after immersion of: (a) 0; (b) 1; (c) 2 months

![Figure 3](image3.png)

**Figure 3.** Visual appearance of cement paste DE-20 after immersion of: (a) 0; (b) 1; (c) 2 months

![Figure 4](image4.png)

**Figure 4.** Visual appearance of cement paste DE-30 after immersion of: (a) 0; (b) 1; (c) 2 months

3.2. Mass Change

The change in the mass of the specimens was measured after exposure in Na$_2$SO$_4$ solution for 1 and 2 months. The results of the mass change of the specimens are shown in Table 3.
Table 3. Mass loss of composite cement specimens due to Na$_2$SO$_4$ attack

| Exposure time | DE content | Average mass before immersion (gr) | Average mass after immersion (gr) | Average mass loss (%) |
|---------------|------------|-----------------------------------|----------------------------------|-----------------------|
| 1 month       | 0%         | 272.67                            | 272.67                           | 0                     |
|               | 10%        | 268.00                            | 268.00                           | 0                     |
|               | 20%        | 254.00                            | 254.00                           | 0                     |
|               | 30%        | 253.67                            | 253.67                           | 0                     |
|               | 0%         | 264.00                            | 263.67                           | 0.13                  |
| 2 months      | 10%        | 261.33                            | 261.33                           | 0                     |
|               | 20%        | 260.67                            | 260.67                           | 0                     |
|               | 30%        | 248.67                            | 248.67                           | 0                     |

3.3. Compressive Strength

The compressive strength test was carried out after the composite cement paste was exposed in Na2SO4 solution for 1 and 2 months. The results of the compressive strength test are shown in Table 4 and Figure 5.

Table 4. Change in compressive strength

| Exposure time | DE content | Average compressive strength (MPa) | Ratio of compressive strength after being exposed to that without exposure, $f'_c/f_{co}$ (%) |
|---------------|------------|-----------------------------------|-----------------------------------------------------------------------------------------------|
| Not expose    | 0%         | 76.98                             | 100.00                                                                                        |
|               | 10%        | 73.78                             | 100.00                                                                                        |
|               | 20%        | 69.73                             | 100.00                                                                                        |
|               | 30%        | 61.30                             | 100.00                                                                                        |
| 1 month       | 0%         | 70.81                             | 91.98                                                                                        |
|               | 10%        | 69.73                             | 94.51                                                                                        |
|               | 20%        | 66.87                             | 95.90                                                                                        |
|               | 30%        | 56.64                             | 92.40                                                                                        |
| 2 months      | 0%         | 68.74                             | 89.29                                                                                        |
|               | 10%        | 68.03                             | 92.21                                                                                        |
|               | 20%        | 65.65                             | 94.14                                                                                        |
|               | 30%        | 55.82                             | 91.07                                                                                        |

Figure 5. Reduction of compressive strength of cement paste specimens due to Na$_2$SO$_4$ attack

4. Conclusions

Based on the results in this study the following conclusions can be drawn:
a. Cement paste changes color, which were getting white with the length of immersion in Na$_2$SO$_4$ solution.
b. After 2 months of exposure, the composite cement paste which have suffered the least damage due to Na$_2$SO$_4$ were composite cement paste with content 10% and 20% of diatomaceous earth.
c. Mass loss to Na$_2$SO$_4$ attack only occurred in the second month of normal cement paste, which was 0.13%. Meanwhile, the composite cement paste with the addition of diatomaceous earth did not have mass loss.
d. The least decrease in compressive strength due to Na$_2$SO$_4$ attack occurred in the specimens with 20% diatomaceous earth in the first month and the second month with residual compressive strength of 95.90% and 94.14% respectively of the initial compressive strength.
e. The use of 20% diatomaceous earth by weight of cement can increase the durability of the composite cement paste against Na$_2$SO$_4$ attack.

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