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Long-term performance of the healed mortar with polymer containing phosphazene after exposed to sulfate attack

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HIGHLIGHTS

- Mortar samples were exposed to sulfate attack for one year.
- Then, they healed using the polymer containing phosphazene.
- This article applied the Taguchi method and Anova analysis.
- The samples exposed to the sulfate attack were found to be healed with the polymer containing phosphazene.

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ABSTRACT

In this study, mortar samples were exposed to sulfate attack for 1 year and then healed with the polymer that contains phosphazene. After this healing, the microstructure and mechanical properties of these mortars were investigated. In the experimental work, the percentage of phosphazene in the monomer (0, 1%, 2%, 3% and 4%), the curing time (30, 60, 90, 180 and 365 days), the cement dosage (300, 350, 400, 450 and 500 kg/m^3) and percentage of sulfate solution (0%, 1%, 2%, 4% and 6%) were selected as experimental parameters. Due to the high number of experimental parameters, experiments were reduced using the Taguchi L_{25} (5^6) orthogonal array as the test plan. After the design of the experiment, 50 × 50 × 50 mm cubes and 25 × 25 × 285 mm prismatic samples were prepared. The produced samples were exposed to the sulfate attack for the predetermined curing times. The samples were dried at 105 ± 5 °C for 24 h after sulfate attack. Following this procedure, the monomer containing phosphazene was impregnated to samples at atmospheric conditions for 24 h. 1% benzoyl peroxide was used as the monomer initiator. The samples were heated to 60 °C for 4 h to polymerize the monomers. The compressive strength, weight changes and length changes of mortar specimens healed using polymer containing phosphazene after sulfate attack were investigated. Furthermore, SEM, EDX and XRD analyzes were conducted on the samples.

1. Introduction

Various studies were conducted on the defects that affect the durability of concrete and cause degradation such as the sulfate attack, reinforcement corrosion, alkali-aggregate reactions and carbonation [1–3]. Chemical deterioration could occur in the cementitious matrix due to the products that cause expansion [4]. Sulfate sources that are harmful to concrete are divided into two categories of internal and external sources [5]. Groundwater, sea water and sulfated floors are the main sources of external sulfate. The internal sulfate resources can be found in cement, aggregate, water and additives used in concrete production. Sulfates from internal and external resources that attack the concrete penetrate the matrix and form a chemical reaction with the hydration products of the cement. These reactions result in new substances such as gypsum and ettringite [1,6]. In case of sulfate penetration into the concrete from the external environment, C_3A and monosulfate react with sulfate to form ettringite, resulting in concrete expansion, fracture formation, cracking of the surface, flaking off of the surface and hence a loss of strength [7,8]. This loss would accelerate the deterioration of the concrete structure by means of harmful ions penetration through the concrete cracks [9–12]. The precautions could be performed to ensure durability of the concrete against sulfate attack such as ensuring the impermeability of the concrete, limitation of C_3A and $Ca(OH)_2$ content in cement, using pozzolanic additives, protection of cement against external sulfate with cladding if necessary and choosing a solution depending on the severity of the environmental effect [13]. Several studies were

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conducted on the sulfate attack on concrete. Tanyildizi [14] investigated the strength and microstructure of mortar samples that contained fly ash and silica fume exposed to sulfate attack. This study concluded that the compressive strength of mortar samples that contained silica fume was higher. Wang et al. [15] studied the strength of concrete containing fly ash and silica fume exposed to freeze-thaw cycles and sulfate attack. Their results revealed that fly ash and silica fume increased the strength against sulfate attacks and increased the freeze-thaw resistance. A study by Mangat and Khatib [16] investigated the sulfate resistance of the concrete that contained blast furnace slag aggregate and silica fume at different dosages. The total binder content was 350 kg/m^3 and 450 kg/m^3 and the water/cement ratio was 0.45. Porosities and pore structures of the samples per unit volume were measured by calculating the amount of mercury intrusion porosimetry and carbonation amount. The samples were cured at temperatures that ranged between 20 °C and 45 °C for 14 days with a moisture content of about 25.55%. These samples were then stored in a sulfate solution. In conclusion, they found that concrete produced with fly ash aggregate between 22% and 32% was resistance to sulfate attack. Qi et al. [17] examined recycled aggregate concrete exposed to sulfate attack and wetting-drying cycles. It was found that recycled aggregates could severely ameliorate the damage when they were exposed to processes such as wetting and drying cycles and to the sulfate solution. The study obtained findings that would allow the accumulation and crystallization of sulfate in concrete. Jo et al. [18] examined the effect of hydrogen-rich water on the acid and sulfate resistance of cement mortars. As a result, they found that hydrogen-rich water improved the acid and sulfate resistance of the cement mortars.

There are many studies on the use of polymers in concrete or mortar [19–25], but the use of the polymer containing phosphazene in mortar hasn't been found in the literature. Therefore, this work has been done. In this study, the strengthening using the polymer containing phosphazene of the mortar samples exposed to sulfate attack was investigated.

2. Experimental details

2.1. Material

The Portland cement used in the experiments is CEM I 42.5 according to TS EN 197-1 standard [26]. The properties of the cement were presented in Table 1. In this study, 0–3 mm aggregate was used. Vinyl acetate monomer was used as the monomer in the experiments. Monomer and sodium sulfate properties were given Table 2 and 3.

2.2. Experimental design and mixture proportions

In general, the strength properties mainly depend on the manufacturing conditions employed such as: maximum size of aggregate, curing conditions, mineral admixtures and curing time etc.

Table 1
The chemical properties of cement.

Portland Cement	% by mass
SiO_2	21.12
Al_2O_3	5.62
Fe_2O_3	3.24
CaO	62.94
MgO	2.73
LOI	1.42
Specific surface area (cm^2/g)	3430
Particle size	–
Specific gravity (g/cm^3)	3.10

Table 2
Sodium sulfate properties.

Loss on Ignition	≤0.5%
Insoluble matter	≤0.01%
Chloride (Cl)	≤0.001%
Nitrogen Compounds	≤5 ppm
Phosphate (PO_4)	≤0.001%
Calcium (Ca)	≤0.01%
Magnesium (Mg)	≤0.005%
Potassium (K)	≤0.01%
Iron (Fe)	<=0.001%

Table 3
Vinyl acetate monomer properties.

Boiling point, (°C)	101
Freezing point, °C	−48
Density, (kg/l)	0.933–0.936
Purity,%	99.99

[27]. In this study, parameters and levels selected for the experiments were presented in Table 4. The factors affecting compressive strength, weight loss and length change were selected as the percentage of phosphazene used in the polymer, curing time, cement dosage and sulfate solution rate. Based on these variables, the L_{25} (5^6) orthogonal array was selected as the experimental plan and presented in Table 5. The mixture ratios used to produce the mortar samples were given in Table 6. 25 × 25 × 285 mm and 50 × 50 × 50 mm mortar samples were prepared for the sulfate attack experiments. The total number of the samples is 150. The samples remained in the mold at 24 h. The samples that were removed from the mold were kept in at 20 ± 2 °C standard water curing for 28 days. Then, they kept in 0%, 1%, 2%, 4% and 6% sulfate solution ($NaSO_4$) for 30, 60, 90, 180 and 365 days. Finally, the monomer containing phosphazene was impregnated to samples at atmospheric conditions for 24 h. 1% benzoyl peroxide was used as the monomer initiator. The samples were heated to 60 °C for 4 h to polymerize the monomers.

2.3. Sulfate attack experiment

In this study, 75 cubic mortar samples of 50 × 50 × 50 mm and 75 prismatic mortar specimens of 25 × 25 × 2858 mm were produced for the experiments using L_{25} (5^6) orthogonal array presented in Table 5. The samples were stored at 20 ± 2 °C in standard water curing for 28 days. Later, they were exposed to 0%, 1% (10.000 mg/l), 2% (20.000 mg/l), 4% (40.000 mg/l) and 6% (60.000 mg/l) sulfate solution ($NaSO_4$) for 30, 60, 90, 180 and 365 days. Lastly, the compressive strength, weight and length changes of the samples that were exposed to sulfate attack were investigated. In ACI-225R-85 [28] and ACI-201.2R-77 [29], any sulfate solution with a sulfate ion concentration of 1500 ppm < SO_4 = <10.000 ppm is described as "Heavy sulfate solution environment". When the sulfate concentration is higher than 10.000 ppm, it is defined as "very severe sulfate solution environment". According to ACI standards, the mortar samples manufactured in this study were exposed to very severe sulfate attack. According to ASTM C 1012 [30], it is necessary to conduct the experiments for 4, 6, 9 and 12 months. Accordingly, 365 days maximum cure time was selected. Thus, mortar samples were exposed to very severe sulfate solution conditions for the maximum curing time according to ACI standards. The length changes in mortar specimens produced in 25 × 25 × 285 mm size were periodically measured using a measurement device with a sensitivity of 0.01 mm, according to the ASTM C 1012 standard. Before measurement, the samples were