

Research Article

Experimental Study on Damage Characteristics of Solidified Mud under Coupling Action of Freeze-Thaw Cycle and Chloride Salt

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The physical properties of solidified mud will deteriorate under the combined action of freezing-thawing cycle and chlorine salt, which will affect the safety and stability of solidified mud engineering. In order to study the damage characteristics of solidified mud under the action of freeze-thaw cycle and chloride salt, the solidified mud samples were soaked in different concentrations of sodium chloride solution and treated with freeze-thaw cycle. Besides, the unconfined compressive strength test was carried out to study the damage characteristics of the samples caused by different concentrations of sodium chloride solution and freeze-thaw cycles. The test results show the following. (1) Under the action of freeze-thaw cycle and sodium chloride solution, the surface of the sample deteriorated in varying degrees. With the increase in freeze-thaw cycle times and sodium chloride solution concentration, the mass loss of the sample was greater. (2) Under the action of chlorine salt and freeze-thaw cycle, the strength of the sample decreases with the increase in freeze-thaw cycle times. With the increase in the concentration of sodium chloride solution, the strength of the sample decreases and the damage degree increases gradually. (3) Under the action of freeze-thaw cycle, the internal pores of the samples gradually expand and more honeycomb holes appear, and the content of hydration products is higher. As the concentration of sodium chloride solution increases, the particle connection inside the sample gradually weakens, and the sample is more likely to be damaged.

1. Introduction

Engineering mud is a semifluid mixture composed of water, bentonite, or clay particles and additives. With the rapid development of construction industry, mud is widely used in engineering construction. When the project is completed or the mud cannot meet the use requirements, a large number of engineering construction waste mud will be produced. The random discharge of these waste mud wastes not only wastes resources but also pollutes the environment. Therefore, it is very important to seek an efficient and inexpensive method to treat waste mud for resource utilization. After years of continuous research by scholars at home and abroad, the chemical curing method has become the most direct and effective common method for mud treatment. The waste mud treated by different curing methods can be

used as foundation treatment or road landfill material according to its corresponding mechanical and durability, making it a building material and achieving the purpose of resource utilization. Luo [1] carried out the solidification test of drilling waste mud by mixing lime and fly ash as the main curing agent and found that the indexes of the solidified mud test block meet the requirements of the specification, which is suitable for subgrade materials. Zhou et al. [2] used strong stirring curing equipment to solidify waste mud in situ, making it a highway filling material with certain strength. Zalihe [3] has achieved good results by adding fly ash into bentonite mud, which promotes the development of bentonite mud solidification research and provides important reference basis. At present, solidified mud is widely used in road engineering and hydraulic construction. However, due to the vast territory of our country, the climate

TABLE 1: Chemical composition of the test materials (%).

Component	SiO ₂	Al ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	FeO	Fe ₂ O ₃	SO ₃	Loss
Bentonite	71.2	18.52	0.82	0.54	1.56	0.98	0.32	0.12	—	5.94
Slag	35.32	20.46	28.41	5.97	0.22	—	—	1.54	1.46	6.62
Quicklime	2.25	0.93	90.50	1.78	0.60	—	—	0.85	0.90	2.19

conditions in different regions are also different; in particular in cold regions, the materials need to have freeze-thaw resistance, so it is very important to study the durability of solidified mud under freeze-thaw cycles. Powers' hydrostatic pressure hypothesis [4] and osmotic pressure hypothesis [5] explain the mechanism of freeze-thaw damage. Chen et al. [6] studied the microscopic characteristics of expansive soil under freeze-thaw cycles and found that with the increase in freeze-thaw cycles, the porosity of the sample increased gradually, and the initial cycle effect was the most obvious. Wang et al. [7] studied the improvement effect of bentonite on the freeze-thaw resistance of solidified silt soil and found that there was an optimal value of bentonite content. Near this value, bentonite had the best improvement effect on the freeze-thaw resistance of solidified silt soil. Cheng et al. [8] used cement and industrial waste residue to solidify drilling waste mud and conducted a freeze-thaw cycle test to test the frost resistance of solidified mud. It is found that the strength of solidified mud decreases with the increase in freeze-thaw cycles. The increase in phosphogypsum content in curing agent can reduce the strength loss of solidified mud after freeze-thaw cycles.

In the northeast and some coastal areas, there are often saline soil and bridge subgrade engineering construction; the subgrade will also suffer salt erosion damage [9–11]. He et al. [12] studied the apparent and unconfined compressive strength of solidified sludge under salt erosion environment and discussed the action mechanism of erosion solution on solidified sludge. Jiang et al. [13] found that the corrosion of cement soil was stronger under the combined action of acid and sodium salt through the corrosion test of cement soil under sodium salt and acid-base solution. Tu and Tang [14] added fly ash, mineral powder, or silica powder to the grouting material to conduct the erosion test of grouting material in seawater and found that the service life of the material was significantly improved. Di Maio et al. [15, 16] studied the effect of salt solution concentration on the strength of bentonite and found that with the increase in salt solution concentration, the internal friction angle of residual strength of bentonite increased. Jiang et al. [17] studied the microscopic changes of concrete freeze-thaw cycles in different sodium chloride solutions and quantitatively characterized the pore structure evolution process combined with the fractal dimension of pore structure. Wang et al. [18] studied the durability of polypropylene fiber-reinforced concrete under the coupling effect of chloride salt and freeze-thaw and studied the mass damage and relative elastic modulus damage of the sample. These studies show that the salt solution and freeze-thaw cycle have a great influence on the damage characteristics of the specimen, but there are few studies on the damage properties of the solid-

ified slurry under salt erosion and freeze-thaw cycles, and further studies are needed.

In this paper, the solidified slurry under different freeze-thaw cycles and different concentrations of chlorine salt erosion was taken as the research object. The unconfined compressive strength of the sample was tested, and the changes of the physical and mechanical properties of the sample were studied. The microscopic damage mechanism of the solidified slurry was further analyzed, providing reference for engineering practice.

2. Test Materials and Methods

2.1. Test Materials. The bentonite needed for the preparation of the test mud is sodium bentonite, which is taken from Renshou County, Sichuan Province. Its composition is shown in Table 1. The pure alkali used in the experiment is edible pure alkali, which is produced in Tianjin Binhai New Area, and the main component is sodium carbonate (Na₂CO₃). The test sand is China ISO standard sand produced by Xiamen Aisiou Standard Sand Co., Ltd. The particle size ranges from 0.08 mm to 2 mm; the thickening agent is sodium carboxymethyl cellulose (CMC), and the model FVH9 has high viscosity. S105 slag powder produced by Lingshou Dehang Mining Products Co., Ltd. is selected as the main solidified material slag. The main components are Ca₂Al₂SiO₇ crystal and Ca₂MgSiO₇ crystal. Calcium oxide (CaO) is the main component of lime, which is purchased from Jiangxi Province. The content of CaO is 90.5%, which belongs to high calcium lime. Calcium carbonate is the main component of raw materials, and CO₂ is discharged by appropriate calcination. The chemical compositions of slag and lime are shown in Table 1. The activator sodium silicate is industrial liquid sodium silicate, which meets the quality standard of GB/T 4209-2008 industrial sodium silicate, and the modulus is 2.3. The water used in the test is Huainan water; sodium chloride is the analytical purity produced by Tianjin Comiou Chemical reagent Co., Ltd., the content is not less than 99.5%, and three different concentrations of sodium chloride solution (10 g/L, 20 g/L, and 40 g/L) are configured.

2.2. Preparation of Solidified Mud Samples. The engineering mud used in this experiment was prepared manually in the laboratory. Combined with the ratio of mud in practical engineering and the related research of domestic scholars on the preparation of mud by bentonite [19], the mass ratio of mud composition was set as water : bentonite : soda ash : CMC = 100 : 12 : 0.5 : 0.06. When the mud was prepared, the bentonite that was not dissolved or accumulated at the bottom should be fully stirred and rubbed. When the



FIGURE 1: Mud sample.

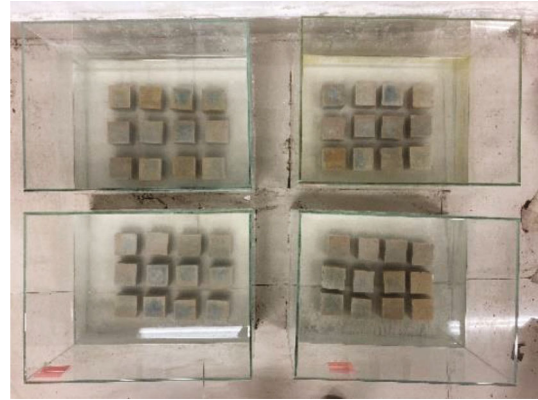


FIGURE 2: Sample soaking.

bentonite was fully dissolved and there was no sediment at the bottom, it should be stored in the shade of the room for further use. The tested prepared mud met the various indicators [20].

After the mud is configured, the material is weighed according to the selected ratio, and the total dosage of curing agent is 25%. The mass ratio of slag and quicklime is 17:3, and the dosage of sodium silicate and sand is 5% and 40% of the mud quality, respectively. The solidifying material with good accurate weighing was properly stirred in the stirring pot with a shovel, and then, the mud and sodium silicate which were previously configured were weighed and poured into the stirring pot. The cement mortar mixer was used to stir for 5 min until the mixture was mixed uniformly. After the stirring, the mixture was poured into the cube mold of 50 mm × 50 mm × 50 mm and formed on the vibration table. After the sample is prepared, the sample is sealed with the preservative film. After 3 days of maintenance in the standard maintenance room, the sample is demoulded. After the demoulding, the sample is numbered and placed in the sealing bag and then placed in the standard maintenance room for 28 days to prepare the test, as shown in Figure 1.

2.3. Test Scheme. The samples that reached the curing age were taken out to check whether the appearance was intact. The samples were soaked in water and 10 g/L, 20 g/L, and 40 g/L three different concentrations of sodium chloride solution for 5 days, and the water temperature was 20°C. When soaking, the liquid surface was kept above 20 mm the top surface of the sample (Figure 2). After the end of soaking, remove the sample to dry the surface moisture and put it into the sealing bag.

The freeze-thaw cycle test adopts the TEST-1000 type rapid freeze-thaw testing machine. The sample after soaking is taken out and put into the test machine for freezing. The freezing temperature is -15°C, and the freezing time is 12 h. After the freezing is completed, the sample is transferred to the standard curing room for melting 12 h, and the melting temperature is 15°C, which was a freeze-thaw cycle. The freeze-thaw cycles were set to 0, 2, 4, 6, 8, and 10 times. The specimens after a certain number of freeze-thaw cycles were taken out and weighed, and the unconfined compressive

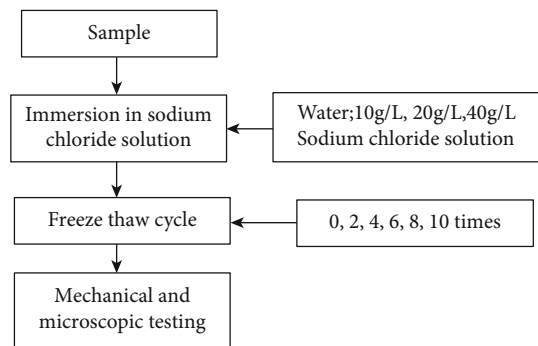


FIGURE 3: Test flowchart.

TABLE 2: Test scheme.

Freeze-thaw times	Strength of solution			
	Water	10 g/L	20 g/L	40 g/L
0 time	1-1	—	—	—
2 times	2-1	2-2	2-3	2-4
4 times	3-1	3-2	3-3	3-4
6 times	4-1	4-2	4-3	4-4
8 times	5-1	5-2	5-3	5-4
10 times	6-1	6-2	6-3	6-4

sive strength tests were carried out. The unconfined compressive strength test is carried out according to the standard of geotechnical test method (GB/T50123-1999). The WDW-50 microcomputer controlled electronic universal testing machine was used, and the test was carried out according to the displacement loading mode. The preload was set at 0.05 kN, and the loading rate was 1 mm/min. After the test, the microscopic changes of some samples were observed by SEM. The specific process is shown in Figure 3. The specific test scheme is shown in Table 2.

3. Test Results and Analysis

3.1. Apparent Characteristic Analysis. The apparent characteristics of samples with different freeze-thaw cycles were

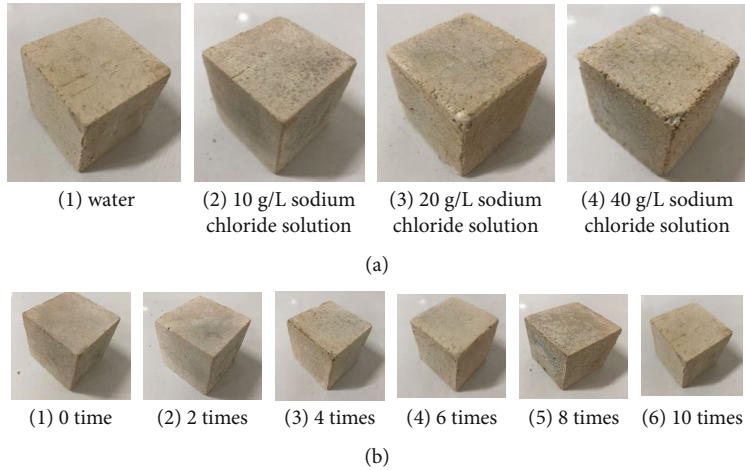


FIGURE 4: Sample appearance: (a) appearance diagram of sample under different concentration solutions; (b) appearance diagram of samples under different freeze-thaw cycles.

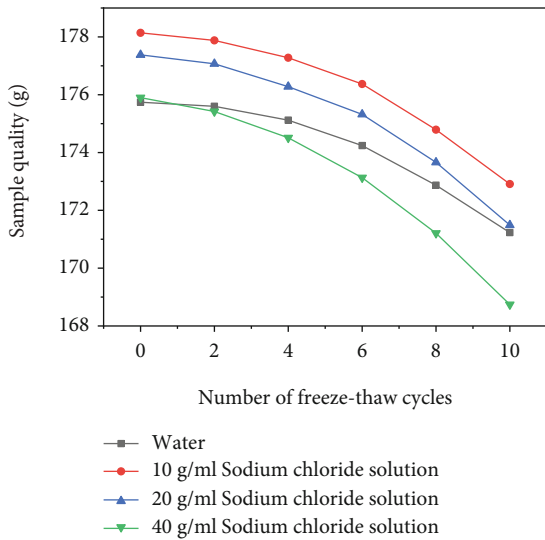


FIGURE 5: Variation of sample mass with the number of cycles.

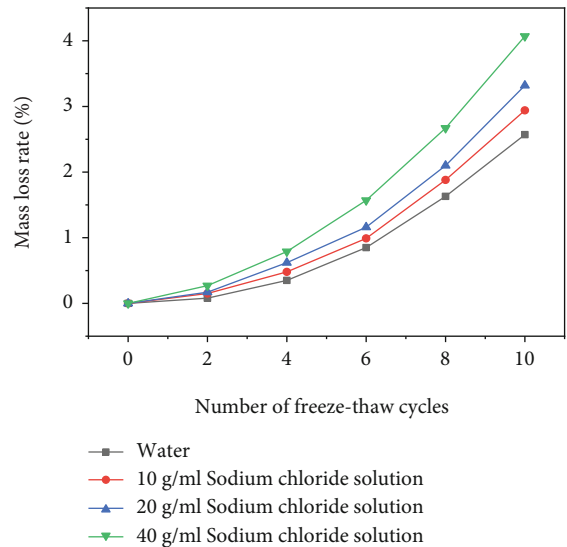


FIGURE 6: Variation of sample mass loss rate with the number of cycles.

studied. After 10 freeze-thaw cycles, the apparent images of samples with different solution concentrations are shown in Figure 4(a), and the apparent images of samples with different freeze-thaw cycles in water are shown in Figure 4(b).

Figure 4(a) shows that the surface of the sample in water is relatively flat, and there is no obvious crack, while the sample in sodium chloride solution has obvious numbness, holes, and cracks. The corner of the solidified mud sample has begun to fall off under the erosion of 20 g/L sodium chloride solution; more holes and cracks appear on the surface. The specimen corroded by 40 g/L sodium chloride solution was the most seriously damaged. The holes and cracks began to cover the entire surface of the specimen, and the edges and corners fell off seriously. This is because different concentrations of chloride react with the internal components of the sample, resulting in dissolution of the material. With the increase in salt solution concentration, the effect is strengthened, and more defects appear on the surface of the

sample. It can be seen from Figure 4(b) that with the increase in freeze-thaw cycles, more defects appear on the sample surface, which is because the residual water on the sample surface is frozen during freezing, and the volume expansion will lead to cracks and damage on the sample surface. At the same time, under the action of multiple freeze-thaw cycles, the cracks caused by the change of internal structure will gradually develop to the surface of the sample, resulting in the change of the apparent characteristics of the sample.

3.2. *Quality Changes.* With the increase in freeze-thaw cycles, the quality change of the sample can reflect the damage degree of the sample to a certain extent. 10 times of freeze-thaw cycles were selected to study the change of sample quality. Figure 5 is the change law of sample quality under different freeze-thaw cycles. In order to study the change law of sample quality, the mass loss rate is

TABLE 3: Unconfined compressive strength of the specimen.

Freeze-thaw cycles/times	Unconfined compressive strength of solutions with different sodium chloride concentrations (MPa)			
	Water	10 g/L	20 g/L	40 g/L
0	4.19	—	—	—
2	2.53	2.47	2.35	2.14
4	2.39	2.26	2.13	1.98
6	2.21	2.16	2.04	1.79
8	2.13	2.02	1.86	1.62
10	2.06	1.91	1.75	1.59

introduced. The formula is shown in (1), and the mass damage rate of the sample without the freeze-thaw cycle is shown in Figure 6.

$$\omega_n = \frac{m_0 - m_n}{m_0} \times 100\%. \quad (1)$$

In the formula, ω_n is the mass loss rate of solidified mud sample (%), m_0 is the initial mass of solidified mud sample before freezing and thawing (g), and m_n is the quality of the solidified mud sample after n freeze-thaw cycles (g).

It can be seen from Figure 5 that the quality of samples soaked in water and different sodium chloride solutions decreases gradually with the increase in freeze-thaw cycles. This is because during the freezing-thawing cycle, the free water in the sample will be frozen and melted continuously. During the freezing, the water changes from liquid to solid, and the formed ice crystals experience the expansion of the sample volume, resulting in large expansion force. Due to the irregular shape of ice crystals, large concentrated stress will be generated in the sample, resulting in cracks in the sample. During melting, due to the increase in temperature, ice crystals change from solid to liquid water, and these water will migrate to the new cracks generated inside the specimen, resulting in hydrostatic pressure inside, which intensifies the expansion and development of cracks [21]. These cracks will make the surface and corner of the sample to have holes and fall off, and the moisture in the sample is gradually evaporated during the freeze-thaw cycle, resulting in water loss, and ultimately leads to the continuous decline of the quality of the sample.

It can be seen from Figure 6 that the mass loss rate of the sample increases with the increase in freeze-thaw cycles. In the early stage of freezing and thawing cycle, the mass loss rate was small, and the difference in mass loss rate of samples after different solutions was small. In the later stage of the freeze-thaw cycle, the mass loss rate gradually increased, and the difference of mass loss rate of samples after each solution was also gradually increased. Under the action of the freeze-thaw cycle, the quality loss of the sample mainly comes from the evaporation of water in the process of the freeze-thaw cycle and the hole and fall off caused by the freeze-thaw cycle. In the early stage of the freeze-thaw cycle, due to the less number of freeze-thaw cycles, the cracks on the surface of the sample are not obvious, and there are fewer holes and fall off. At this time, the main reason for

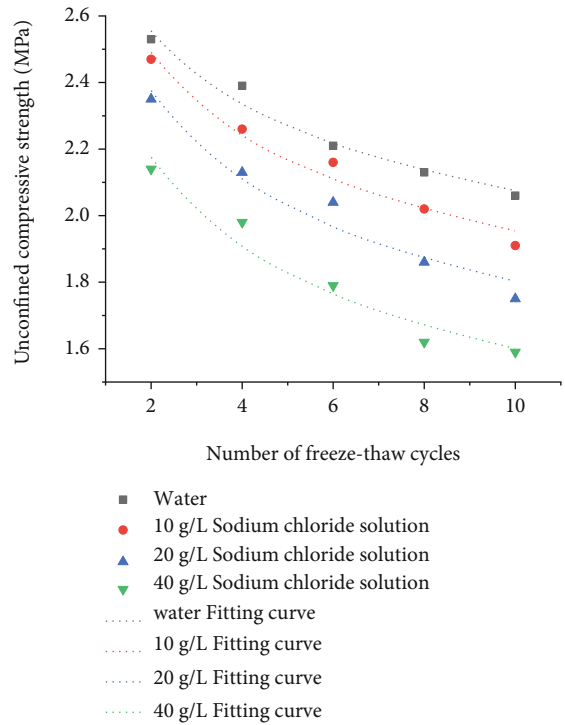


FIGURE 7: Relationship between unconfined compressive strength of the sample and cycle times.

the decrease in the quality of the sample is the loss of water in the process of the freeze-thaw cycle. The effect of water loss on the quality of the sample is relatively small, so the weight loss rate increases slowly. There is little difference in the quality of the sample under different solution erosions. After several freeze-thaw cycles, more cracks appeared on the surface of the sample, and the holes and surface shedding of the sample were the main reasons for the decrease in the sample quality. Therefore, the mass loss rate increased greatly at this time, and the mass loss rate of the sample was larger.

3.3. Unconfined Compressive Strength. The unconfined compressive strength of the samples after different freeze-thaw cycles (0, 2, 4, 6, 8, and 10 times) and different solution concentrations was tested. The test results are shown in Table 3.

It can be seen from Table 3 that the unconfined compressive strength of the sample in the same solution

decreases with the increase in the number of cycles. After 10 freeze-thaw cycles, the unconfined compressive strength of the specimen decreased by 50.84% compared with that of the specimen without freeze-thaw cycles. It can be seen that the damage of the specimen was more obvious under multiple freeze-thaw cycles, which was mainly due to the change of the internal structure of the specimen under the action of freeze-thaw cycles. The sample before freezing and thawing is in a saturated state after soaking. When the temperature is reduced during freezing, the free water in the pores of the sample changes from liquid to solid, releasing heat and generating volume expansion. When the expansion stress is greater than the bonding force of the internal cementing material, small and irreversible cracks will occur [22]. When the sample melts, it absorbs heat and forms a temperature difference inside the sample [23]. At the same time, the solid ice melts into liquid water and continues to migrate to the new cracks, forming hydrostatic pressure, resulting in a decrease in strength. Through comparison, it can be seen that after the first two freeze-thaw cycles, the unconfined compressive strength decreased most, while the unconfined compressive strength decreased slowly in 2 to 10 freeze-thaw cycles. This is because in the first two freeze-thaw cycles, the cracks in the sample have already existed, resulting in that the change of the sample is irreversible. In the later freeze-thaw cycles, the solution is not soaked, and the pores in the sample cannot be supplemented by water. Only the microcracks generated in the previous internal continue to develop and expand, resulting in a slower decrease in strength. Therefore, the later freeze-thaw cycles have little effect on the strength of the sample.

Further, from the data in Table 3, it can be seen that the unconfined compressive strength of the sample decreases gradually with the increase in sodium chloride solution concentration under the same freeze-thaw cycles. It shows that chloride affects the unconfined compressive strength of the sample, and the influence is more obvious with the increase in concentration. The reason for this phenomenon is that NaCl solution penetrates into the sample and a large number of chloride ions and sodium ions are separated. The charge adsorption of chloride ions and sodium ions hinders the sample. Internal hydration reaction and precipitation of crystals at the same time that the sample internal produced a huge expansion stress result in cracks in the sample internal. At the same time, with the increase in the concentration of sodium chloride solution, the internal saturation of the sample is larger, and greater tensile stress will be generated during the freezing expansion, which will also increase the internal cracks, resulting in the gradual decrease in the strength of the sample with the increase in the number of freeze-thaw cycles [24].

In order to study the influence of different freeze-thaw cycles and different solutions on the unconfined compressive strength of solidified mud samples, the unconfined compressive strength of samples after different concentrations was fitted with the number of freeze-thaw cycles. The fitting formula is shown in (2), and the fitting curve is shown in Figure 7.

$$\sigma_u = an^b. \quad (2)$$

TABLE 4: Sample fitting parameters.

Different kinds of solution	a	b	R^2
Water	2.795	-0.129	0.974
10 g/L sodium chloride solution	2.767	-0.151	0.973
20 g/L sodium chloride solution	2.679	-0.172	0.958
40 g/L sodium chloride solution	2.486	-0.191	0.955

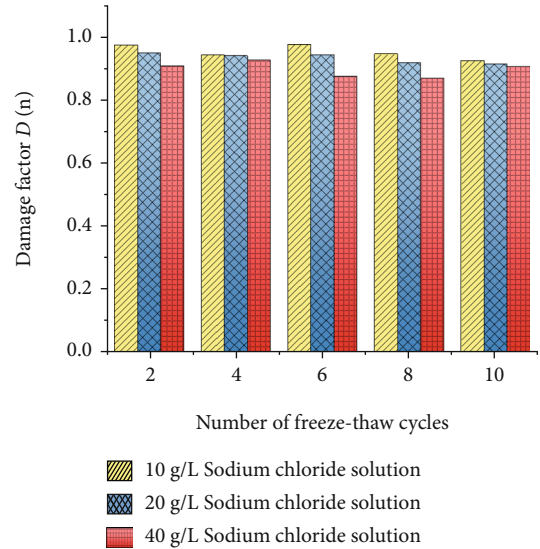


FIGURE 8: Relationship between sample damage and cycle times and solution concentration.

In the formula, σ_u is the unconfined compressive strength of the solidified mud sample (MPa), a, b are sample-related material parameters, and n is the number of freeze-thaw cycles (times).

It can be seen from Figure 7 and Table 4 that the fitting effect between the unconfined compressive strength and the number of freeze-thaw cycles of the samples after the action of different concentrations is good, indicating that formula (2) can better predict the relationship between the unconfined compressive strength and the number of freeze-thaw cycles of the samples with different concentrations of chlorine salt solution and can predict the strength variation law of the solidified slurry samples under the coupling of chlorine salt and freeze-thaw cycles. Further, it can be seen from the fitting curve that with the increase in freeze-thaw cycles, the decrease in sample strength gradually slows down.

3.4. Damage Factor. In order to describe the influence of chlorine salt erosion and freeze-thaw coupling on the unconfined compressive strength damage of solidified slurry samples, the damage characteristics of solidified slurry under different concentrations of erosion solution and freeze-thaw cycles were further analyzed and compared. The damage factor $D(n)$ was introduced to evaluate the durability of solidified slurry under chlorine salt erosion and freeze-

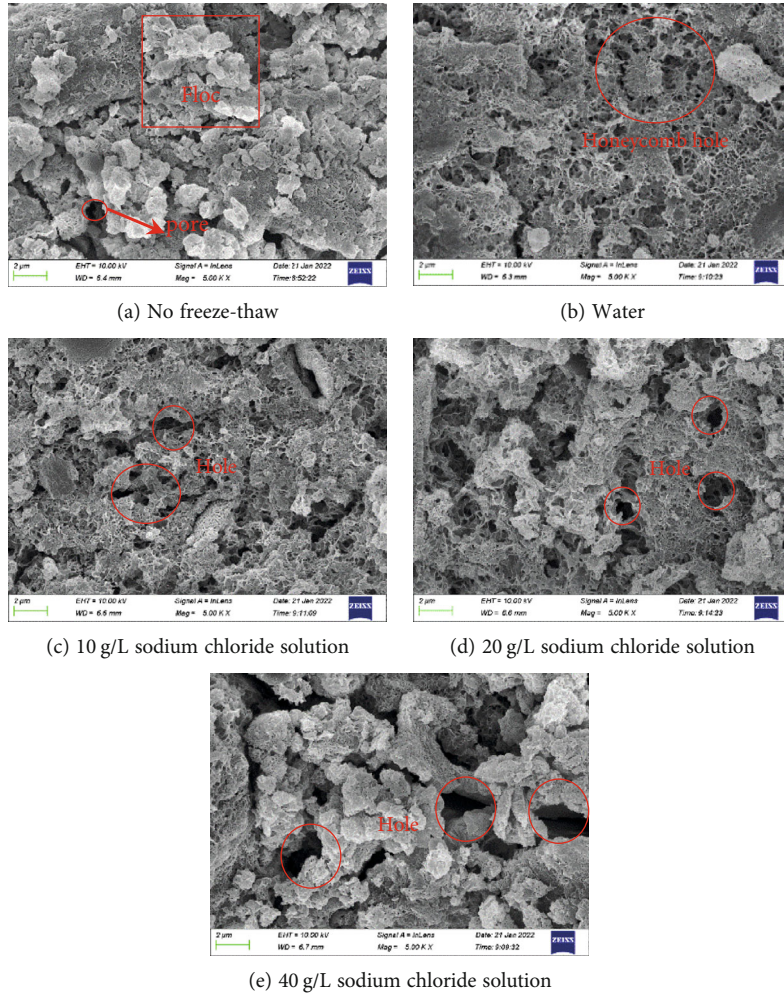


FIGURE 9: Microstructure of samples with different treatment methods.

thaw coupling [25]. The calculation formula of compressive strength damage is shown in

$$D(n) = \frac{f_1(n)}{f_w(w)}. \quad (3)$$

In the formula, $D(n)$ is the damage factor, $f_w(n)$ is the unconfined compressive strength of the solidified slurry sample soaked in water for n times (MPa), $f_1(n)$ is the unconfined compressive strength of the solidified slurry sample after the n th freeze-thaw cycle in different solution concentrations (MPa). By calculating the damage factor of solidified mud samples, the relationship between the strength damage of samples and the solution concentration and freeze-thaw cycles is obtained, as shown in Figure 7.

It can be seen from Figure 8 that with the increase in the number of freeze-thaw cycles, the damage factors of the samples under different solutions gradually decreased, indicating that under the action of freeze-thaw cycles, the compressive strength of the samples gradually decreased, and the internal structure of the samples was continuously destroyed. Under the action of freeze-thaw cycles, the resis-

tance to freeze-thaw corrosion of the samples gradually weakened. Further, it can be seen from the figure that under the same freeze-thaw cycle, the higher the concentration of sodium chloride solution is, the lower the damage factor of the sample is, indicating that the sample has different degrees of damage under the action of different sodium chloride solutions. The higher the concentration of sodium chloride solution is, the stronger the internal corrosion of the sample is and the more serious the compressive strength damage of the sample is.

4. Microanalysis of Samples after Chloride and Freeze-Thaw Cycles

Figure 9 shows the microstructure of the samples treated with different solutions after 10 cycles of nonfreezing and freezing-thawing. It can be seen from Figure 9(a) that there are a small number of small pores in the solidified slurry sample without freezing and thawing cycles. The particle structure of the sample is dense, and there are many hydration products. There are a large number of flocculent calcium silicate hydrate gel (C-S-H) and calcium hydroxide crystal (C-H). These hydration products are bonded to each

other to form a stable spatial structure, which fills the internal pores and makes the solidified slurry form a solid whole [26]. Macroscopically, it shows high strength and stable performance. In Figure 9(b), after 10 freeze-thaw cycles in clear water, the pores inside the sample began to expand under the action of freeze-thaw cycles, and there were many honeycomb holes. The overall performance of the sample was reduced [27, 28], and the hydration products with high content could be seen. However, compared with the samples without freeze-thaw cycles, there was less, which was also an important reason for the decrease in its macroscopic strength.

It can be seen from Figures 9(c)–9(e) that under the same freeze-thaw cycle, with the increase in the concentration of sodium chloride solution, the cracks in the sample gradually increased, the holes gradually increased, the structure became more loose, and the integrity of the sample was poor. Under the action of 40 g/L sodium chloride solution, there have been a lot of pores and cracks in the solidified mud sample, and less hydration products can be seen, resulting in that the bonding performance of the internal cementing material greatly weakened and the strength decreased. It can be seen that the erosion of chlorine salt has a great influence on the internal structure of the solidified slurry. The higher the concentration of sodium chloride is, the more serious the microscopic damage inside the sample is.

5. Conclusions

In this paper, the unconfined uniaxial compressive strength test of solidified mud samples under different freeze-thaw cycles and different concentrations of chloride salt erosion was carried out. Combined with the observation of microstructure, the physical and mechanical properties of samples with different freezing-thawing cycles and different concentrations of sodium chloride solution were studied. The damage and fracture mechanism of samples under different freeze-thaw cycles and different solutions was revealed. The following conclusions can be drawn:

- (1) With the increase in freeze-thaw cycles and sodium chloride solution concentration, the surface of the sample exhibited different degrees of damage and the mass of the sample decreases gradually. Under the same freeze-thaw cycles, with the increase in sodium chloride concentration, the erosion degree of sodium chloride solution on solidified mud samples is more serious, and the mass loss rate is greater
- (2) The strength of solidified mud samples decreases with the increase in freeze-thaw cycles, and the unconfined compressive strength of the samples decreases greatly when the freeze-thaw cycles are less. Under the same freeze-thaw cycles, the higher the concentration of sodium chloride solution is, the stronger the erosion ability of solidified slurry sample is. Under the action of the freeze-thaw cycle, the resistance to freeze-thaw corrosion of the sample gradually weakened. Under the action of sodium chloride solution, the damage degree of the sample gradually increased
- (3) Under the coupling of sodium chloride solution erosion and freeze-thaw cycles, the internal microstructure of solidified mud is characterized by the increase in pores, the decrease in hydration products, and the expansion and development of cracks. The higher the concentration of sodium chloride solution, the more serious the microscopic damage in the sample

Data Availability

The data can be obtained from the corresponding author.

Conflicts of Interest

The authors declare no conflict of interest.

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