

Article

Preparation of a Low-Cement-Content Silty Soil Stabilizer Using Industrial Solid Wastes

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Abstract: Silty soil performs poorly when used in roads. Cement is generally used as a stabilizer to treat silty soil and enable it to meet the requirements for roadbed filling. However, cement is an environmentally unfriendly material and can cost much. Meanwhile, solid wastes of ground granulated blast furnace slag (GBFS), fly ash (FA), and flue gas desulfurized (FGD) gypsum are produced in large quantities annually. Therefore, stabilizer A (cement:ground GBFS:fly ash:FGD gypsum = 30:44:15:11) and stabilizer B (cement:ground GBFS:fly ash:FGD gypsum = 40:38:13:9) were investigated in this study by reducing the cement content in the stabilizer and improving the utilization rate of solid wastes. The compressive strength development, California bearing ratio (CBR), temperature shrinkage, mineral composition, and micro-morphology of the stabilized silty soil were measured. The main findings are as follows: firstly, the addition of solid wastes can mitigate the adverse effect of delay time on compressive strength development. Secondly, the proposed stabilizers can significantly improve the CBR, which can reach 60% with a 4% dosage. Additionally, Stabilizer B is believed to improve the resistance to temperature shrinkage, and a higher stabilizer dosage can reduce the rate of decrease in water stability coefficient. Both X-ray diffraction analysis and scanning electron microscope observations show that the main hydration products that contribute to the stabilization are C-S-H and ettringite.



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Keywords: silty soil; soil stabilizer; delay time; mechanical properties; temperature shrinkage coefficient; water stability

1. Introduction

Silty soil is widely distributed in the alluvial plain of the Yellow River in Shandong Province, China. It is difficult for silty soil to meet the requirements for roadbed filling due to the loose structure of silt [1,2]. Generally, silty soil must be replaced [3,4]. This treatment is costly, and the materials used for backfilling are not always available in some regions.

Chemical soil stabilization has been considered an effective means of enhancing the engineering properties of soils [5]. It has obvious advantages, such as the ability to improve strength and stability, a wide source of raw materials, and more convenient on-site construction and maintenance [6]. Stabilizers can be classified into four different groups, namely, inorganic, organic, ionic, and bioenzymatic. Among them, inorganic stabilizers have an especially wide range of applications since they can stabilize various soils, such as clay, silt, and sand [7,8].

The most commonly used inorganic stabilizers are cement and lime [9]. However, the production of these two materials leads to a large amount of CO₂ emissions; this is especially true of cement, which contributes 6–8% of total global anthropogenic CO₂ [10] and consumes approximately 12–15% of total industrial energy [11,12]. Additionally, Ji et al. [6] revealed that a pure cement stabilizer leads to large shrinkage, while a pure lime stabilizer causes poor early age mechanical properties. Thus, there is a demand for stabilizers that meet the performance requirements for soil stabilization while minimizing

environmental pollution. Using industrial solid waste as a stabilizing material is thought to be a promising means for achieving this [13]. Industrial by-products such as fly ash (FA), flue gas desulfurized (FGD) gypsum, and granulated blast furnace slag (GBFS) are produced in a great amount every year. The global annual output of FA is approximately 900–1000 Mt [14,15], and it is approximately 140–330 Mt for GBFS [14,16]. China is the world's largest FGD gypsum producer [17], with an annual production of over 71.5 Mt [18]. The current treatment method for solid waste is mostly stacking and storage, which causes serious environmental pollution and a low utilization rate of solid waste. This study selected multiple types of solid waste to prepare stabilizers, which can help improve the utilization rate of solid waste and alleviate environmental pollution.

According to the literature, the performance of stabilizers can be modified by using the aforementioned solid wastes as admixtures. Xiao et al. [19] used OPC, fly ash, and silica fume along with phosphogypsum to stabilize oily sludge. The results showed that when the content of the stabilizer reached 20% (OPC/fly ash/silica fume = 1:0.7:0.8), the soil samples' 28-day compressive strength increased compared to the samples with the same content of OPC due to the refined pore structure. Rodriguez et al. [20] suggest that tropical soils can be stabilized using blends of electric arc furnace slag and fly ash. Kong et al. [21] found that 7.62% of reactive MgO, 11.31% of FGD gypsum, and 5.80% of steel slag can make the dredged sediment's microstructure more stable, thus improving its mechanical properties.

Almuaythir et al. [22] applied cement kiln dust to stabilize expansive soil. The results showed that the swell-collapse potential of the stabilized soil sample was fully diminished, and the compressive strength increased considerably compared to the unstabilized soil sample. Wang et al. [23] used ground GBFS, FGD gypsum, and calcium carbide slag (CCS) to treat marine soft soil. The experimental results showed that the best proportion of ground GBFS, FGD gypsum, and CCS was 61:8:31; the 28-day compressive strength of the stabilized soil exceeded 8.7 MPa, which was 1.23 times higher than that of OPC-stabilized soil.

In order to effectively address the problem of environmental pollution and a low utilization rate of solid waste, this work aims to investigate the feasibility of using a variety of solid wastes as a partial replacement for cement in the preparation of a new type of soil stabilizer. The mechanical properties of the stabilized soil specimens were tested for compressive strength and CBR. In practice, a certain delay period exists between mixing the stabilizer with soil and on-site operations. Therefore, a series of time intervals were set to figure out the effect of the delay time on strength development. Afterwards, temperature shrinkage and water stability performance were studied.

2. Experiment

2.1. Materials

The soil was classified as fine-grained soil according to Chapter 3.2 and T0115 of the Chinese standard JTG 3430—2020 [24]. According to T0188, the liquid limit and plastic limit were found to be 34.2% and 21.6%, respectively. The plasticity index is 9.6. Finally, according to Chapter 3.4 of the standard, the soil was defined as low-liquid-limit silt (ML). A compaction test was also conducted according to [24] to determine the optimum moisture content and maximum dry density of the soil. Figure 1 shows that the maximum dry density (MDD) was 1.84 g/cm³, and the optimum moisture content (OMC) was 15.3%. The densitometer method was employed to measure the particle distribution. The particle size analysis is shown in Figure 2.

P·I 42.5 ordinary Portland cement, slag, fly ash, and FGD gypsum were used to prepare the soil stabilizer. Their chemical compositions are provided in Table 1.

2.2. Experimental Design and Test Methods

Two soil stabilizers were designed. Their mixture proportions are given in Table 2. For soil stabilization, the stabilizer dosage was set at 4%, 6%, 8%, and 10%.

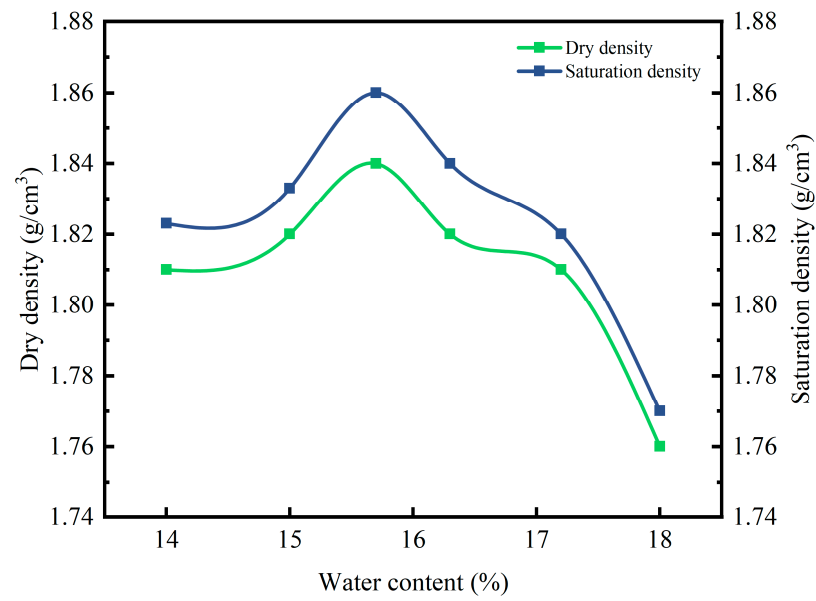


Figure 1. Relationship between dry density and water content of the silt.

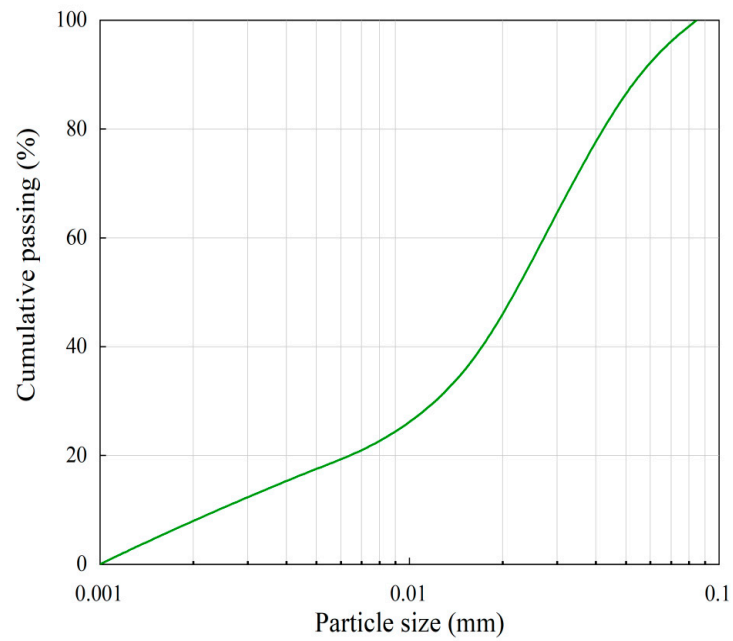


Figure 2. Particle size distribution of particles.

Table 1. Chemical composition of the cement and solid wastes.

Component	CaO (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	MgO (%)	Fe ₂ O ₃ (%)	SO ₃ (%)	TiO (%)	K ₂ O (%)	MnO (%)
Cement	63.21	18.48	6.74	3.24	3.45	3.16	0.35	—	—
Ground GBFS	44.71	29.29	14.85	7.33	0.39	1.28	0.68	0.41	0.42
Fly ash	3.44	49.93	36.17	0.79	5.8	1.12	1.1	1.17	—
FGD gypsum	45.35	1.56	0.8	0.35	0.12	50.63	0.02	0.41	—

Table 2. Mixture proportions of stabilizer A and B.

Stabilizer	Cement	Ground GBFS	Fly Ash	FGD Gypsum
A	30%	44%	15%	11%
B	40%	38%	13%	9%

The ground GBFS, fly ash, and FGD gypsum in the stabilizer were first mixed with the dried soil. Afterwards, a portion of water was added and mixed for 90 s [25]. At this stage, the moisture content of the mixture was 1–2% less than the OMC. Then, it was placed in sealed plastic bags for 6–8 h for uniform moisture distribution. Finally, cement and another part of water were added 1 h before static compaction [26], and the samples were mixed again to reach the OMC. The static compaction method was used to ensure that the specimens had a compaction degree of 98%.

For compressive strength measurement, the samples were compacted into cylindrical specimens of 50 mm in diameter and 50 mm in height [26,27]. The specimens were cured at a temperature of $20\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, with the relative humidity higher than 95%. Curing ages were 7, 28, 90, and 180 days, respectively. The loading speed was 1 mm/min. Six specimens in parallel for each group of specimens were used to test the compressive strength values.

In practice, a certain delay period exists between mixing the stabilizer with soil to on-site operation. The influence of this delay period on the development of compressive strength was investigated. Delay periods of 0, 2, 4, 6, and 8 h were selected. The operating steps were as follows: the soil–stabilizer mixture was thoroughly mixed after adding the second part of water. The mixtures thus prepared were placed in sealed plastic bags and kept in moisture-controlled desiccators during the time delay period. After the desired duration of time delay (0, 2, 4, 6, and 8 h), the mixtures were compacted statically to achieve the corresponding maximum dry unit weights. Eventually, their 7- and 28-day strengths were tested.

The California Bearing Ratio (CBR) test was conducted according to JTG 3430-2020 [24]. Firstly, the soil sample with 4% dosage stabilizer was cast in 3 layers and compacted into specimens under OMC. Three specimens were made for each mixture, and then they were soaked in water for 96 h. Finally, a strength tester for pavement materials with an experimenting rod was employed for loading. The penetration speed of the experimenting rod was 1–1.25 mm per minute, and the stress was recorded when the rod's penetration depth reached 2.5 mm.

The temperature shrinkage test was performed following JTG E51-2009 [26], and the specimen size was 50 mm \times 50 mm \times 200 mm. Three specimens were tested for each mixture, and the curing age was 7 days. An environmental chamber was employed to control the temperature, and the dial indicator was used to measure the specimens' length. The specimens were first soaked in water for one day prior to the test. Afterwards, they were dried in an oven, and their initial lengths were measured. The temperature setting range was between $-10\text{ }^{\circ}\text{C}$ and $40\text{ }^{\circ}\text{C}$. Every specimen was equipped with two dial indicators to ensure the reliability of the measurements.

Water stability test specimens were prepared in accordance with the preparation method in JTG E51-2009 [26]. A total of 12 specimens were prepared for each mixture. After 48 h of standard curing, 6 specimens continued standard curing, while the remaining specimens were soaked into water. The curing periods were 28 and 90 days (including the first 48 h of standard curing). The water stability coefficient was defined as the ratio between the compressive strength of the specimen under soaked conditions and that under standard curing conditions.

The mineralogical characterization was studied using X-ray diffraction analysis (XRD). First, the shattered pieces after the compressive strength test (curing ages 28 and 90 days) were collected. Then, the shattered pieces were soaked in ethanol to stop the further reaction and finally oven-dried at $110\text{ }^{\circ}\text{C}$ for 24 h [27]. A diffraction angle ranging from 5° to 90° with step size 0.05° and a scanning rate of 5° per minute was adopted for the test.

The reaction products were identified from the diffractograms of the stabilized samples. A scanning electron microscope (SEM) was used for hydration product characterization. A gold coating was applied on stabilized samples at 30 mA for 120 s before loading the sample for analysis. The working distance was 9.1–9.9 mm, and the accelerating voltage was 5.0 kV.

3. Results and Discussion

3.1. Compressive Strength

Figure 3 shows the solidified soil’s compressive strength for different curing ages and dosages. It can be seen that the compressive strength increased with stabilizer dosage. And, as expected, the longer the curing time, the higher the strength. Moreover, the 7-day strength was over 65% of the strength at 28 days for all mixes. After 7 days, the growth rate of the compressive strength decreased gradually. Under the same stabilizer dosage, stabilizer B had a higher strength than stabilizer A. This could be attributed to the fact that stabilizer B had a higher cement content. On the other hand, as shown in Figure 4, a high value of the coefficient of determination (R^2) indicated that a strong linear relationship existed between stabilizer dosage and compressive strength for both stabilizers.

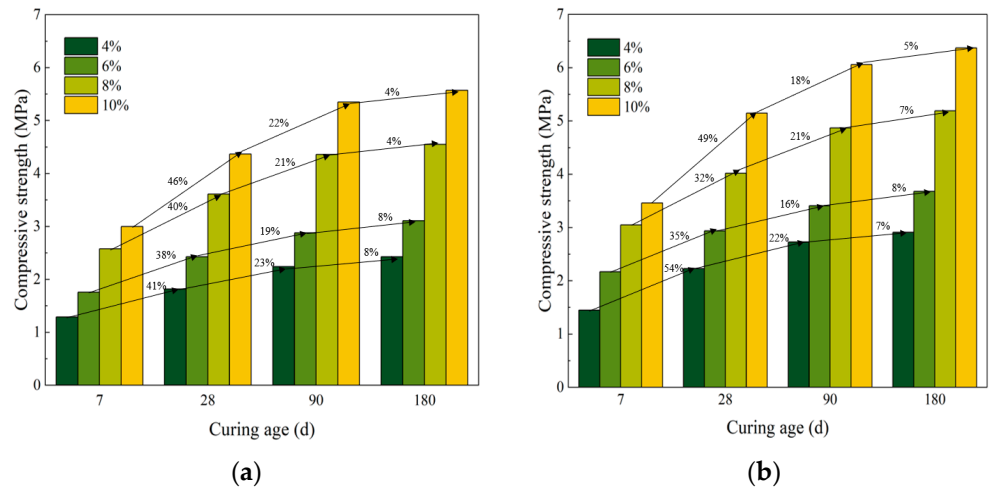


Figure 3. Development of compressive strength with curing age. (a) Stabilizer A; (b) stabilizer B.

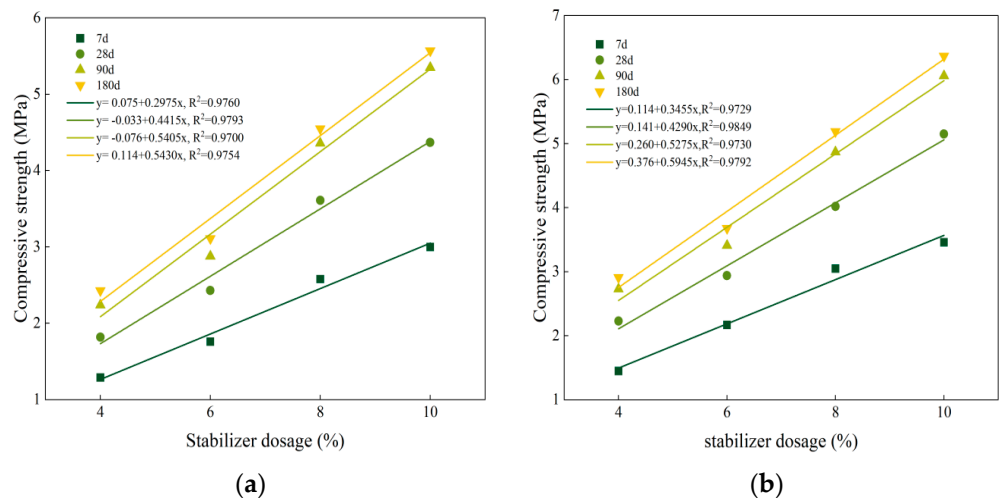


Figure 4. The linear relationship between stabilizer dosage and compressive strength. (a) Stabilizer A; (b) stabilizer B.

Figure 5 shows the influence of the delay period on the 7- and 28-day compressive strengths with various stabilizer dosages. Both the 7- and 28-day strengths declined with the prolonged delay period. This could be explained by the fact that a time delay could increase the density of macropores in the soil samples [28], which decrease the strength [29]. Strong linear relationships were found between compressive strength and delay time, with R^2 values ranging between 0.9631 and 0.9984.

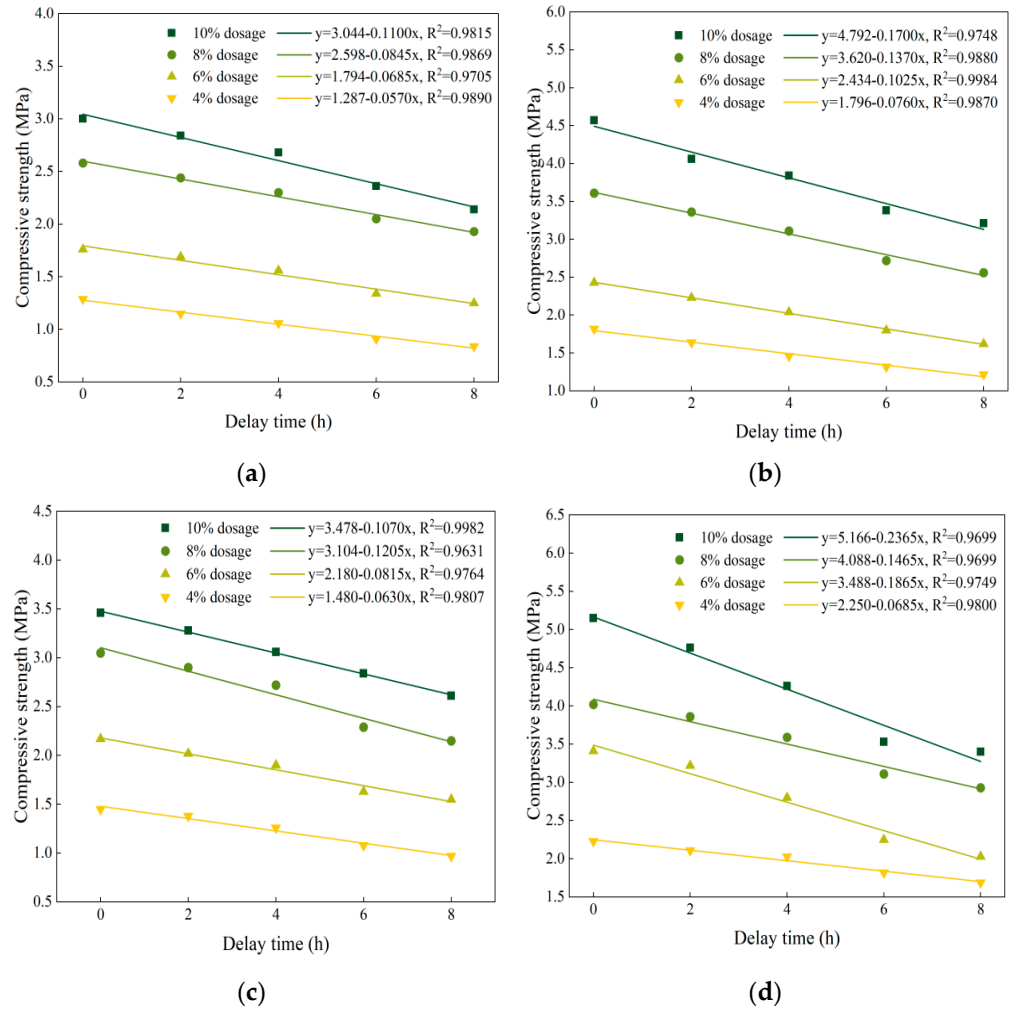


Figure 5. Effect of stabilizer dosage and delay period on compressive strength. (a) 7-day strength of stabilizer A; (b) 28-day strength of stabilizer A; (c) 7-day strength of stabilizer B; (d) 28-day strength of stabilizer B.

The influence of stabilizer proportion and delay time on compressive strength is shown in Figure 6. As regulated in JTG D50-2017, the 7-day strength of stabilized soil should be greater than 0.8 MPa when used for bases and subbases [30]. Figure 6 shows that both stabilizers A and B could satisfy the standard even after 8 h of delaying, which offered sufficient time for the operation at the construction site.

Figure 6b shows the 28-day compressive strength of A and B with a stabilizer dosage of 10%. When the delay time was shorter than 2 h, the decrease in strength in stabilizer A is slightly more than that of stabilizer B. This was probably because the cement in the stabilizer was in the initial setting state during that period. Static compaction destroyed the newly formed bond structures between soil particles and hydration products [31]. Stabilizer B had more cement in the stabilizer; therefore, the decrease in strength is more notable. When the delay time was longer than 2 h, the decrease in strength in stabilizer A is smaller than that of stabilizer B. This could be due to the fact that stabilizer A contained

more FGD gypsum. According to Raja et al. [32], sulphate could slow down the pozzolanic reaction. Thus, the strength development of stabilizer A was delayed, and correspondingly, the strength of stabilizer A was less disturbed by the delay period. Furthermore, according to X. Pu et al. [33], the pozzolanic reaction contributed to strength development at a later stage than cement hydration. It could, therefore, be inferred that stabilizers with a higher solid waste supplementary content could compromise the adverse effect of delay periods on strength, especially when the delay period was longer than 2 h.

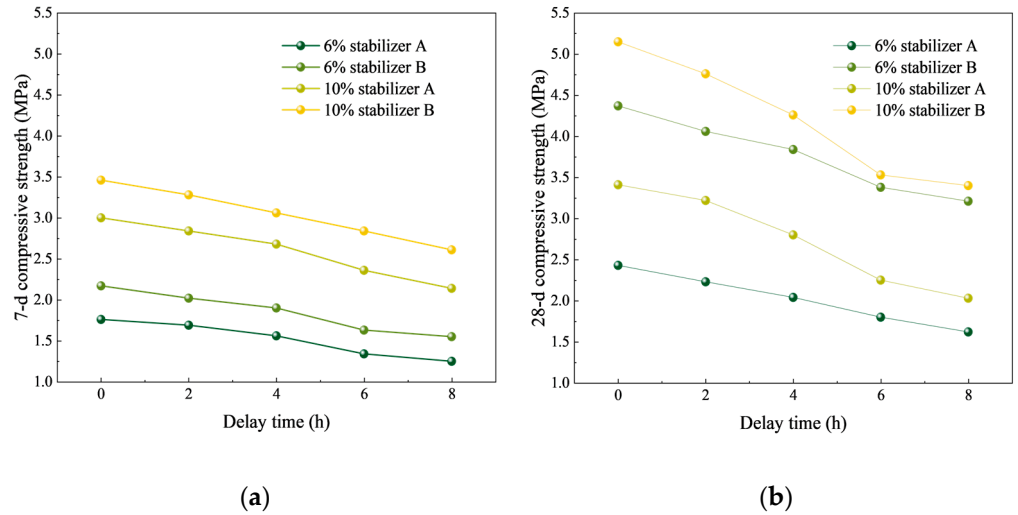


Figure 6. Effect of stabilizer proportion and delay time on compressive strength. (a) The 7-day compression strength for both 6% and 10% stabilized soils; (b) the 28-day compression strength for both 6% and 10% stabilized soils.

3.2. CBR Results

Figure 7 shows the CBR value of the stabilized soil with a 4% stabilizer content. It can be seen that the CBR values of all stabilizers are above 70%, which is far higher than the 8% that is required for the upper base of expressways and first-class highways, as specified by the Chinese standard JTG D30-2015 [34]. The CBR of the stabilized soils in previous relevant studies is presented in Table 3. Although the MDD and OMC are similar to those reported in previous studies, the CBR obtained in the current study is much higher.

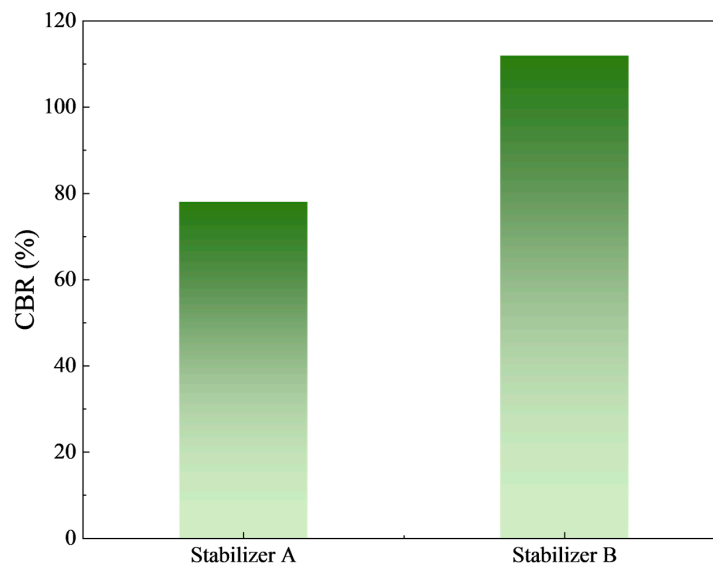


Figure 7. CBR of stabilizers A and B.

Table 3. Comparison of CBR values in different studies.

Relevant Works	MDD	OMC	CBR
Muthu Lakshmi S. et al. [35]	2.009 g/cm ³	12.63%	less than 6.02%
A. Bharath et al. [36]	1.59–2.87 g/cm ³	12.76–19.4%	1~3%
Anjali Gupta et al. [37]	1.64 g/cm ³	13.5%	less than 15%
Haibin Wei et al. [38]	1.93 g/cm ³	12.2%	0.9–44.9%
The current study	1.84 g/cm ³	15.3%	more than 70%

In addition, when the stabilizer dosage was 4%, stabilizer B's CBR value reached 112%. According to the Technical Guidelines for Construction of Highway Roadbases JTG/T F20-2015 [39], it can, therefore, be used in the subbase under the conditions of first-class highways and heavy-traffic-load levels.

3.3. Temperature Shrinkage

Figure 8 shows the variation in temperature shrinkage coefficients and strain with temperature. The temperature shrinkage coefficient decreased with rising temperature. When the temperature was below 0 °C, the decrease is more apparent than when it was above 0 °C. Furthermore, stabilizer B had a smaller temperature shrinkage coefficient than A, which was due to the higher cement content. The hydration of cement produces calcium hydroxide, which is essential to the pozzolanic reaction of slag and fly ash [40,41], and, thus, generates a larger amount of ettringite. Its volume expansion can offset part of the temperature shrinkage of stabilized soil [42].

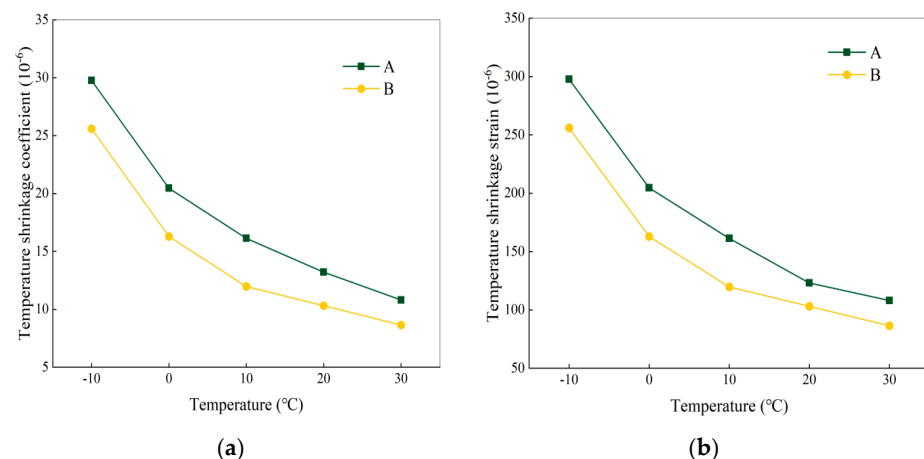


Figure 8. The variation of temperature shrinkage coefficient and strain with the temperature. (a) The relationship between temperature shrinkage coefficient and temperature; (b) the relationship between temperature shrinkage strain and temperature.

3.4. Water Stability

Figure 9 shows the 28 d and 90 d water stability coefficients of samples with different stabilizer dosages. A larger coefficient means better resistance to water immersion.

The water stability coefficients of the specimens were all less than 1. This was because water entered the pores of the soil, broke the bond between soil particles [43], and dissolved some of the hydration products (such as calcium hydroxide). When the soaking time increased from 28 d to 90 d, the coefficients showed a downward trend. This is possibly because the rate of hydration product formation was slower than that of the water erosion of the soil sample [44]. Furthermore, it was also found that a high stabilizer dosage could reduce the magnitude of decrease in the water stability coefficient. A higher dosage would generate more hydrates, which could strengthen the bonds between the soil particles [45]. This phenomenon was more noticeable in stabilizer A; when the stabilizer dosage was 10%, the 90-day water stability coefficient of stabilizer A even overtook that of 28 days. This

could be due to the additional hydrates obtained from the pozzolanic reaction, since the reaction contributed to strength at a late age [33].

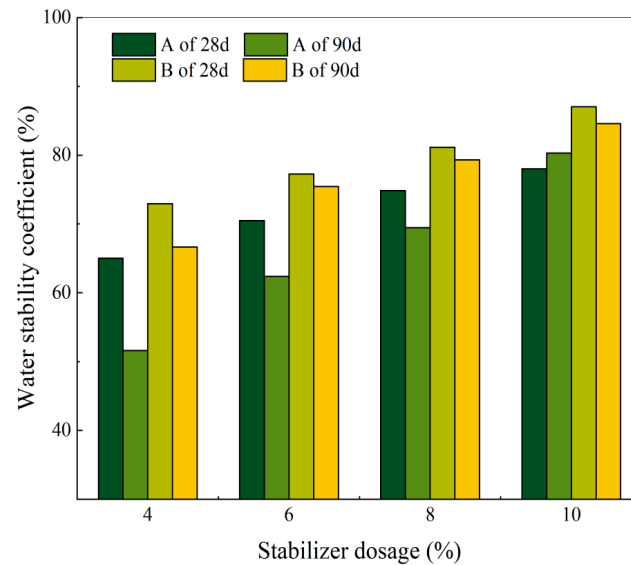


Figure 9. The relationship between water stability coefficient and stabilizer dosage.

3.5. XRD Analysis

Figure 10 shows the XRD analysis of samples using stabilizer A; different dosages (4% and 10%) and different curing ages are compared herein. Minerals of quartz (SiO_2), alumina (Al_2O_3), calcium carbonate (CaCO_3), ettringite, and calcium silica hydrates (C-S-H) were detected in the stabilized soil. And the hydration products of all stabilizer dosages and curing ages was similar. When the stabilizer dosage and curing age increased, the intensities of ettringite and C-S-H became stronger, while the intensities of SiO_2 became weaker. The reason for this phenomenon could be explained as follows: first, the hydration of cement could consume an amount of SiO_2 and produce calcium silica hydrates. Besides, the Si-O bond of fly ash could be broken in alkaline environments and then reacted with OH^- , Ca^{2+} , SiO_2 , and FGD gypsum to generate calcium silica hydrates [46]. The interaction of AlO_4^{2-} ions with the SO_4^{2-} and Ca^{2+} ions in FGD gypsum led to the formation of calcium sulfoaluminate hydrates, a part of which could react with FGD gypsum to produce ettringite.

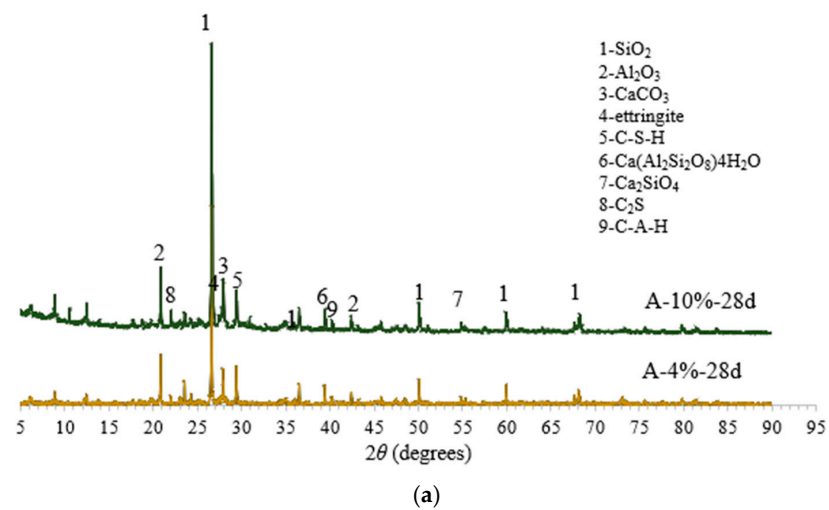


Figure 10. Cont.

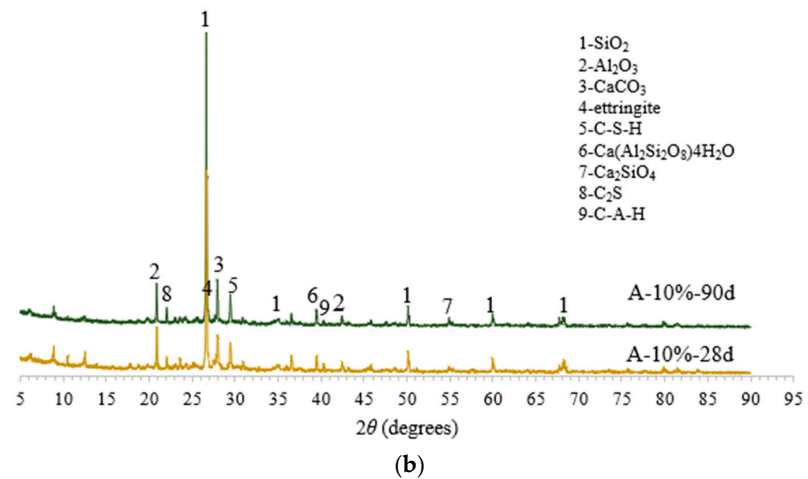


Figure 10. Graph of X-ray diffraction analysis. (a) Influence of stabilizer dosage; (b) influence of curing age.

3.6. SEM Analysis

As can be seen in Figure 11, it was apparent that specimens with a higher stabilizer dosage had more hydration products. When the stabilizer dosage was 4%, soil particles and voids between particles could be easily observed. When the dosage reached 10%, the exterior of the soil particles was covered by calcium hydro silica gel, ettringite, and other hydration products. With increasing curing time, the compactness and uniformity of the samples began to improve continuously, which contributed to the growth of strength and CBR.

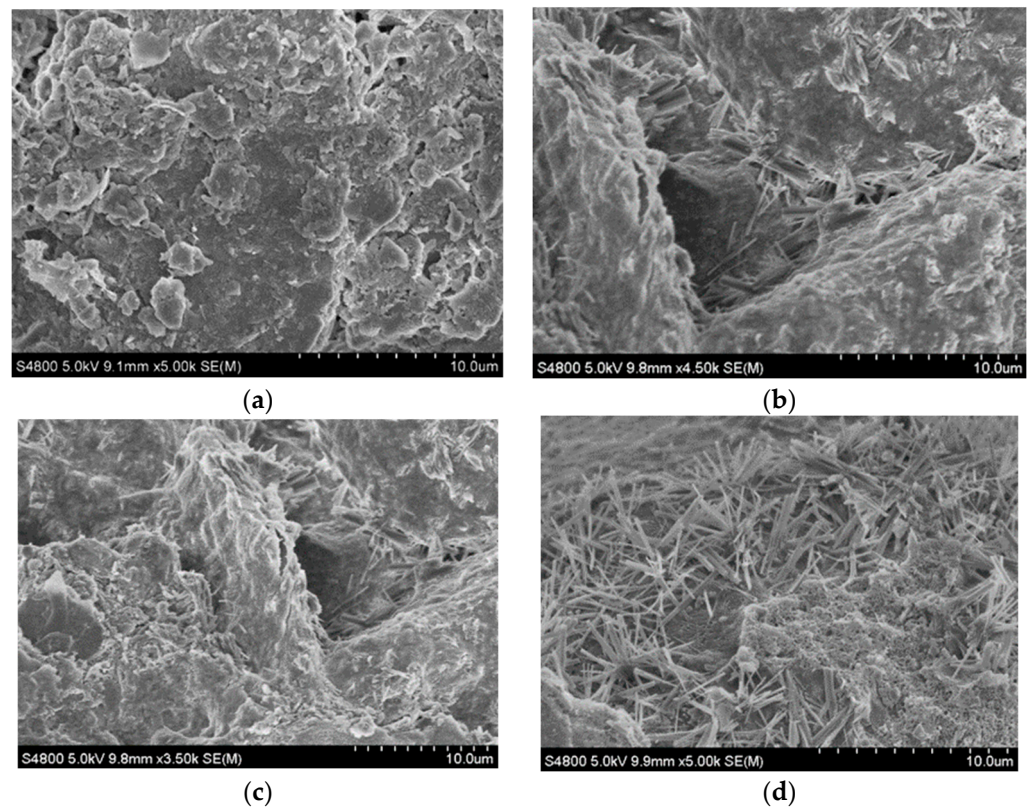


Figure 11. SEM images of stabilized soil specimens with stabilizer B. (a) 28 days, 4% stabilizer dosage; (b) 90 days, 4% stabilizer dosage; (c) 28 days, 10% stabilizer dosage; (d) 90 days, 10% stabilizer dosage.

4. Conclusions

Two mixtures with a low cement content were proposed and used as silt soil stabilizers, i.e., stabilizer A (cement:ground GBFS:fly ash:FGD gypsum = 30:44:15:11) and stabilizer B (cement:ground GBFS:fly ash:FGD gypsum = 40:38:13:9). The compressive strength development, CBR and temperature shrinkage, mineral compositions, and micromorphology of the stabilized soil were measured. The following conclusions can be drawn:

- The compressive strength increased with the stabilizer dosage, and they showed a strong linear relationship. Longer delay times led to a lower strength. Notably, stabilizers containing large amounts of solid waste mitigated the adverse effect of the delay time on compressive strength. When the stabilizer dosage was 6%, both stabilizers A and B satisfied the standard even after 8 h of delay, which offered sufficient time for the operation at the construction site.
- Both stabilizers with a 4% dosage significantly improved the CBR. The specimens were soaked in water for 96 h, and their CBR value reached 60%, which was about seven times higher than the one (i.e., 8%) specified in JTG D30-2015.
- The temperature shrinkage coefficient decreased with the increase in temperature. The cement and solid waste in the stabilizer contributed to the hydration of the system. The formation of ettringite within the system caused volume expansion, reducing the harm caused by temperature shrinkage.
- The water stability coefficient decreased as the immersion time increased. Such a reduction could be mitigated by increasing the stabilizer dosage. When the stabilizer dosage of stabilizer A (30% cement content) reached 10%, the 90-day water stability coefficient was higher than that at 28 days.
- Quartz (SiO₂), alumina (Al₂O₃), calcium carbonate (CaCO₃), ettringite, and C-S-H were detected in the XRD analysis. When the stabilizer dosage and curing age increased, the intensities of ettringite and C-S-H became stronger. Soil particles covered by C-S-H, ettringite, and other hydrates could be observed in the SEM images.

Author Contributions: Conceptualization, H.L. and W.M.; methodology, S.Z. (Shengya Zhou); software, H.Z.; validation, K.W., Y.F. and S.Z. (Shengtao Zhang); formal analysis, H.L. and S.Z. (Shengya Zhou); investigation, W.M.; resources, K.W.; data curation, H.Z.; writing—original draft preparation, H.L.; writing—review and editing, W.M. and S.Z. (Shengya Zhou); visualization, H.Z.; supervision, K.W.; project administration, Y.F.; funding acquisition, K.W. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: Author Haijun Li, Kai Wang, Shengtao Zhang, and Hanming Zhang were employed by the company Shandong Hi-speed Group. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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