Solidification/Stabilization of Textile Sludge as Subgrade: Usage of Binders and Skeleton Material

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1. Introduction

As the demand of textile products increases, textile wastewater derived from the textile industry constitutes a large proportion of the industrial wastewater around the world. The textile wastewater was disposed by biochemical and physical methods, resulting in a lot of textile sludge over time [1]. Textile sludge has poor engineering properties of high moisture content, high organic content, high volume, causing decay odor, minimum particle, good fluidity, and the presence of flocculation state [2]. Furthermore, textile sludge has a complex constitution, containing organic pollutants and heavy metals. The sludge accumulated by occupying land could cause land waste and trigger eco-environmental problems. It is highly contaminative and harmful for both public health and environment [3]. Considering these characteristics, a lack of safe disposal of textile sludge will lead to severe secondary pollution of the environment. Therefore, a scientific and economic disposal of the textile sludge becomes a worldwide problem.

Solidification/stabilization (S/S) technology is an effective, economic feasible, and environmental protection sludge disposal method [4]. Currently, in situ S/S technology has been applied to large-scale engineering, such as soft foundation treatment and contaminated soil disposal [5, 6]. The disposal method avoids excavating and backfilling and thus saves both money and time. S/S technology can translate weak sludge into encapsulated solid and stabilize the chemical properties of contaminants by adding binders and, thus, to overcome the difficulties of the weak strength...
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2.1 Textile Sludge. This is a stacking area for textile sludge (110 m long, 50 m wide, and 2 m deep) to be solved in a highway construction in Zhejiang, China. The sludge needs to be excavated and replaced by other filling material in the original design. This would increase construction time and cost as well as environmental problems caused by transporting textile sludge. Samples of textile sludge below a depth of 0.2 m were collected by an excavator after removal of bare surface sludge. The samples were filled into sealed containers and preserved in the dark (25 ± 5°C in temperature and more than 80% in humidity) to prevent dehydration and decomposition. The surface of the samples is black-yellow, while the interior is black (Figures 1(a) and 1(b)). The physical properties of the sludge are presented in Table 1. The moisture content was determined by the oven drying method with a temperature of 65–70°C, since the organic matter content is above 5% [30]. Solid matter is concatenated by the mitoplast after being dried (Figure 1(c)). The ignition method was adopted to determine the organic matter content. The dried sludge samples catch bright fire with black smoke when being put into a high-heat muffle furnace (Figure 1(d)). The chemical oxygen demand (COD) was measured by the dichromate method following the guideline of GB/T11914-1989 [31].

Moreover, hydration reactions will be obstructed by the multiorganic matter in the sludge, which affect the curing effect and thus delay both the setting time and strength formation [26, 27]. Therefore, the drawbacks (high content of water and organic matter, lacking of solid particles) add the difficulty to stabilize textile sludge. Therefore, Li et al. [28] introduced the skeleton construction method to stabilize chemistry sludge by adding binders and inorganic solid materials. The aim is to increase the inorganic solid content in the sludge, thus dilute the negative impact of high water and organic matter. This furthermore constructs the structure under the cementation of cement hydrates, thus consequently improves the strength. Some studies focused on industrial by-products, such as lime and fly ash [16]. However, if only industrial by-products were added, a large quantity of them are required to meet the strength requirements. This will cause a significant additive fee. Therefore, research studies have concentrated on the partial substitution of waste or supplementary materials for binders in order to reduce the binder cost. Several experimental studies used supplementary material such as silt, bentonite [28], and clay bricks [29]. However, the differences and the type choices of the skeleton materials have not been studied. This study investigated the method to reuse textile sludge as construction material in the laboratory and applied it to a practical subgrade project. In the laboratory, UCS tests were conducted to analyze the strength differences among several proportions mixtures, thus seeking an economical proportion for the solidification of textile sludge. Scanning electron microscopy (SEM) tests were to explore the strength formation mechanism of the physical microstructure. Evaluate the stability of the metal elements and organics in the mixture by leaching tests. Finally, field tests were conducted to compare the results obtained in the laboratory and to further validate the feasibility of S/S textile sludge for subgrade.

2.2 Binder Materials and Skeleton Materials. In this study, ordinary Portland cement (OPC 42.5) and quicklime were used as the main binders. Fly ash, GGBS, and gypsum were used as auxiliary additives. The corresponding chemical ingredient analysis (determined by a spectrophotometer) of these materials is presented in Table 2.

The moisture content of sludge is over 500% and far more than the usual waste sludge. Cheap and convenient local soil is considered as skeleton material. Three different soil types were chosen in this study. The particle content and specific gravity, as shown in Table 3, were obtained via the standard test method (ASTM D6913; D7928; D854). Atterberg limit tests were conducted following ASTM D4318. The results indicate these soil materials, respectively, belong to high liquid limit clay (CH) (IP ≥ 0.73 × (WL-20) and IP ≥ 7, WL ≥ 50%), low liquid limit clay (CL) (IP ≥ 0.73 × (WL-20) and IP ≥ 7, WL < 50%), and low liquid limit silt (ML) (IP < 0.73 × (WL-20) or IP < 4, WL < 50%) (ASTM D2487).

2.3 Test and Method

2.3.1 Unconfined Compressive Strength (UCS) Test. The specific program of UCS test is shown in Table 4. The figures in the table are the weight ratio of the materials to textile sludge. The binders, skeleton material, and sludge were proportionally weighed into a blender and mixed for about 15 min to achieve maximal uniformity. The mixture was compacted by vibration in cylindrical molds with 50 mm in diameter and 100 mm in height (height-to-diameter ratio is between 2.00 and 2.50 following ASTM D5102). The molds were then wrapped with plastic film and stored in a curing container with 20 ± 2°C in temperature and 90% in humidity for 7 or 28 days. Specimens were prepared and duplicated into six pieces. After being taken out of the mold, the specimens were objected to USC tests via unconfined pressure apparatus (TKA-WCY-1) with a constant strain rate of 2 mm/min. The UCS tests were performed on all...
specimens, as shown in Table 4. Each specimen was loaded until the peak stress was obtained, following the guideline of JGJ/T 233 [32]. The values of UCS were obtained by calculating the average of three specimens.

2.3.2. Leaching Test. Textile sludge has other complex constitution, containing dye, sizing agents, and additives. These substances bring persistent organic pollutants (POPs) degraded difficultly into the textile wastewater, such as polycyclic aromatic hydrocarbon (PAHs) and absorbable organic halogens (AOX). To further evaluate the stability of the pollutants in the S/S sludge, extraction procedure for standard of HJ/T 299 [33] was implemented. Subgrade samples obtained from three different depths were taken to the laboratory for leaching tests as well. First, the samples (raw textile sludge, mixtures of 1.8CL+0.2C and 1.8CL+0.2C+0.04B, and subgrade samples) were crushed and sieved (2 mm); with a liquid-solid ratio of 10:1 (L/kg), the crushed sample was moved in an extraction bottle before the extraction solvent (diluted solution of sulphuric acid and nitric acid as proportional) was added. Second, the extraction bottle was oscillated 18 h. Finally, the supernatant was filtered for the next determination. In this study, the metal elements were determined using inductively coupled plasma-mass spectrometry (ICP-MS) [34]. PAHs
2.3.3. X-Ray Fluorescence (XRF) Test and Scanning Electron Microscopy (SEM) Test. XRF test can quickly measure the main metal elements or other elements of solid, fluid, and liquid outdoor. For this study, a DELTA hand-held XRF analysis meter was used to measure the main elements of the skeleton materials (CL and HL) to explore the clay mineral. The results are shown in Table 5. The silicon and aluminum contents of CH are higher than that of CL. The clay mineral (main mineral composition of the clay particle) is hydrated aluminum silicate mineral. Soil with higher plasticity index or liquid limit always contains more clay mineral [37]. This is consistent with the results.

SEM tests were implemented on samples of raw textile sludge and S/S sludge mixture (1.8CL + 0.2C + 0.04B) after seven days of curing. The samples were sliced to less than 2 mm in height, ensuring a horizontal bottom of the slices. The slices were dried at low temperature (40°C) to avoid the deformation caused by surface tension of water during drying, stuck onto aluminum stubs, covered with gold, and then observed under Hitachi S-4700 SEM.

2.3.4. Field Test. In situ S/S technology was adopted to process the textile sludge as subgrade by using a professional equipment containing an automatic quantitative feeding system and a mixing system. First, the sludge was divided into 5 × 5 m processing areas. Second, skeleton material was calculated and evenly put on the sludge. The feeding system of binder material was set at designed content. Then, every divided area was mixed up and down by the mixing system, which ejected binder material when running. Finally, the surface was cleaned and levelled. After 28 days, in situ vane shear tests, following the guideline of SL237–044 [38], were conducted to compare the results of the laboratory tests. The project design report indicated that the characteristic value of bearing capacity for the subgrade should be greater than 100 kPa. Thus, plate-bearing tests were performed to examine the bearing capacity of subgrade [39].

3. Results and Discussion

3.1. UCS Comparison of S/S Mixture with Binder Materials. The relationship of the specimens UCS (Qu) (cured for 28 days) versus the content of cement and quicklime is shown in Figure 2. There is a significant approximate linear growth tendency between UCS and the dosage of quicklime or cement. The strength is 7–80 kPa with a dosage of 0.15–0.4 quicklime, which is higher than that with cement (4–41 kPa). Quicklime has a better effect than cement to solidify the raw sludge.

UCS of cement-soil in common engineering always shows a more reliable than quicklime [40, 41]. However, the comparison results for the strength in this study reverse other study’s results [42]. For cement and quicklime, there is some difference in chemical reactions with sludge resulting from the difference chemical composition proportions. Cement containing oxides of calcium, silica, alumina, and iron [43] can cause hydration reaction with water and lead to cementitious products such as calcium silicate hydrate (C-S-H, C-A-H ... ) [44, 45]. Cement solidifies soil through the intergranular cementation and pore filling by the hydration products. For quicklime, CaO, as the main ingredient, initially reacts with the water in the sludge to reduce the water content. This generates calcium hydroxide (Ca(OH)₂) and releases heat to promote chemical reaction. Later, the pozzolanic reaction between Ca(OH)₂ and the active constituents of quicklime in the presence of water leads to the formation of CSH and CAH [46]. Generally, lime stabilizes clay through flocculation and cementation mechanisms to improve the workability and its bearing capacity [47]. However, in this study, a huge amount of water (ω > 500%) causes few solid particles in the textile sludge. There are not enough solid particles in per unit volume to satisfy the cementation of hydration products. The cementation from cement cannot be compared with the reduction of water content and cementation from quicklime.

UCS increases with the increase of fly ash and GGBS (Figure 3(a)). GGBS as auxiliary additive shows a better treatment effect than fly ash to solidify textile sludge. GGBS (containing 16 main PAHs defined by USEPA) were determined by high performance liquid chromatography (HPLC) [35]. The microcoulometric method was used to test AOX [36].

### Table 4: Specific program of UCS test (g/g).

| Material and weight | Cement (C) | Quicklime (L) | GGBS | Fly ash (F) | Gypsum (B) | Skeleton soil |
|---------------------|------------|---------------|------|------------|------------|--------------|
| Cement (C)          | 0.15; 0.2; 0.3; 0.4 | —          | —    | —          | —          | —            |
| Quicklime (L)       | —          | 0.15; 0.2; 0.3; 0.4 | —    | —          | —          | —            |
| GGBS                | 0.05       | 0.1; 0.2      | 0.15; 0.2; 0.3 | —          | —          | —            |
| Fly ash (F)         | 0.05       | 0.2          | —    | 0.15; 0.2; 0.3 | —          | —            |
| Gypsum (B)          | 0.05       | 0.2          | —    | —          | 0.04       | —            |
| Skeleton soil       | 0.2        | —            | —    | —          | —          | 0.02         |

### Table 5: Results of XRF test.

| Chemical element | Si (%) | Al (%) | Fe (%) | Mg (%) | Ca (%) |
|------------------|--------|--------|--------|--------|--------|
| CL               | 17.52  | 6.08   | 5.66   | 4.01   | 2.99   |
| CH               | 20.77  | 6.42   | 5.06   | 2.25   | 2.22   |

Figures present the weight ratio of materials to textile sludge and the units is 1.
and fly ash are widely used as cement additives to improve the engineering performance of cement-soil [48]. The main ingredient of fly ash is silicon dioxide (SiO$_2$) and a lesser amount of aluminum oxide (Al$_2$O$_3$) than GGBS. And GGBS is constituted by CaO, SiO$_2$, and more Al$_2$O$_3$. Siliceous and alumina-siliceous based additives require the presence of an alkaline activator, for example, Ca(OH)$_2$ or CaO, for pozzolanic reactions to proceed [49–51]. Fly ash is pozzolanic material but has no cementitious value by itself; thus, it cannot be used as binder alone. While, GGBS is hydraulic material and therefore requires no additives for hydration and hardening to take place [52]. Therefore, as the additive increases, the strength disparity for specimens with GGBS and fly ash grows.

UCS increases to a peak when the dosage of gypsum content is 0.04 (Figure 3(b)). Without and with fly ash, growth rates are 50% and 110% for seven days, and 290% and 300% for 28 days, respectively. However, UCS slightly

**Figure 2:** Relationship of the specimen strength versus cement and quicklime content.

**Figure 3:** Specimen strength versus content of auxiliary additive. (a) GGBS and fly ash. (b) Gypsum (bassanite) (B).
decreases after the peak. By adding gypsum with only one-tenth the amount of GGBS or fly ash, the strength of mixture can increase to a same or higher strength. Earlier, research suggested that calcium sulphate is a successful activator to create an appropriate environment for the reaction process without necessarily participating in the reaction [53]. Later, research shows that gypsum makes carbonization more sufficient in early curing time [23], which causes a significant growth in strength. Furthermore, the hydration of C3A reacts fast to produce ettringite (Aft) finally with sufficient gypsum [54, 55]. The microstructure will be discussed in SEM test results. In macroview, gypsum as additive shows significant effect on the strength growth for solidifying textile sludge.

In conclusion, although with cement or quicklime added even to 40% or with other additives added, the UCS of mixture is far less than 400 kPa, which does not meet the strength requirement for subgrade [56].

### 3.2. UCS Comparison of S/S Mixture with Binder and Skeleton Materials

The UCS comparison of S/S specimens after adding same proportional skeleton materials (CH, CL, and ML) is shown in Figure 4(a). In addition, the skeleton materials also have approximate moisture content (10% left and right). The particles of skeleton material and sludge form a complete structure by cementitious products from binders. The strength is improved significantly after adding any soil material. And the highest strength is the mixture with CH, followed by CL, and the mixture with ML is the lowest. A higher plasticity index of the skeleton material has a better impact on the strength of S/S mixture. The clay mineral, particularly montmorillonite, can adsorb water and transform into other ionic or crystalline modality [37]. The higher the clay mineral in the material is, the more water material sorbs, and thus to increase the solid-liquid rati of sludge. Li et al. [28] adopted silt (liquid limit 73.4% and plastic limit 29.3%) and calcium-bentonite (main composition is montmorillonite) to build the frame and to process city sewage sludge via the S/S method. The selection of skeleton material is important for the strength of the S/S sludge.

After adding CL, the moisture content of mixture changes, which is marked with brackets in Figure 4(b). The method for calculation is summarized as

$$ r = \frac{V_m}{V_t} = \frac{m_t + m_b + \rho_t}{m_t + m_b} \times \frac{\rho_m}{\rho_t}, $$

where $V_m$ and $V_t$ are the volume of mixture and textile sludge, respectively, $m_t$, $m_b$, and $m_s$ are the mass of textile sludge, binder, and skeleton material, respectively, and $\rho_t$ and $\rho_m$ are the density of textile sludge and mixture, respectively. Enlargement ratio ($r$) of each S/S mixture can be calculated by the dosage of material and the density of S/S mixture.

The relationship of $r$ and moisture content of CL is shown in Figure 5. The enlargement ratio of sludge increases from 1.945 to 2.089 with the increase of moisture content, but still remains around 2.0. That means the volume of textile sludge increases 100% after adding material with the dosage ratio of 1.8 CL (with different moisture contents) + 0.2C + 0.02B. The textile sludge in this study, lying in a pond and lower than the surrounding area, is designed to be used as subgrade. The extra subgrade material can be used in other low-lying areas. While, the strength of mixture after adding CL with 10% moisture content is 462 kPa, and 104 kPa with 38% moisture content. The difference in strength is more worth considering than the increase in the enlargement ratio.

### 3.3. Enlargement Ratio of S/S Mixture

After adding so much skeleton material and binders, the change in volume of mixture is worthy consideration while the strength requirement is satisfied. Enlargement ratio ($r$) is defined to express the change in volume of sludge. Assuming the conservation of mass before and after curing, the enlargement ratio of sludge ($r$) can be calculated by

$$ r = \frac{V_m}{V_t} = \frac{m_t + m_b + \rho_t}{m_t + m_b} \times \frac{\rho_m}{\rho_t}, $$

where $V_m$ and $V_t$ are the volume of mixture and textile sludge, respectively, $m_t$, $m_b$, $m_s$ and $m$ are the mass of textile sludge, binder, and skeleton material, respectively, and $\rho_t$ and $\rho_m$ are the density of textile sludge and mixture, respectively. Enlargement ratio ($r$) of each S/S mixture can be calculated by the dosage of material and the density of S/S mixture.

### 3.4. SEM Tests

The scanning electron micrographs of both raw sludge and S/S mixture (1.8CL + 0.2C + 0.04B) are shown in Figure 6. The micrographs of raw textile sludge show a complex microstructure with lamellar, block, and particle substance (Figure 6(a)). Figure 6(b) presents a unique reticulate microstructure cemented by needle-like crystal substance. The interlaced needle column crystal substance is Aft [55]. The microstructure of the textile sludge is loose with independent particles, without binding power between them. This leads to a poor performance in
Figure 4: Strength of S/S specimens constructed by skeleton material: (a) skeleton materials (ML, CL, and CH) with approximate moisture content and (b) different moisture contents of the CL.

Figure 5: Enlargement ratio of textile sludge after S/S treatment.

Figure 6: Micrographs (SEM) at 30000 magnification of (a) raw sludge and (b) S/S mixture.
strength. After adding binders and CL, skeleton material improves the amount of solid particle in unit volume to reduce the intergranular porosity. And the cementitious products from binders further fill the pores. Furthermore, AFt cement the particles and form a particular space grid structure. The physical microstructure of sludge has been greatly improved, which makes its macroscopic strength remarkably improve. The SEM test provides a microcosmic perspective for the observation of the microphysical structure and accounts for the mechanism of strength growth.

3.5. Leaching Tests. The results of the leaching tests are listed in Table 6. The metal elements, PAHs, and AOX in leaching liquid of raw sludge are below the standard values of the groundwater quality requirements for agricultural and industrial use [57]. The standard values of PAHs and AOX are the sum of the standard values of each substance. The metal, PAHs, and AOX in leachate of S/S mixture are further decreased after S/S treatment. Gypsum is useful to stabilize Zn and Cd in particular, but no use for Ni and As. The concentrations of Cr and Cu increase with gypsum added. In this study, the risk of environmental contamination from sludge leachate is below the standard after S/S disposal of textile sludge.

3.6. Field Tests. According the results of the laboratory tests, the strength of sludge solidified by binders (cement and gypsum) and CL can meet the requirements of engineering practicality. The material ratio of 0.2C + 0.02B + 1.8CL (approximately 10% in moisture content) is adopted to S/S the textile sludge for highway subgrade. After 28 day, field tests are performed. In situ vane shear tests are performed on ten different depths of four representative test sites (A–D). Static plate-bearing tests are implemented on the subgrade surface of other two randomly selected sites (E and F). The vane shear strength (Cu) of the subgrade is shown in Table 7 and the bearing capacity is shown in Table 8.

Cu shows a decreasing trend with the curing depth. The difference strength in depth is maybe due to the uneven mixing of field construction. The shear strength of mixture in laboratory test is 231 kPa, which is half of UCS (462 kPa). The average of vane shear strength for the subgrade is 223.6 kPa. The strength result from field tests is in good agreement with the laboratory test result.
As the standard of load test [39], the characteristic value of bearing capacity is defined as half of the ultimate load. The results show the subgrade ultimate load is 200 kPa. Thus, the characteristic value of bearing capacity of the subgrade is 100 kPa, which meets the design requirement. The results of in situ tests indicate that the shear strength and bearing capacity of this section subgrade after processing meet the design requirements.

4. Conclusion

An investigation of S/S technology on the disposal of textile sludge was conducted both in the laboratory and in the field. The results indicate the partial substitution of skeleton materials for parts of binders to be an effective, economic, and environmental solution to process raw textile sludge with high moisture content. The obtained conclusions are

1. The mixture with quicklime shows a higher strength (UCS) than that with cement. Compared to fly ash, GGBS as hydraulic material is preferable for the solidification of textile sludge. Gypsum as auxiliary additive plays a significant role in the hydration reaction. The mixture UCS after adding gypsum grows approximately 3-fold for 28 days.

2. Adding solid material as skeleton material to raw textile sludge decreases the liquid-solid ratio in mixture and make cementitious products work better. In the laboratory, UCS of the mixture after adding skeleton material improves from 80 kPa to more than 400 kPa. In field, the shear strength and bearing capacity of the practical subgrade also meet the design requirements.

3. SEM tests indicate the physical microstructure of sludge is greatly improved after forming a particular space grid structure by cementitious products, which makes its macroscopic strength remarkable to improve. The metals and hazardous organics in the leachate are below the standard value and are further decreased after S/S treatment.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Additional Points

UCS of binder-sludge grows 3-fold with gypsum and increases from 80kPa to 400 kPa with skeleton material. Physical microstructure of textile sludge is greatly improved by the particles and cementitious products. Strength of field test is consistent with the lab result and meets the design requirement. Metal, PAHs, and AOX in leachate are decreased after S/S treatment and below the standard value.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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