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Mechanical Characteristics and Micro-Mechanism of Modified Dredged Sludge Based on Calcium-Containing Solid Waste Used as Landfill Cover Materials

Kejian Shang

School of Management, Henan University of Urban Construction, Pingdingshan 467036, China; 20181031@hncj.edu.cn

Abstract: In order to prepare a new type of landfill covering material for closure, we used industrial calcium-containing waste (construction rubbish, slag, desulfurized gypsum and fly ash) to modify the dredged urban sludge. Shrink, unconfined compression, shear and infiltration tests were performed to obtain the volume shrinkage, compressive strength, shear strength and permeability coefficient of the modified sludge, as well as the permeability coefficient under the action of wet and dry cycles. Comprehensive characterization of the modified sludge using X-ray diffraction, Fourier-transform infrared spectroscopy, and scanning electron microscopy with energy dispersive spectroscopy detection methods, resulted in the hydration products, molecular groups and microstructure characteristics of the modified sludge and revealed the modification mechanism of calcium-containing waste to sludge. After natural curing for 28 d, the volume shrinkage rate of the modified sludge sample was 2.6~8.3%, the unconfined compressive strength was 7.9~14.5 MPa, the cohesion force c was 179~329 kPa, and the internal friction angle φ was 42.59~53.60°. After six wet and dry cycles, there were no cracks in the modified sludge; the permeability coefficient of the modified sludge reached stability at $0.84–11.1 \times 10^{-7}$ cm/s; and the permeability coefficient of MS7 sample was less than 1×10^{-7} cm/s, which met the engineering anti-seepage requirements of the landfill closure cover. The industrial calcium-containing waste by alkali formed C–S–H and C–A–S–H gelled geopolymer, which filled the gaps between soil particles to form a strong soil cement skeleton. Therefore, the mix ratio of sludge:construction waste:slag:fly ash:desulfurized gypsum was 50:22:15:8:5. Calcium-containing solid waste modified sludge can be used as a cover material for landfill closure.

Keywords: dredged sludge; calcium-containing solid waste; landfill cover; anti-seepage performance; modification mechanism

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1. Introduction

Naturally deposited sludge in dynamic and static water environments is extensively distributed in rivers and lakes around the world. In China, the volume of dredged sludge generated annually is quite large. Statistics indicate that more than 20 billion m^3 of dredged sludge is currently deposited in rivers and lakes in China and is increasing at a rate of about 160 million m^3 per year [1]. Dredged sludge, as a fine-grained soil rich in water content and clay content, has some organic matter and a small amount of organic or inorganic pollutants. With particles mainly arranged in honeycomb and flocculent fabrics, dredged sludge is characterized by high porosity, high compressibility, and low bearing capacity [2]. However, due to the poor natural engineering properties and high yield of dredged sludge, the safe disposal and resource utilization of sludge has become an imminent problem to be solved.

Constituting an integral part of the ecological barrier system in landfills, the closure and coverage system plays a crucial role in stopping rainwater infiltration and landfill gas leakage, and its safety is a key factor to guarantee the performance of the landfill [3]. Existing landfills adopt the closure and coverage system with compacted clay and an

HDPE geomembrane. However, according to the survey on landfill operation by the Ministry of Housing and Urban–Rural Development of the People's Republic of China, the traditional closure and coverage system with compacted clay has defects such as easy cracking and difficult soil extraction, while the closure and coverage system with the HDPE geomembrane has defects such as tearing and bulging. The cost of traditional covering materials is high, and there is a risk of deterioration of service performance in the landfill closure and covering system, which greatly improves the construction and operation cost of landfills. The landfill covering system of compacted clay and HDPE geomembrane does not conform to the development idea of low-carbon and green circular economy in China. Therefore, many scholars at home and abroad have generally started to turn their focus to the research and development of new landfill closure and coverage materials.

Taking dredged sludge as the base material, the sludge is modified and solidified by using such materials as cement, slag and fly ash to obtain the modified sludge closure and coverage materials for landfill, which is an effective method to realize the circular economy of solid waste disposal [4]. There are large reserves of dredged sludge and calcium-containing solid wastes, such as slag and gypsum, and it is convenient to obtain materials. Therefore, the construction cost of landfill covering system with modified dredged sludge based on calcium-containing solid waste is low, which is also in line with the development idea of national low-carbon emission reduction and green circular economy. Foreign scholars have carried out a large number of studies on the modification and solidification methods of sludge, as well as the microstructure and engineering mechanical properties of modified sludge materials. Numerous researchers used cement as the main curing agent, together with industrial wastes, such as fly ash, desulfurized gypsum and slag, to solidify the dredged sludge, and systematically analyzed the unconfined compressive strength, shear strength, consolidation characteristics, matrix suction and other mechanical indexes of saturated and unsaturated solidified sludge. By means of microscopic characterization, the action mechanism of solidification of sludge by curing agents, such as cement, was analyzed, and the dosage of curing agent was optimized [5–13]. As dredged sludge has high water content, high specific surface area and single soil particle size distribution, it is necessary to add a large amount of cement to achieve the expected strength of solidified sludge. Considering that cement is a fairly expensive building material with high energy consumption, there is an urgent need to find an environmentally friendly, efficient and low-cost curing agent for dredged sludge.

In recent years, researchers have attempted to solidify dredged sludge, using only industrial by-products (fly ash, steel slag/slag/alkali slag, phosphogypsum, etc.), and select the optimal mix ratio of sludge curing agent through the engineering indexes, such as compressive strength, shrinkage deformation, and water absorption, and discuss its micromechanism [14–17]. In addition, a few researchers have also explored the use of sludge incineration ash, active MgO, nano-silica and other materials to solidify dredged sludge; analyzed the strength growth characteristics of solidified sludge; and obtained the engineering characteristic indexes of solidified sludge as building materials [18–22]. The synthesis of the above research results revealed that cement, slag and fly ash are the main sludge curing agents at present, and the engineering properties of the mechanical strength of solidified sludge are widely concerned with the mechanical strength. However, the study of using construction waste combined with industrial solid waste to solidify dredged sludge has not been carried out, and the service performance of the modified sludge with calcium-containing solid waste (construction waste, slag/fly ash, etc.) as landfill covarage material needs to be evaluated.

In this present study, construction waste powder and industrial by-products (desulfurization gypsum, slag and fly ash) were used as curing agents to modify and cure dredged sludge in urban inner lakes so as to prepare landfill closure and coverage materials. The mechanical strength index of modified sludge was obtained through the shrinkage test, unconfined compressive test and shear test. The influence law of dry–wet cycles on the permeability coefficient of modified sludge was analyzed by means of the flexible wall permeability test for the purpose of evaluating the service performance of the modified sludge coverage material. The hydration products and microstructure of modified sludge with calcium-containing solid waste were analyzed by means of XRD, FT-IR, SEM + EDS, thus revealing the modification and solidification mechanism of modified sludge with calcium-containing solid waste. This study provides an important scientific basis for the resource utilization of dredged sludge and the construction of the landfill barrier system.

2. Test Materials and Methods

2.1. Test Materials

The dredged sludge used in the test was taken from East Lake Dredging Project in Wuhan, Hubei Province, China, with an initial moisture content of 53~55% and a dry density of 1.44 g/cm^3. The cement-based construction waste was crushed to obtain construction waste powder with a particle size of less than 0.1 mm. The desulfuration gypsum is a secondary flue gas desulfurization gypsum, which is white powder. The grade of the fly ash is Grade II, and it is a gray-black powder. The grade of the superfine slag powder is S95, and it is a gray-white powder, with a particle size of 1~35 μm and a specific surface area of 400~450 m^2/kg. The chemical composition and content of the test materials are shown in Table 1.

Table 1. Chemical composition of test materials.

Name of Raw Material	Main Chemical Composition/%							
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	Other
Dredged sludge	56.91	18.40	9.25	6.84	2.30	0.81	0.73	4.76
Construction waste powder	39.77	8.81	6.28	35.11	2.09	2.28	1.13	4.53
Desulfuration gypsum	32.01	12.25	0.68	31.46	8.15	0.55	0.58	14.32
Fly ash	47.28	26.85	4.89	4.52	0.33	1.42	0.78	13.93
Superfine slag powder	29.81	13.49	1.08	36.46	6.57	0.52	0.37	11.70

The sludge was put into a JJ-5 cement mortar mixer (Wuxi Jianding Jiangong Instrument Factory, Wuxi, China) and stirred at 140 r/min for 5 min; after that, the construction waste powder, desulfuration gypsum, fly ash and slag were added in different ratios in sequence and stirred at 60 r/min for 5 min, and then a sodium hydroxide solution was added and stirred at 60 r/min for 10 min to ensure that the solidified material and sludge were evenly mixed. The mixture was then taken out and pressed into a mold with a specified size to prepare a sample of modified sludge with calcium-containing waste. The material proportion of MS1~MS9 modified sludge samples is shown in Table 2. The ratio of the modified sludge was designed by orthogonal experiment. In the modified sludge, the mass percentage of internal lake sludge is 50%; the mass percentage of construction waste powder is 20~26%; the mass percentage of slag powder is 10~15%; the mass proportion of fly ash is 8–13%; the mass proportion of desulfurized gypsum is 3–8%; and the mass proportion of sodium hydroxide is 0.5%. The leaching amount of heavy metals in the modified sludge is lower than the standard limit of “Identification Standard for Hazardous Waste-Identification of Leaching Toxicity” (GB5085.3-2007), indicating that the modified sludge has good environmental effects.

2.2. Test Methods

For MS1~MS9, modified sludge samples with calcium-containing waste naturally cured indoors for 1~28 days, the unconfined compressive strength of the modified sludge samples was tested using the pressure tester (YAW-A, Changchun New Testing Machine Co., Ltd., Changchun, China), and the sample was a cylinder with a diameter of 5 cm and a height of 10 cm. The shear strength of the modified sludge samples was tested using the automatic direct shear apparatus (Digi-Shear, GCTS, Tempe, AZ, USA), and the sample was a cylinder with a diameter of 6.18 cm and a height of 2.0 cm. Meanwhile, the volume shrinkage of the modified sludge sample was observed.

Table 2. Material mix proportion of modified dredged samples.

No.	Inner Lake Sludge	Construction Waste Powder	Superfine Slag Powder	Fly Ash	Desulfuration Gypsum
MS1	50%	20%	13%	10%	7%
MS2	50%	22%	13%	10%	5%
MS3	50%	26%	12%	9%	3%
MS4	50%	22%	15%	10%	3%
MS5	50%	22%	13%	8%	7%
MS6	50%	22%	10%	13%	5%
MS7	50%	22%	15%	8%	5%
MS8	50%	22%	13%	12%	3%
MS9	50%	22%	10%	10%	8%

The MS1, MS5, MS7 and MS9 samples, which were naturally cured indoors for 28 days, were dried in a vacuum drying oven at 50 °C for 72 h, and then saturated in a vacuum saturation cylinder with negative pressure of 0.8 MPa and temperature of 20~25 °C for 48 h. There were six dry–wet cycles in total. An environmental geotechnical flexible-wall permeameter (PN3230M, GEOEQUIP, Boston, MA, USA) was used to conduct the permeability test of modified sludge during the dry–wet cycle. The permeability test was carried out according to the ASTM standard (D5084-2010). The sample was a cylinder with a diameter of 5 cm and a height of 10 cm. The confining pressure for test was 350 kPa, and the back osmotic back pressure was 30 kPa.

According to the MS7 samples after the permeability test, the samples at 0~2 cm, 4~6 cm and 8~10 cm were selected, and the microstructure of the samples was comprehensively tested by various micro-test methods to explain the action mechanism of the modified inner lake sludge with calcium-containing waste. X-ray diffractometer (XRD, D8 ADVANCE, Bruker, Karlsruhe, Baden-Württemberg, Germany) and Fourier transform infrared spectrometer (FT-IR, Nicolet 6700, Thermofisher Scientific, Waltham, MA, USA) were used to detect the hydration products and molecular groups of the samples. Moreover, SEM and EDS (SEM, Gemini SEM 300, Carl Zeiss AG, Jena, Germany) were adopted to detect the surface micro-morphology and elemental composition of the samples.

3. Results and Discussion

3.1. Mechanical Strength

The volume shrinkage of the modified dredged sludge samples with calcium-containing waste is shown in Figure 1, from which it can be seen that the volume shrinkage of the modified sludge samples under natural curing conditions increased with the prolongation of time, and it mainly occurred in the first 7 days. After 28 days, the volume shrinkage of modified sludge samples of MS1~MS9 was 2.6~8.3%; the MS7 sample showed remarkable resistance to shrinkage deformation; and the MS1 sample had the weakest resistance to shrinkage deformation. After 7 days, the volume shrinkage of MS1~MS9 samples was 2~5.8%, accounting for 58.3~89.5% of the volume shrinkage deformation after 28 days. Under natural indoor curing conditions, none of the MS1~MS9 modified sludge samples showed cracks, and the surface had no peeling and falling off, thus presenting a good structure.

The unconfined compressive strength of the modified dredged sludge samples with calcium-containing waste is shown in Figure 2, from which it can be seen that the unconfined compressive strength of the modified sludge samples increased rapidly from 0.4~1.2 MPa to 4.2~9.8 MPa within 3~7 days, and then slowed down obviously, reaching 7.9~14.5 MPa after 28 days. The MS7 sample showed remarkable compressive bearing capacity. In conjunction with the results of the volume shrinkage test, the natural curing time of the covering material of the modified sludge landfill was 7 days, which also showed that the alkali-activated reaction between calcium-containing wastes, such as slag and construction waste powder, and the sludge of the inner lake mainly took place in the first 7 days; a large number of gelled geopolymers were produced in the modified sludge samples at this stage.

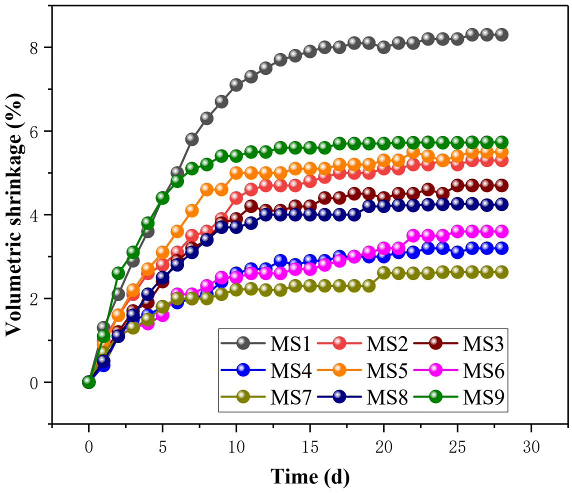


Figure 1. Volume shrinkage of modified dredged sludge samples.

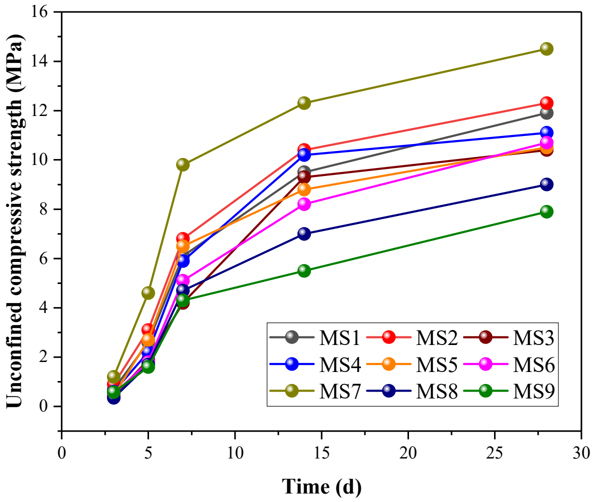


Figure 2. Unconfined compressive strength of modified dredged sludge samples.

The shear curve of the modified sludge samples with calcium-containing waste is shown in Figure 3, from which it can be found that the shear stress showed a trend of increase and then decrease with the increase in shear displacement, while the shear strength increased and the shear damage displacement decreased with the increase in the curing time. For MS9 sample, the shear strength was 141.47~254.69 kPa, and the corresponding shear displacement was 5~7.72 mm after curing for one day; after curing for 7 days, the shear strength increased to 213.59~785.07 kPa, and the corresponding shear displacement decreased to 1.18~3.17 mm.

The shear strength parameters (cohesive force c, internal friction angle φ) of MS7 and MS9 modified sludge samples are shown in Figure 4. The shear strength parameters of MS7 and MS9 samples showed basically the same rule. During the first 7 days of curing, the values of c and φ increased rapidly, and on the 7th day, the values of c and φ reached the maximum, and remained basically stable for the next 7~28 days. The c and φ values of MS7 sample were significantly higher than those of MS9 sample, and the c and φ values of

MS7 sample increased from 140 kPa and 26.1° to 329 kPa and 53.6° respectively. The c and φ values of MS9 sample increased from 120 kPa and 17.8° to 179 kPa and 42.59° respectively. The above data also indicated that the calcium-containing solid waste underwent intense alkali-activated reaction in the first seven days, and the strength at this stage increased rapidly, so it can be judged that the curing time of seven days was more reasonable for the landfill covering material of modified sludge with calcium-containing solid waste.

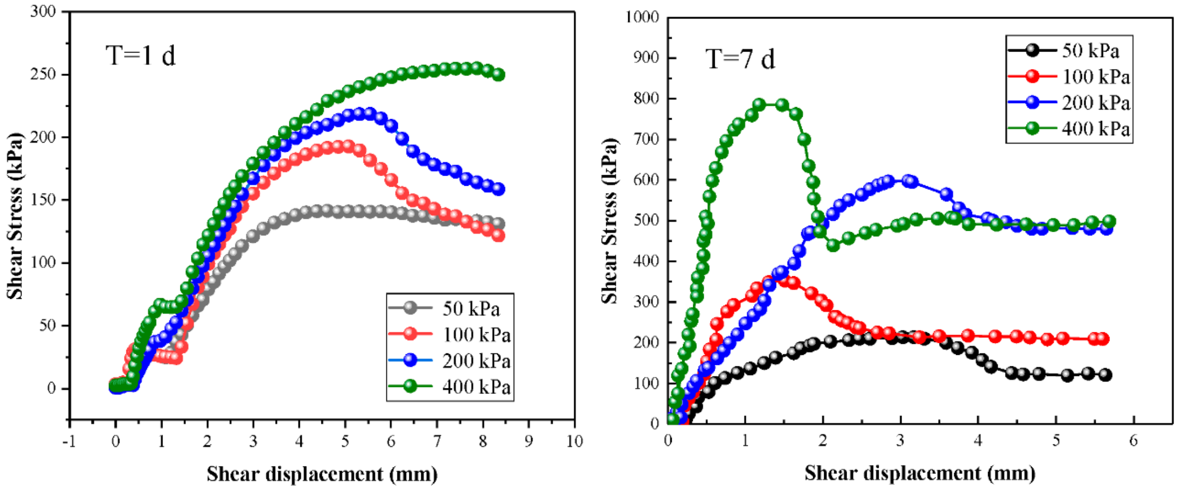
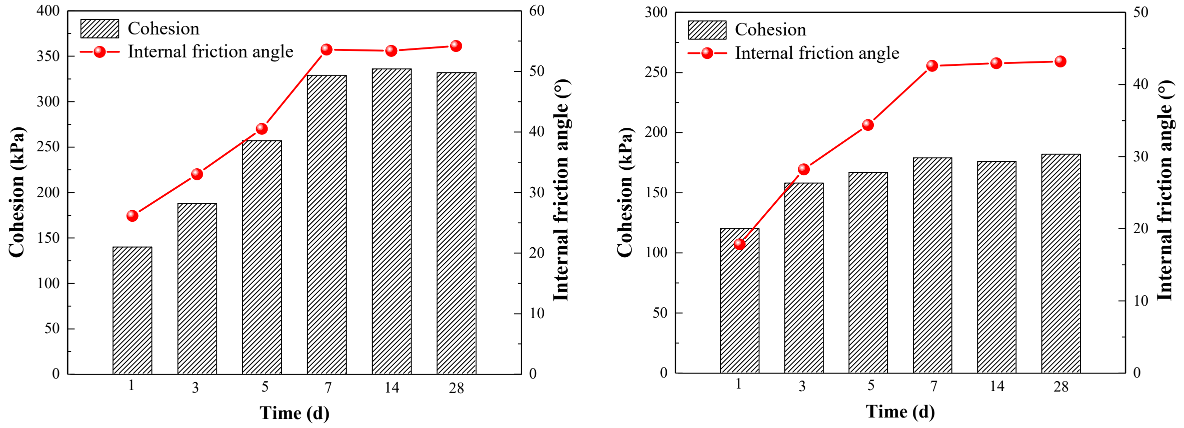


Figure 3. Relationship curve between shear stress and displacement of MS9 modified dredged sludge sample.



(**a**) (**b**)

Figure 4. The cohesion and internal friction angle of modified dredged sludge samples. (**a**) MS7 sample, (**b**) MS9 sample.

3.2. Hydraulic Conductivity Coefficient

The change in permeability coefficient of MS1, MS5, MS7 and MS9 samples after curing after 28-day curing with permeation time is shown in Figure 5. It can be found from Figure 5 that the permeability coefficients of the samples firstly increased, then decreased and finally tended to be stable. During 0~100 h, the permeability coefficient of the sample was in a stage of rapid increase; at 100~250 h, the permeability coefficient was slowly decreasing; after 250 h, the permeability coefficient was basically stable, and the permeability coefficient of MS1~MS9 test was 0.39~5.75 $\times 10^{-7}$ cm/s. At the initial stage of permeability, some of the pores in the sample were not filled with water and were in an unsaturated state, which led to the larger seepage velocity of water under the action of capillary force, and thus the increase in the permeability coefficient [23,24]. With the extension of the permeation time, the sample reached the saturation state, and the unreacted calcium-containing solid waste fully contacted with water to form a gelled geopolymer under the action of hydration reaction, thus reducing the porosity and permeability coefficient of the sample.

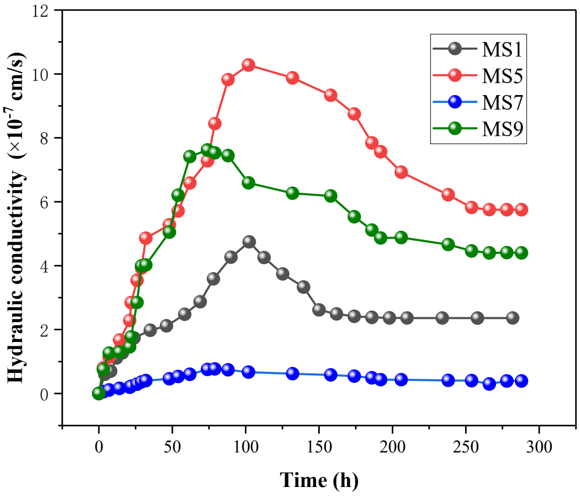


Figure 5. Hydraulic conductivity of modified sludge dredged samples.

The dry–wet cycle has a strong destructive effect on the anti-seepage performance of landfill covering materials. After 3~5 dry–wet cycles, the traditional compacted clay covering material showed obvious cracks in the sample, and its permeability coefficient can be increased by 1~2 orders of magnitude [25]. To check whether the modified sludge with calcium-containing solid waste can maintain the anti-seepage performance under the action of the dry–wet cycle is a key index to evaluate its applicability as a landfill covering barrier material. The permeability coefficient of the modified sludge sample under dry-wet cycle conditions is shown in Figure 6. It can be found from Figure 6 that the permeability coefficients of MS1, MS5, MS7 and MS9 samples maintained the trend of slow increase during the first four dry–wet cycles, and none of them exceeded one order of magnitude. After the fourth dry–wet cycle, the permeability coefficient of the sample remained basically stable, the value of which was 0.84 to 11.1 × 10^{-7} cm/s, and no crack appeared in the sample. The MS7 sample showed the best anti-seepage performance, which can withstand the damage of dry–wet cycle, and the anti-seepage performance can meet the engineering requirements of less than 1 × 10^{-7} cm/s. The dry–wet cycle results in the change of the inter-particle pores to intra-granular pores, and the change of intra-granular pores to inter-particle pores, resulting in a larger aperture and larger permeability coefficient [26].

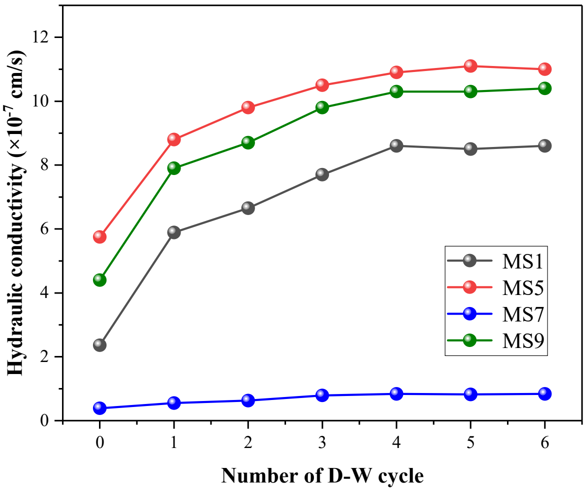


Figure 6. Hydraulic conductivity of modified dredged sludge samples during drying–wetting cycles.

3.3. Micromechanism Analysis

3.3.1. Hydration Products

The changes in crystalline phase materials during sample preparation can be obtained by XRD characterization. The XRD detection results at different depths (0~2 cm, 4~6 cm, and 8~10 cm) of MS7 modified sludge samples with calcium-containing waste are shown in Figure 7. It can be found from Figure 7 that the XRD crystallization peaks of samples at different depths had little difference, and a large number of porous zeolite and ettringite crystals in the skeleton structure were found in the samples. Quartz (SiO_2) and gypsum ($CaSO_4{\cdot}2H_2O$), accompanied by a small amount of abrazite ($CaAl_2Si_2O_4{\cdot}4H_2O$), calcium aluminate carbonate hydrate ($Ca_4Al_2O_6CO{\cdot}11H_2O$), calcium silicate hydrate ($Ca_4Al_2O_6CO_3{\cdot}11H_2O$), ettringite ($Ca_6Al_2(OH)_{12}(SO_4)_{32}{\cdot}6H_2O$) and typical 11Å structure of tobermorite constitute the main crystalline phases. Abrazite is a hydrated calcium aluminosilicate substance, and ettringite is a crystalline hydrated calcium sulphoaluminate substance produced by the combination of hydration products, C–A–H and SO_4^{2-} ions. C–S–H is formed together with ettringite, so the sample has a higher strength.

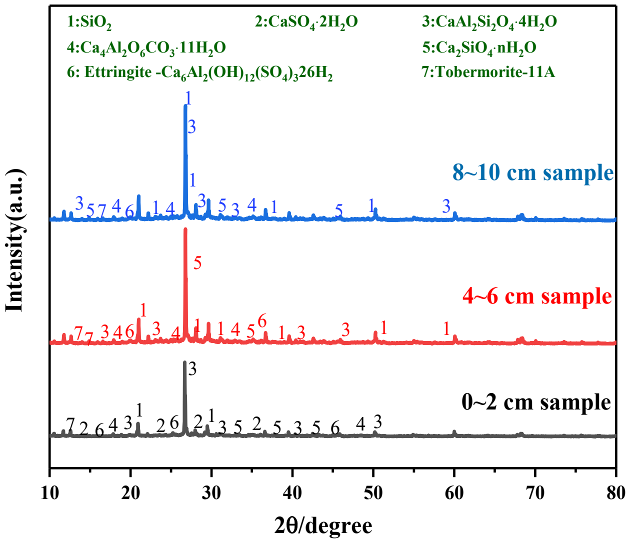


Figure 7. X-ray diffraction pattern of MS7 modified dredged sludge sample.

3.3.2. Molecular Groups

The FT-IR pattern of the MS7 modified sludge sample is shown in Figure 8. It can be observed from Figure 8 that the stretching vibration peak of -OH and the bending vibration peaks of H–O–H were located at the 3412 cm^{-1} and 1623 cm^{-1} bands, respectively, indicating that the sample contains free water and bound water. The peak changes at 1450 and 876 cm^{-1} resulted from the carbonation of calcium and aluminum in the hydration process when they contacted with CO_2 in the air, which led to the asymmetric stretching vibration of the C–O bond, to generate the calcium aluminate carbonate hydrate and zeolite. Si–O and Al–O with a peak value of 1020 cm^{-1} prove that a typical characteristic structure of C–A–S–H gel exists in the sample. The peak value of 450 cm^{-1} can be attributed to the bending vibration of the SiO_4 tetrahedron. According to the theory of aluminosilicate, the formation of these strips and tetrahedral silicon oxide substance is related to the reticular aluminosilicate gel.

3.3.3. Micro-Morphology and Element Composition

The surface micro-morphology of the modified sludge sample is shown in Figure 9. Figure 9a–c shows that there are cluster-like structural substances on the surface of the sample, and the cluster-like substances are closely connected with soil particles. The cluster-like substances in Figure 9c are significantly more than those in Figure 9a. In addition, many pores appeared on the surface of the sample in Figure 9a, but the pores obviously decreased in Figure 9c, and the original pores were filled by cluster-like substances. This may be due to the fact that the calcium-containing solid waste generates hydration products, C–S–H and C–A–S–H hydration gels, under the action of alkali activation, to fill the pores between soil particles, thus forming a structure with firm skeleton characteristics between soil particles and gels, which is an important reason for the high mechanical strength of modified sludge.

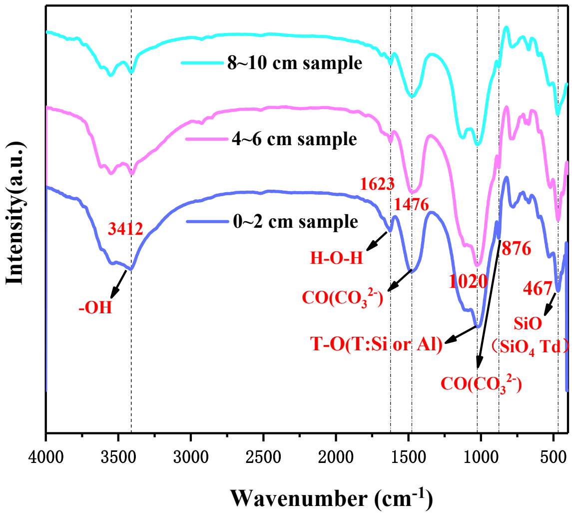
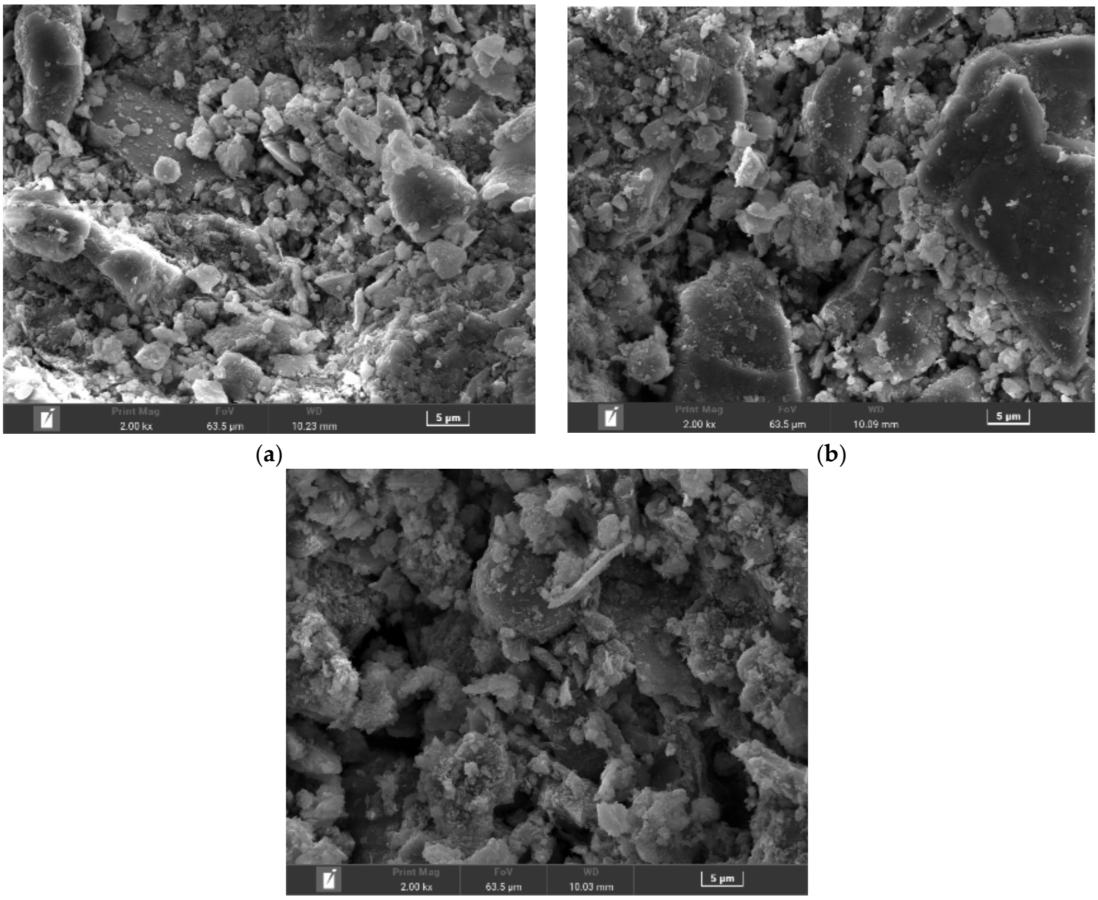


Figure 8. FT-IR spectrum of MS7 modified dredged sludge sample.



(**a**) (**b**)

(**c**)

Figure 9. SEM Images of MS7 modified dredged sludge sample. (**a**) 0~2 cm sample, (**b**) 4~6 cm sample, (**c**) 8~10 cm sample.

The EDX energy spectrum of the MS7 modified sludge samples is shown in Figure 10. It can be found from Figure 10 that the substance elements in the sample are mainly O, Si, Al and Ca, and a small amount of Fe, Na, Mg, S are also included. The formation of C–S–H and C–A–S–H hydrated gels with a Tobermorite structure can be judged according to the microscopic detection results by means of XRD, FT-IR and SEM. In the formation chain, some Si and Al replaced each other, and alkali metal sodium ions replaced some calcium ions. Meanwhile, Na with a content close to 1% was detected in the sample, which may form the N–A–S–H hydrated gel product. The pores inside the structure were filled so as to make the structure more compact, resulting in higher strength and lower permeability of the sample.

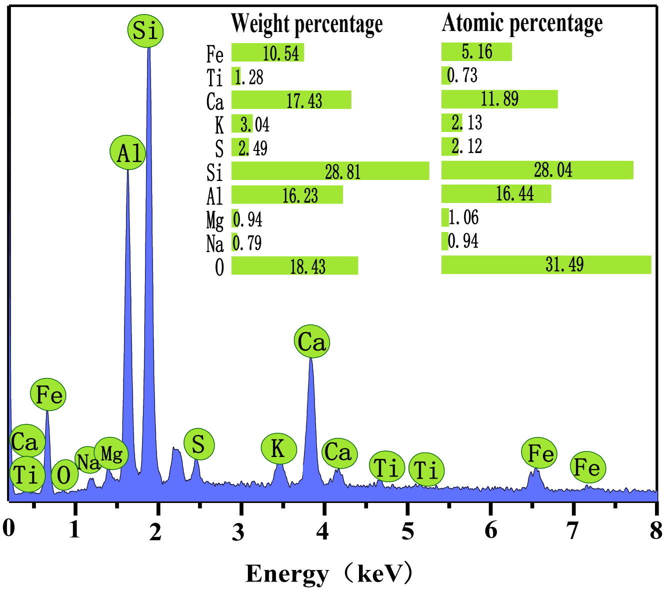


Figure 10. EDX general spectrum of MS7 modified dredged sludge sample.

4. Conclusions

In this present study, dredged sludge from urban inner lakes was taken as the base material, and the sludge was modified with calcium-containing wastes (construction waste, slag, desulfuration gypsum and fly ash) in order to obtain a landfill closure and coverage material with long-term service performance. The service performance of the modified sludge covering layer was systematically analyzed by testing the volume shrinkage, unconfined compressive strength, shear strength and permeability coefficient of the modified sludge samples. The modification mechanism of calcium-containing waste on sludge was revealed through the analysis of hydration products, molecular groups, surface micromorphology and elements of modified sludge samples.

The covering material for the modified sludge with calcium-containing waste had a high mechanical strength. The volume shrinkage of the modified sludge sample was 2.6~8.3%; the unconfined compressive strength was 7.9~14.5 MPa; the cohesive force c was 179~329 kPa, the internal friction angle φ was 42.59~53.60°, and the permeability coefficient was 0.84~11.1 $\times 10^{-7}$ cm/s. The MS7 sample showed remarkable mechanical strength and anti-seepage performance. After six dry–wet cycles, the permeability coefficient of MS7 sample wa less than 1×10^{-7} cm/s, which meets the anti-seepage requirements of landfill overburden engineering. Compared with the compacted clay covering material, the covering material for the modified sludge with calcium-containing solid waste showed good damage resistance to dry–wet cycles. The hydration reaction of calcium-containing solid waste under the action of alkali activation mainly occurred in the first seven days, and the C–S–H and C/N–A–S–H gelled geopolymers with a Tobermorite structure were formed, which wrapped sludge particles, filled pores and formed a firm skeleton. To sum up, the modified inner lake sludge with calcium-containing waste (construction waste, slag, desulfuration gypsum, and fly ash) can be used as landfill closure and coverage material.

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Data Availability Statement: The datasets used in the current study are available from the corresponding author on reasonable request.

Conflicts of Interest: The author declare no conflict of interest.

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