Study on the performance of low water-binder ratio cement mortar with excavated soil exposed to NaCl freeze-thaw environment

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Abstract

In this paper the mechanical strengths (compressive and flexural strengths) of cement mortar with subway excavated soil and low water-binder are studied. Moreover, the following NaCl freeze-thaw resistance is investigated. The water-binder ratio of cement mortar is 0.3 and the corresponding soil-cement ratios are 0%, 5%, 10%, 15% and 20%. The mass and mechanical strengths losses, the loss of the relative dynamic elastic modulus and permeability coefficient of chloride ions are determined during freeze-thaw cycles. Besides, the scanning electron microscope (SEM) photos of cement mortar are observed to study soil’s influence on the microstructures of cement mortar and x-ray diffraction is determined to study the crystal composition. Results show that the mass and mechanical strengths losses and the permeability coefficient of chloride ions increase in the form of quadratic function with that of the number of freeze-thaw cycles. However, the relationship between the relative dynamic elastic modulus and freeze-thaw cycles is quadratic function. Additionally, the relationship between the permeability coefficient of chloride ions and freeze-thaw cycles fits positive linear function. Moreover, the mechanical strengths and freeze-thaw resistance of cement mortar are deteriorated with soil introduction. The cement mortar with a soil content of 5% exhibits the lowest strength and the worst freeze-thaw resistance, but a high soil content of 15% displays that the mortar with low water-binder ratio and soil content shows the best freeze-thaw resistance.

1. Introduction

In recent years, with the urbanization, cities in China have witnessed the rapid construction of subway lines. However, along with subways’ construction, a large amount of construction waste is produced. According to data from the journal of China building materials, by 2030, the construction waste will reach 7.3 billion tons in China [1–5], of which around 30% is excavated soil [6, 7]. Currently, except a small amount of soil is used as backfill soil for road construction, a tremendous amount of soil is disposed into low-lying land, wasteland or quarry [8, 9]. This extensive treatment method not only has potential safety hazards, affects the urban landscape, and causes pollution of urban roads, atmosphere, and water bodies, but also occupies a large amount of land, which seriously affects the sustainable development goals [10].

Reusing the disposing soil to prepare low-strength cement soil can reduce the project cost and alleviate the environmental problems. Many studies have been conducted to solve this problem, and it is found that utilization of the excess excavated soil as backfill and subgrade material provides the right solution for this problem [11]. It has been reported that the addition of cement or other binder materials can effectively improve the mechanical properties of soil [12]. Although some researchers reported applying low-strength cement soil for the tamping foundation and subgrade [13, 14], little attention has been paid to the application of excavated soil in the high-performance marine concrete.

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Concrete with low water-binder ratio is known as the ‘concrete of the 21st century’ due to its useful properties such as high strength and high durability. It is expected to be widely used in infrastructure construction such as bridges, tunnels, and road traffic in the next few decades. However, its high cost significantly limits the widespread application [15–21]. Compared with the high-quality silica fume, which can only be imported from abroad, construction waste has the advantages of enormous scale and easy availability [22]. It has been well acknowledged that with the proper processing, the construction waste can be reused as high valuable construction material [23–25]. Using construction waste as an alternative to silica fume into cement-based materials can reduce the production cost of high-performance concrete and effectively alleviate the environmental issues. Therefore, this method is a high value-added treatment method for construction waste [26].

Concrete construction buildings are usually exposed to various corrosive environments when applied in coastal areas. The corrosive seawater action includes freeze-thaw cycles, dry-wet alternations, and scouring of seawater [27–31]. And the coupling effects of these factors and external load. These factors lead to the acceleration of the chloride penetration, the spalling and inner cracks of cement concrete. Moreover, the embedded steel bars in cement concrete are prone to corrosion. Due to these reasons, a lot of research work about the evolution regular of performance attenuation should be carried out on the concrete with excavated soil. In this paper, the construction waste is used as the additive of cement mortar with low water-binder ratio. The effects of excavated soil on the strengths (compressive strength and flexural strength) of cement mortar are studied. To this end, freeze-thaw resistance and strength changes under the action of freeze-thaw cycles in a NaCl solution with a concentration of 3.0% are studied. This research provides an outlet for the disposal of construction waste.

2. Laboratory experiments

2.1. Raw materials

In this study, the ordinary Portland cement with the initial time of 173 min and final curing time of 246 min respectively produced by Zhejiang Ningbo Conch Co., Ltd. was used. The volume stability and other technical indicators meet standard ordinary Portland cement requirements. River sand was used as the aggregate. The sand was produced by Tianjin Yongfa Yongqiang Building Material Factory and belonged to grade II with a fineness modulus of 2.75. The water-reducing agent used in the test was a polycarboxylate high-performance water-reducing agent (SP) produced by Henan Pingdingshan Admixture Co., Ltd. It is a brown microemulsion solution, and the recommended dosage is around 1.0%, which can achieve a water reduction rate of up to 40%. For the mortar samples, cement to sand to water ratio was 1:1:0.15, and a 3.0% mass of water reduction agent was used. The construction waste used in the study is the subway excavated soil provided by the Shanghai Institute of Building Research. The soil has a maximum dry density of 1.66 g cm$^{-3}$, and the relevant technical properties are shown in table 1 and table 2.

2.2. Specimen preparation and maintenance

In this study, the Hobart A200C mixer was used to prepare the cement mortar. A microcomputer full-automatic cement bending test machine (model YAW300) produced by Jinan Hengruijin was used to conduct the strength test.

| Table 1. Particle passing percentage of raw materials. |
|-------------------------------------------|
| Particle size $\mu$m$^{-1}$ | 0.3 | 0.6 | 1 | 4 | 8 | 64 | 360 |
| Types | Cement | 0 | 0.33 | 2.66 | 15.01 | 28.77 | 93.59 | 100 |
| | Soil | 31.2 | 41.2 | 48.8 | 82.3 | 100 | 100 | 100 |

| Table 2. The chemical composition of cement. |
|--------------------------------------------|
| Chemical composition/\% | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | Loss | MgO | CaO | SO$_3$ | R$_2$O | P$_2$O$_5$ | Organics |
|--------------------------------------------|
| Types | Cement | 20.86 | 5.47 | 3.94 | 2.63 | 1.73 | 62.23 | 2.66 | 0.48 | 0 | 0 |
| | Soil | 46.07 | 13.68 | 4.81 | 11.5 | 3.27 | 12.09 | 0.26 | 4.1 | 0.31 | 5.2 |
The mixture design used is presented in Table 3. Weighed raw materials (cement, sand, water, and water reducing agent) were mixed in the mixer at a speed of 140 ± 5 rpm to prepare the mixture. After 30 s, medium sand was added and mixed at 285 rpm for 30 s and then stop for 90 s and finally stirred rapidly for 60 s. After stirring the mixed mixture was poured into the mound. After that, the specimens were sealed by plastic film and maintained at a temperature of 20 ± 2°C with humidity higher than 40%. After curing for 24 h, the mound was removed and then placed in a standard curing room with a temperature of 20 ± 2°C and humidity >95%. The specimen was used to test after curing for 28 d. The prepared specimen is a prism specimen of 40 mm × 40 mm × 160 mm. The microcomputer automatic cement crush test machine (model YAW300) manufactured by Jinan Hengruijin was used to conduct the compressive and flexural strength test. The loading rate for flexural strength and compressive strength were 0.05 kN/s and 2.4 kN/s respectively. The details can refer to Chinese standard GB/T17671–1999 ‘Cement Mortar Strength Inspection Method (ISO Method)’. For the freeze-thaw test, the prismatic specimens with a size of 40 mm × 40 mm × 160 mm were used to test the strength loss of high-performance mortar after different freeze-thaw cycles, and the specimens with a size of 100 mm × 100 mm × 400 mm were used for the dynamic elastic modulus measurement. Specimen after standard curing for 24 d were immersed in 3.0% NaCl solution for 4 d and then subjected to freeze-thaw test at a temperature from −15 °C to 8 °C following GB/T 50082–2009 Standard for Test Methods of Long-term Performance and Durability of Ordinary Concrete. The whole experimental process is shown in figure 1. The scanning electron microscopy (SEM) was observed by JSM-6360LV scanning electron microscope (Japan electron optics laboratory, Tokyo, Japan) and x-ray diffraction (XRD) spectrum was obtained by D8 ADVANCE x-ray diffractometer (Bruker Corp., Tokyo, Japan) respectively. Before the microscopic experiment, samples should be treated as follows. The specimens with different curing ages were immersed in anhydrous alcohol for 5 d. Then, a vacuum drying oven with the constant temperature of 40 °C was applied for drying the samples for 4 days. The dried samples with soybean size were metallized with gold and used for SEM observations. Meanwhile, else dried samples were pulverized and used for x-ray powder diffraction (XRD) tests.

Table 3. Mix proportion of cement mortar mixed with soil added per unit volume kg⁻¹.

| Water | Cement | Sand | Water reducer | Soil |
|-------|--------|------|---------------|------|
| 183.3 | 1222.1 | 977.9 | 39            | 0    |
| 183.3 | 1161   | 977.9 | 39            | 61.1 |
| 183.3 | 1099.9 | 977.9 | 39            | 122.2|
| 183.3 | 1038.8 | 977.9 | 39            | 183.3|
| 183.3 | 977.7  | 977.9 | 39            | 244.4|

Figure 1. The preparation process of the sample.
3. Results and discussion

3.1. Mechanical strength

Figure 2(a) shows the flexural and compressive strengths of cement mortar at different soil contents. Figure 2(b) shows the flexural strength and compressive strength loss rate of the specimens after different NaCl freeze-thaw cycles. As shown in figure 2 that the flexural and compressive strengths of mortar decrease with the increasing content of soil. At a low content from 0% to 5% (mass ratio of soil to the total mass of cement and soil), the strength of mortar decreases rapidly by showing 26.1% and 9.3% reductions in flexural strength and compressive strength. However, when the soil content ranges from 5% to 15%, the mechanical strengths of mortar increase with the increasing soil content. The mechanical strengths of cement mortar with this dosage level of soil demonstrate slow downward trend. When the soil content increases from 15% to 20%, the mechanical strengths of cement mortar decrease with the increasing soil dosage. Therefore, it can be concluded that the mechanical strengths of cement mortar can be deteriorated by the incorporation of soil and reach the lowest when the dosage of soil is 5%. However, the mechanical strengths increase with the further incorporation of soil and reach the highest when the dosage of soil is 15%. The reduction in strength can be attributed to low chemical activity and the relatively slow hydration of soil at room temperature. Therefore, the addition of soil can delay the hydration capacity of the cementitious material and further decreasing the mechanical strength. Nevertheless, the particle size of the soil used in this study is lower than the cement’s particle size leading eventually to the micro-aggregate effect in the material and improving the mechanical strength. Therefore, the
mechanical strength of the cement mortar with excavated soil can further increase when the soil content increases from 5% to 15% [32, 33].

3.2. Mass loss with NaCl freeze-thaw cycles
Figure 3 shows the mass loss ($\frac{\Delta m}{m}$) of cement mortar mixed with excavated soil and table 4 displays the fitting result of the relationship between the mass loss and NaCl freeze-thaw cycles ($N$). As illustrated in figure 4, the

| Equation | Recycled aggregate | $a$   | $b$   | $c$   | $R^2$ |
|----------|--------------------|-------|-------|-------|-------|
| $\frac{\Delta m}{m} = aN^2 + bN + c$ | 0%     | $1.80 \times 10^{-5}$ | 0.0033 | 0.25  | 0.91  |
|          | 5%     | $-2.33 \times 10^{-5}$ | 0.021  | 0.16  | 0.99  |
|          | 10%    | $-5.52 \times 10^{-6}$ | 0.013  | 0.23  | 0.91  |
|          | 15%    | $-3.14 \times 10^{-6}$ | 0.012  | 0.22  | 0.92  |
|          | 20%    | $-1.36 \times 10^{-5}$ | 0.015  | 0.25  | 0.91  |

Figure 4. Flexural strength loss during freeze-thaw cycles.

Figure 5. Compressive strength loss during freeze-thaw cycles.
mass loss of cement mortar with excavated soil increases with freeze-thaw cycles. This is attributed to the fact that the freeze-thaw cycles can induce the spalling of cement mortar thus leading to the mass loss. It can be found that during different freeze-thaw cycles, the cement mortar with excavated soil shows a higher mass-loss rate than cement mortar without excavated soil and reaches the maximum value when the dosage of excavated soil is 5%. However, the mass loss of this mortar with 10% excavated soil is the lowest when the excavated soil is added in the cement mortar. It can be concluded from figure 3, the mass loss of cement mortar with excavated soil is in the range of 3.02% $\sim$ 4.57% after 300 NaCl freeze-thaw cycles indicating that this cement-based material shows excellent integrity of mass. It can be observed from table 4, the relationship between the mass loss and NaCl freeze-thaw cycles fits the quadratic function. As depicted in table 4, the fitting degrees of all curves are higher than 0.91 indicating the rationality of the fitting equations.

### 3.3. Mechanical strengths during NaCl freeze-thaw cycles

Figure 4 and 5 show the mechanical strength loss of cement mortar with excavated soil during the NaCl freeze-thaw cycles. It can be seen that as the freeze-thaw cycles (N) increases, the mechanical strength loss of cement mortar gradually increases. After 300 NaCl freeze-thaw cycles, the compressive strength loss of cement mortar is 8.62% $\sim$ 23.82%; and the flexural strength loss is 29.3% $\sim$ 38.4%. Previous studies reported [35] that in a 3.0% NaCl solution, the freezing point of the concrete pore solution is $-2.3 \degree C$ to $-1.8 \degree C$, indicating that the concrete was suffered freeze-thaw cycles at $-15 \degree C$ to 8 $\degree C$. In a 3.0% NaCl solution, the cement mortar has high moisture absorption and water retention capability, which give the specimen a high initial water saturation.
before the NaCl freeze-thaw cycles. As the freeze-thaw process continued, the internal seepage pressure and freezing speed of the cement mortar increased, leading to the accumulation of cement mortar damage. Freeze-thaw cycles caused damage to the internal cement mortar and decreased its mechanical strength [36].

At an excavated soil content of 5%, cement mortar shows its highest mechanical loss. With the incorporation of excavated soil, the decrease of cement content and the less hydrated cement worse the mechanical properties. However, with the increase of excavated soil content (more than 5%), its mechanical properties can be improved to a certain extent due to the micro-aggregate filling effect. Thus, the strength loss rate can be lower than cement mortar with 5% excavated soil content. It can also be seen from the figure that the flexural strength loss of cement mortar with soil is significantly higher than the compressive strength loss during the freeze-thaw process, indicating that the reduction rate of flexural performance of cement mortar with excavated soil is higher than that of compressive performance during NaCl freeze-thaw cycles. It can be seen from the figure that cement mortar with 5% excavated soil content shows the highest mechanical strength during NaCl freeze-thaw cycles, and the corresponding soil contents to the strength losses from low to high are 0% < 15% < 10% < 20% < 5%.

Tables 5 and 6 display the fitting result of the relationship between the mechanical strength loss and NaCl freeze-thaw cycles. As depicted in tables 5 and 6, the RDEM values loss increases in the form of quadratic function with positive correlation.

Figure 7 shows the curve of the relative dynamic elastic modulus (RDEM) of cement mortar with the number of NaCl freeze-thaw cycles. It can be seen that the increase in the number of NaCl freeze-thaw cycles leads to a continuous decrease in the relative dynamic modulus of cement mortar. According to previous studies [34, 35, 37], the freeze-thaw cycle in NaCl solution can cause micro-cracks in the cement mortar and further reduce sound wave propagation in cement mortar. Thus, the relative dynamic elastic modulus can be decreased by the increasing freeze-thaw cycles. It can be found that the relative dynamic elastic modulus of cement mortar with excavated soil is lower than that without excavated soil. At an excavated soil content of 5%, the relative dynamic elastic modulus of cement mortar is the lowest and decreases the fastest with the increasing freeze-thaw cycles. The corresponding excavated soil contents to the dynamic elastic modulus from low to high are 0% < 15% < 10% < 20% < 5%. Table 7 illustrates the fitting result of the relationship between the relative dynamic elastic modulus (RDEM) and NaCl freeze-thaw cycles. As depicted in table 7, the RDEM values loss increases in the form of quadratic function with negative correlation.

| Equation                        | Excavated soil | a     | b    | c    | R²     |
|---------------------------------|----------------|-------|------|------|--------|
| RDEM = aN² + bN + c             | 0%             | 2.29 x 10⁻⁴ | -0.15 | 101.13 | 0.96   |
|                                 | 5%             | 2.37 x 10⁻⁴ | -0.17 | 98.19  | 0.97   |
|                                 | 10%            | 2.37 x 10⁻⁴ | -0.17 | 99.14  | 0.98   |
|                                 | 15%            | 2.33 x 10⁻⁴ | -0.16 | 99.42  | 0.98   |
|                                 | 20%            | 2.42 x 10⁻⁴ | -0.17 | 98.85  | 0.98   |

Figure 7. The chloride migration coefficient during NaCl freeze-thaw cycles.
3.4. Chloride migration coefficient during NaCl freeze-thaw cycles

Figure 7 shows the chloride migration coefficient (CMC) during NaCl freeze-thaw cycles. Table 8 is the fitting results of the CMC and number of freeze-thaw cycles. As illustrated in figure 7 and table 8 that the CMC increases linearly with the number of increasing NaCl freeze-thaw cycles. It can be obtained from figure 7 that the addition of excavated soil leads to the increase of CMC. As demonstrated in figure 7, the increasing ratio of CMC of cement mortar ranged from 24.4% to 108.1% with the increasing number of NaCl freeze-thaw cycles due to the number and width of cracks by freeze-thaw cycles [35]. However, the addition of excavated soil can

| Equation | Excavated soil | a     | b     | $R^2$ |
|----------|----------------|-------|-------|-------|
| $f_i = aN + b$ | 0% | 0.04  | 4.26  | 0.98  |
|           | 5%  | 0.046 | 4.76  | 0.97  |
|           | 10% | 0.053 | 4.95  | 0.96  |
|           | 15% | 0.054 | 5.38  | 0.98  |
|           | 20% | 0.053 | 6.00  | 0.99  |

Table 8. The fitting results of the CMC and number of freeze-thaw cycles.

Figure 8. SEM microstructure photos of cement mortar mixed with excavated soil: (a) 0% soil; (b) 5% soil; (c) 10% soil; (d) 15% soil; (e) 20% soil.
lead to the increasing ratio of CMC ranging from 9.4% to 42.7%. The CMC of cement mortar with excavated soil in this study increases in this order: 0% < 15% < 10% < 20% < 5%.

3.5. Microscopic analysis
Figure 8 shows the scanning electron microscope (SEM) photos of cement mortar mixed with excavated soil. All specimens were measured after cured for 28 days. As shown in figure 8(b), at the soil-cement ratio of 5%, more loose parts are found in cement mortar hydration products. However, from figures 8(c)–(e), we can find that as the soil ratios are higher than 5%, the hydration products of cement mortar become more compact with the increasing soil. It can be observed from figure 8, and more flocculent hydrations are generated with the addition of excavated soil, which also can be applied in the analysis of the variation of mechanical properties. It can be found from figure 8, at the addition of excavated soil from 0% to 5%, the generated hydration is looser thus leading eventually to the decrease in flexural and compressive strengths. However, with the soil increasing from 5% to 20%, the hydration of cement mortar become denser, leading to the improvement of flexural and compressive strengths.

3.6. XRD analysis
Figure 9 shows the XRD images of specimens containing different dosages of excavated soil. It can be found that all the spectra show strong diffraction peaks of 3CaO·SiO₂ (C₃S), 2CaO·SiO₂ (C₂S), cristobalite (SiO₂). Besides, diffraction peak of calcium hydroxide (CH) decrease with the increasing dosage of soil, while the diffraction peak of SiO₂ increases. This might be attributed to the fact that the high content of SiO₂ in soil leading to improvement of diffraction peak of SiO₂. Moreover, a reduction of the diffraction peak of calcium hydroxide also can be observed that could be attributed to the high content of SiO₂, which could decrease the hydration and thus leading to the reduction of calcium hydroxide [38]. Therefore, we can conclude that the incorporation of soil can delay the hydration of cement. From this perspective, the addition of soil can decrease the flexural and compressive strengths of cement mortar. However, the soil with lower particle size can lead to the micro-aggregate effect in the material and improving the mechanical strengths.

4. Conclusions
Based on this study, the following conclusions can be drawn:

(1) The compressive strength and flexural strength of cement mortar is deteriorated by incorporating soil. The cement mortar with soil shows its highest and lowest strengths at soil content of 5% and 15%, respectively.

(2) The strength of cement mortar decreases with the increasing freeze-thaw cycles in NaCl solution. The cement mortar shows the fastest strength loss at a soil content of 5% and the lowest strength loss at a soil content of 15%. The mass and mechanical strengths losses and the permeability coefficient of chloride ions increase in the form of quadratic function with that of the number of freeze-thaw cycles.
(3) The relationship between the permeability coefficient of chloride ions and freeze-thaw cycles fits positive linear function. The relative dynamic elastic modulus of cement mortar decreases with the increasing freeze-thaw cycles. Cement mortar with 5% and 15% soil contents exhibits the fastest and lowest relative dynamic elastic modulus reductions.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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