


Article

Study on Microstructure of Cemented Organic Soil in Dianchi Lake, China

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Abstract: The organic soil in the Dianchi Lake area contains much humic acid (HA), and the impact of HA on cemented soil has attracted much attention. In this study, test soil is prepared based on the actual HA content of the soil in Dianchi Lake, and cement is added to prepare samples. Unconfined compressive strength (UCS), scanning electron microscopy (SEM), energy-dispersive analysis (EDS), and X-ray diffraction (XRD) tests are conducted on the samples. The strength development and microstructure changes of cemented organic soil are studied. The results show that (1) cemented soil strength decreases with the increase in HA content and increases with the cement mixing ratio; (2) SEM and EDS tests show that with the increase in HA content, the connection between soil particles gradually changed from surface–surface to point–surface, point–edge, or point–point connection. A large overhead structure is inside the cemented soil, and its integrity weakens with increased HA content. However, elements such as Ca, Si, and Al exist in the pores of cemented soil, indicating that cement hydration products are cemented between soil particles. XRD testing shows that with the increase in HA content, the intensity of the diffraction peaks of hydration products in cemented soil gradually decrease. (3) In general, HA will gradually deteriorate the integrity of cemented soil and reduce its strength. When the HA content is more than 15%, the effect of HA on strength is more prominent. However, increasing the cement mixing ratio can effectively weaken HA's negative effect on cemented soil's strength. When the cement mixing ratio is between 15% and 25%, the strength of cemented soil with high HA content can be effectively increased. However, this approach is not conducive to the protection of the environment.



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Keywords: Dianchi Lake; organic soil; humic acid; strength development; microstructure

1. Introduction

There is a large amount of peat soil around Dianchi Lake and Erhai Lake in Yunnan Province. The peat soil is a poor foundation soil. The high organic matter content leads to its characteristics of large porosity, high compressibility, and low bearing capacity [1–3]. For this reason, engineers often use high-quality and cheap cement to reinforce the poor foundation soil [4–8].

Since the discovery of the humic group (HG), many experts and scholars have conducted much research on HG's separation, extraction, and determination of components and microstructure. In 1826, Sprengel [9] first systematically studied HG's source and chemical nature and explored its many preparation methods. In 1985, Scorenikova et al. [10] conducted many indoor and outdoor studies on the chemical composition and physical–chemical characteristics of peat soil in Moscow for the first time, which is of great significance to the in-depth study of peat soil in Moscow. In 2003, Schmeide et al. [11] considered that the main components of HG were fulvic acid and humic acid (HA). Among them, fulvic acid is soluble in water, alkaline solution, and acidic solution. It has significant mobility in soil and exists in liquid form. HA is soluble in alkaline solution, but not in water

or acidic solution, and occurs in soil as solid particles, which occur in the form of solid particles in the soil body and form the soil framework together with the soil particles [12].

With the prominence of fundamental engineering problems in peat soil environments, experts and scholars have gradually combined research on HG with engineering practices. In 1989, Kamon et al. [13] used cement to solidify fluid ooze and found that the strength of the solidified product decreased with the increase in HG. In contrast, the presence of ettringite would increase the strength of the solidified fluid ooze. In 2000, Xun Yong [14] found that the organic matter reacted with cement hydration products, weakening cement's reinforcement effect on organic soil. In 2002, Tremblay et al. [15] found that organic matter would affect the hydration of cement by changing the pH value of the pore solution, thereby affecting the development of cemented soil strength. In the same year, Zeng [16] and other studies found that the organic matter in the soil is an acid body, which hinders the hydration reaction of cement. In 2003, Valls et al. [17] found that when the soil organic matter content is high, the soil and surrounding solutions are acidic, with high water content and low permeability. In 2005, Bertron [18] found that HG had a corrosive effect on cemented soil. In 2009, Zhu et al. [19] used dredged sludge to explore the effect of HG on hydration products. The results show that HG reacts with the calcium hydroxide produced by cement hydration and generates soluble compounds, which affects the development of the strength of the cured product. In 2009, Zhang [20] studied the effect of HG on cemented soil strength through on-site sampling. The results showed that HG reacted with cement hydration products to form more complex compounds. The total amount of cement hydration products decreased, which changed the microstructure of the cemented soil, resulting in a decrease in the strength of the cemented soil. In 2017, Kang [21] studied the effect of HG on the strength properties of cemented soil. The results showed that increased HG content gradually decreased the total amount of cement hydration products. In addition, the HG and cement content significantly impact the change in cemented soil strength. In 2019, Qi et al. [22] studied the effect of HG on the mechanical properties of cemented soil. The results showed that with the increase in the HG content, the strength of the cemented soil gradually decreased to a specific critical value and then stabilized.

In summary, many scholars regard HG as a whole to study its mechanism of action on cemented soil. However, the impact of a specific component of HG (humic acid, fulvic acid) on the strength of cemented soil is still unclear. In addition, the existing research does not link the strength development of HA on cemented soil with the change in its microstructure. Therefore, given the difference in solubility of HA and its existing form, cemented soil samples are prepared by mixing HA and cement into cohesive soil with low organic content. The UCS, SEM, EDS, and XRD tests study the strength development and microstructure changes of cemented soil containing HA. The strength development and microstructure changes of cemented soil containing HA are studied through the UCS, SEM, EDS, and XRD tests. This research has specific theoretical and practical value for further exploring the cemented organic soil.

2. Materials and Methods

2.1. Materials

1. For the test, the cohesive soil near Sanhe Village, Xinjie Town, Jinning County, Kunming City, was selected as the original test soil (soil not mixed with humic acid reagent). The color of this soil is brownish-yellow or brownish-red (Figure 1). After obtaining the original test soil, several indoor tests were carried out on it according to the "Standard for geotechnical testing method" (GB/T50123-2019) [23]. The basic physical and mechanical properties of the original test soil used in the test are shown in Table 1. The natural moisture content was measured by the drying method. The liquid-plastic limit was measured by the combined liquid-plastic limit method. The specific gravity of soil particles was measured by the pycnometer method. The standard consolidation test measured the modulus of compression. Moreover, it was determined that the HA content of the original test soil is 0.32%, which could reduce

the influence of the organic matter in the original test soil on the test results. The determination method of HA is as follows: Firstly, the carbon content of the original test soil is determined, and then its content is calculated in combination with its corresponding carbon content. Studies [24–26] show that the carbon content of HA is about 50–60%. Comprehensively comparing the molecular structure and molecular formula of HA proposed by previous studies [25], the carbon content of HA is about 60%. Then, combining the measured carbon and element content, the HA content of the original test soil is calculated. Its XRD pattern is shown in Figure 2. The main crystal phases of the original test soil are quartz, kaolinite, orthoclase, and serpentine.



Figure 1. Test soil.

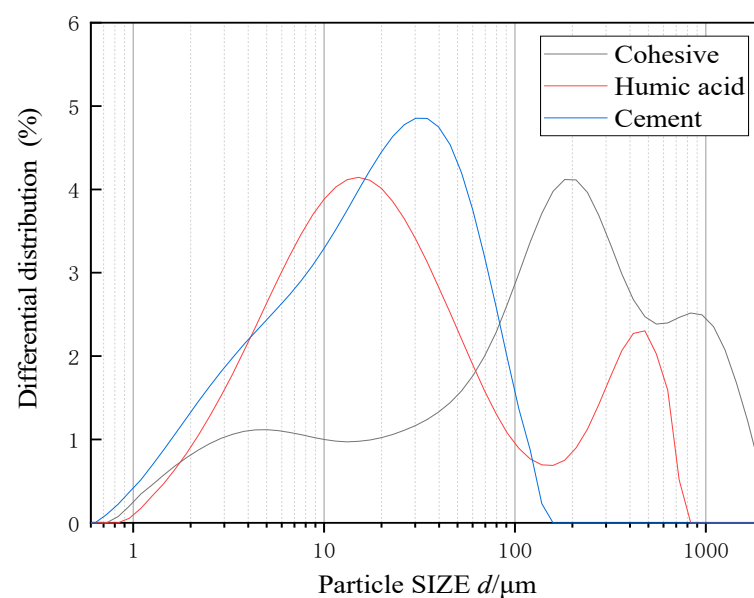
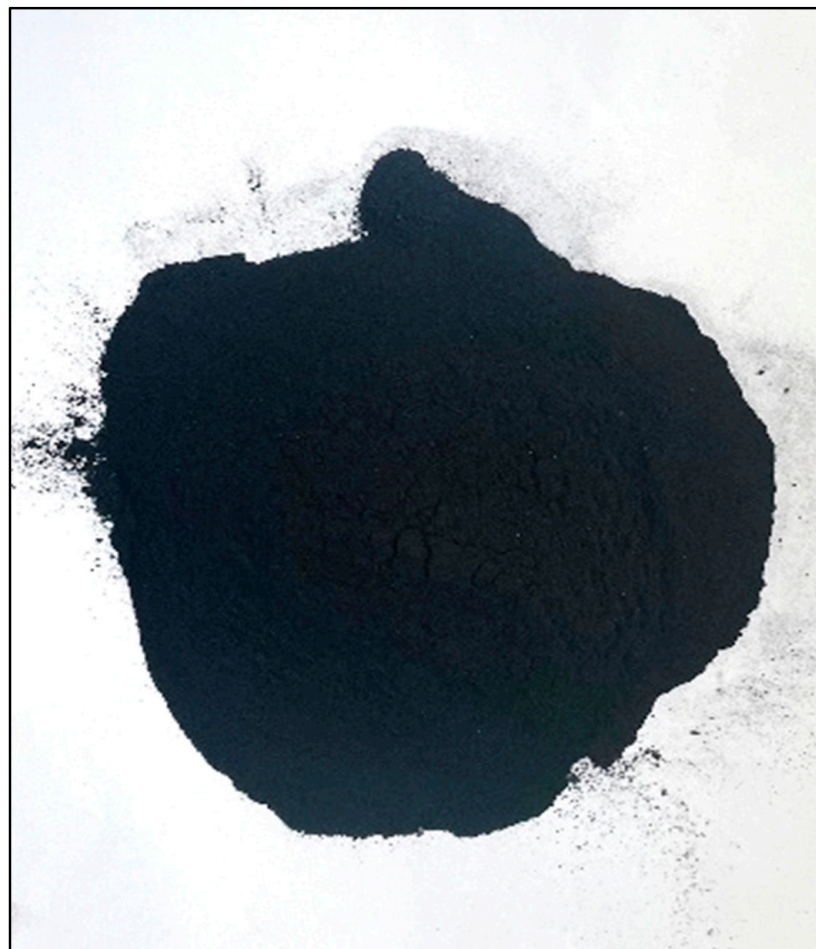


Figure 2. Particle distribution curve of the test materials.

Table 1. Physical and mechanical properties index of undisturbed soil for testing.

Original Test Soil	Natural Moisture Content ω (%)	Liquid Limit w_L (%)	Plastic Limit w_P (%)	Natural Density ($\text{g}\cdot\text{cm}^{-3}$)	The Specific Gravity of Soil Particle G_s	Modulus of Compression (MPa^{-1})	Natural HA Content (%)
Cohesive soil	33.5	39.26	20.38	1.87	2.73	4.65	0.32%

- The humic acid reagent used in the test is produced by a standard chemical reagent factory (Figure 3). Its molecular structure [27] is shown in Figure 4.

**Figure 3.** HA reagent.

- The cement used in the test is ordinary Portland cement.
- The test water is distilled water.

Figure 5 is the particle distribution curve of the test materials. The cohesive soil used in the test is inhomogeneous. (inhomogeneous coefficient $C_u = 41.70 > 5$) The soil has good gradation (curvature coefficient $C_c = 2.63$) and good compactness. The particle diameter of humic acid particles is generally greater than $10\mu\text{m}$. The particle size of ordinary Portland cement is less than $100\mu\text{m}$, which meets the requirements for the fineness of ordinary Portland cement in “Common Portland Cement” (GB175-2007) [23].

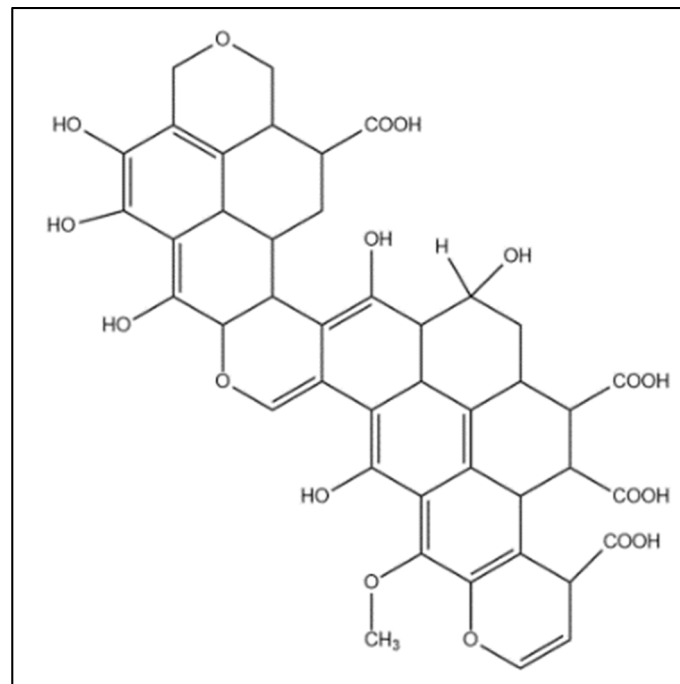


Figure 4. HA molecular structure.

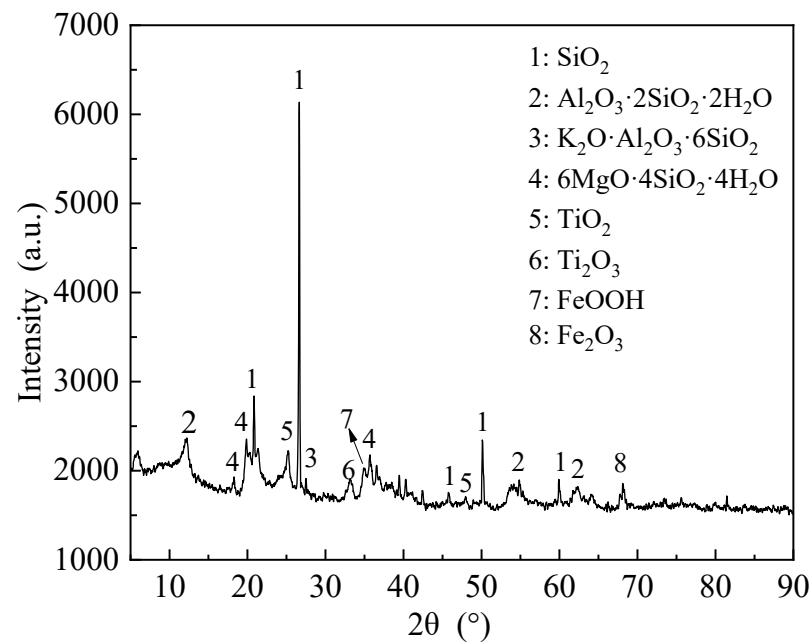


Figure 5. X-ray diffraction pattern of original test soil.

2.2. Methods

The overall plan of this study is as follows. (1) Preparing cemented organic soil samples indoors. (2) Curing the samples. (3) Conducting UCS, SEM, EDS, and XRD tests on the samples after curing. (4) Analyzing test results and summarizing research conclusions.

The test set the water content by mass ($\omega = 24\%$), void ratio ($e = 0.8$), and water-cement ratio ($c = 0.5$) of the sample. According to the previous research results, the total content of HG in the peat soil of Dianchi Lake is between 7.15% and 50.06%. Among them, the HA content is between 2.36% and 28.13% [28]. The results provide the basis for incorporating HA reagents in the samples.. The HA content of the sample (λ) and the cement ratio (β) (check Formula (1)) are shown in Table 2. Considering the stability and reliability of

the testing effect, each test group planned to make four parallel samples, and a total of 560 samples were made for the research.

Table 2. Design of sample preparation.

No.	Tests	Cement Ratio β (%)	HA Reagent Content λ (%)	Immersion Age (d)
1	UCS	5, 10, 15, 17.5, 20, 25, 30	0, 10, 12.5, 15, 20	28
2	SEM, EDS, XRD	17.5	0, 15, 20	

The test was carried out according to the “Standards for Geotechnical Test Methods” (GB/T50123-2019) [23]. The sample preparation process can be described as follows. The original test soil is dried in the oven at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$, then crushed into powder and passed through 2 mm sieves. The mass of soil particles, HA reagent, cement, and distilled water is calculated according to Formulas (1)~(3). In order to ensure the uniformity of the sample, the above materials are thoroughly mixed and divided into three parts and placed in a mold. The three-part mold with an inner diameter of $d = 39.1\text{ mm}$ and a height of $h = 80.0\text{ mm}$ was selected for this test. After the sample is made, it is sealed with a film for conservation. The temperature of the curing box is controlled at $20 \pm 3\text{ }^{\circ}\text{C}$ for seven days, and it is soaked in distilled water after curing. In this test, six parallel samples were made for each ratio to ensure the accuracy of the test. The sample preparation process is shown in Figure 6.

$$\beta = \frac{m_{s(\text{ce})}}{m_{s(\text{HA})} + m_{s(\text{soil})}} \times 100\% \quad (1)$$

$$\lambda = \frac{m_{s(\text{HA})}}{m_{s(\text{HA})} + m_{s(\text{soil})}} \times 100\% \quad (2)$$

$$m_{(w)} = (m_{s(\text{HA})} + m_{s(\text{soil})}) \times \omega + m_{s(\text{ce})} \times c \quad (3)$$

where $m_{s(\text{ce})}$ is the mass of cement particles, $m_{s(\text{HA})}$ is the mass of HA particles, $m_{s(\text{soil})}$ is the mass of the original test soil, and $m_{(w)}$ is the mass of distilled water.

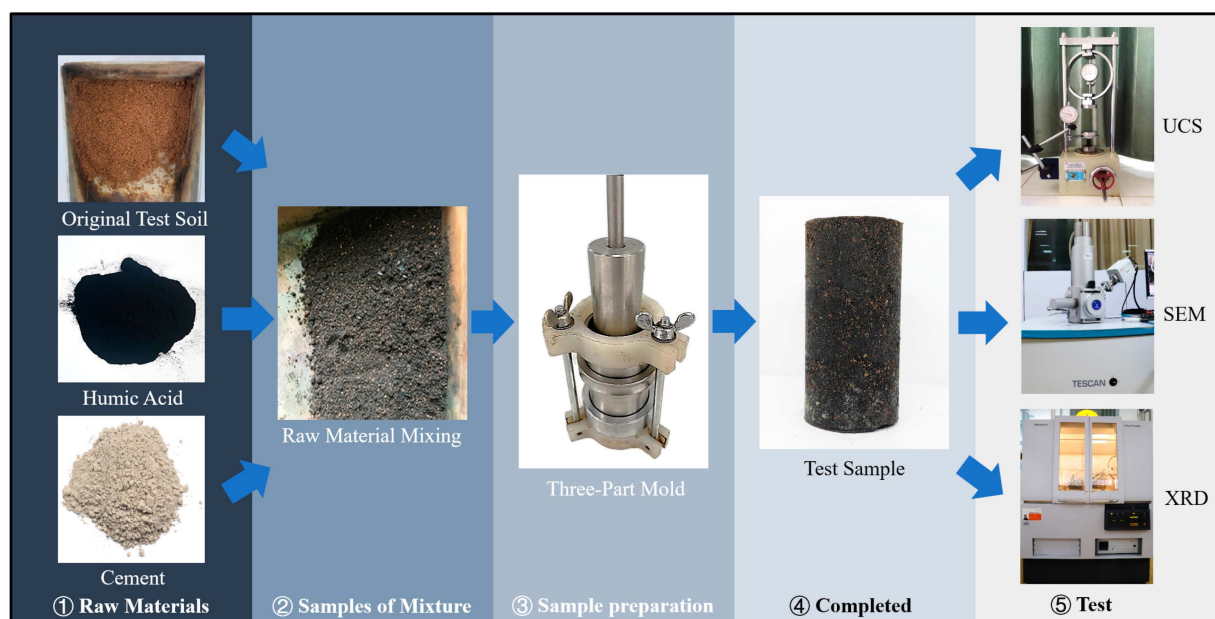


Figure 6. Sample preparation process.

After the sample preparation is completed, it is subjected to UCS, SEM, EDS, and XRD tests. The UCS test apparatus is the YSH-2 lime-soil electromotive UCS instrument. The scanning electron microscope test uses a Czech TESCAN-VEGA3 automatic tungsten filament scanning electron microscope. The X-ray diffraction test uses Holland PANalytical X'Pert3 (Figure 6). Samples were taken from the interior of cemented soil to measure their microscopic morphology and conduct elemental analysis. After the cemented soil samples are ground, they are sieved with a geotextile. The measured samples' particle size is less than or equal to 45 μm [29].

3. Test Results and Analysis

Figure 7 shows an SEM image of the cohesive soil aggregates at a magnification of 5000 times. It can be seen that the cohesive soil aggregates formed by the agglomeration of clay particles have strong integrity. There are few pores on the surface of clay aggregates, and the connectivity between the pores is poor. The connection between clay particles is mostly a surface–surface connection and a point–surface connection. The above reason is that the clay mineral particles are mainly composed of unit cells composed of silicon-oxygen wafers and aluminum-hydroxide wafers [30]. Figure 8 is the SEM image of the humic acid (HA) aggregates magnified 5000 times. It can be seen that the HA pellets formed by stacking the HA pellets are still loose and porous structures. The HA agglomerates have many pores, which is of poor integrity. HA particles are mainly connected between point and line and between point and point. The above reason is that HA is an organic polymer mixture with a very complex molecular structure containing aromatic rings. The connection between the HA molecules results in a spongy HA particle with ample internal space [31,32].

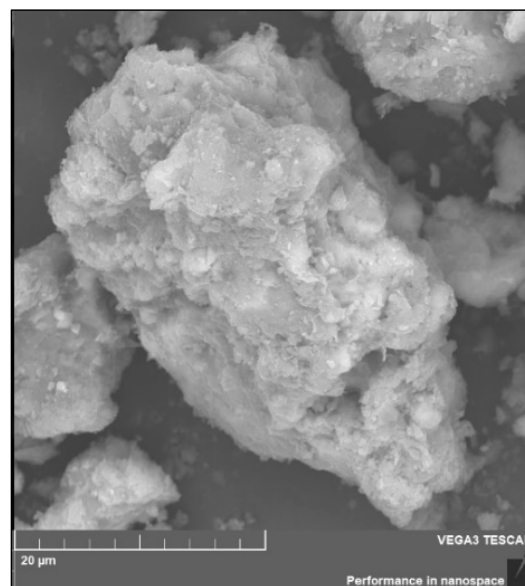


Figure 7. Cohesive soil aggregates.

3.1. UCS Test Results and Analysis of Cemented Soil

The UCS test results will not easily reflect the dominant influence of the two variable factors on the strength of the decoupled sample when the HA content and cement rate are low. Therefore, a confidence interval is set for the test results. Among them, the confidence interval of the HA content is 10~30%, and the confidence interval of the cement ratio is 10~25%.

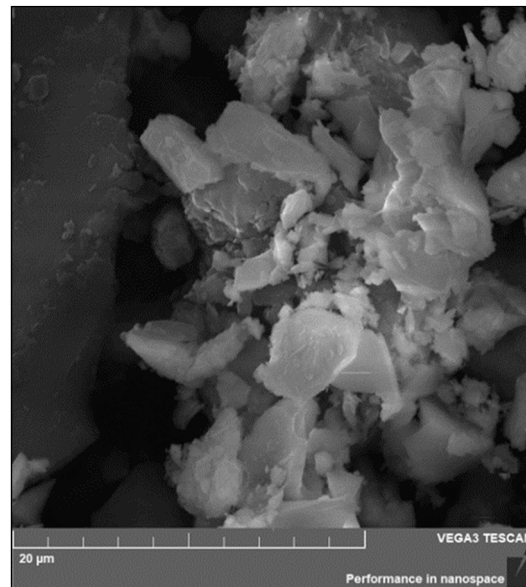


Figure 8. Humic acid aggregates.

Figure 9 shows the relation between UCS and the cement ratio. When the humic acid content is fixed, the UCS of the cemented soil sample increases with the cement ratio. It is worth noting that when the humic acid content is 20% and the cement ratio is lower than 15%, the UCS of the cemented soil increases slowly. It shows that when the humic acid content is high, the solidification effect of cement on organic soil is limited. However, the increase in cement ratio makes the cement hydration products gradually increase, and the influence of humic acid on the cement hydration reaction is gradually weakened. Its UCS increases with the increase in cement ratio. Therefore, the increase in cement ratio can weaken the negative effect of humic acid on the UCS of cemented soil.

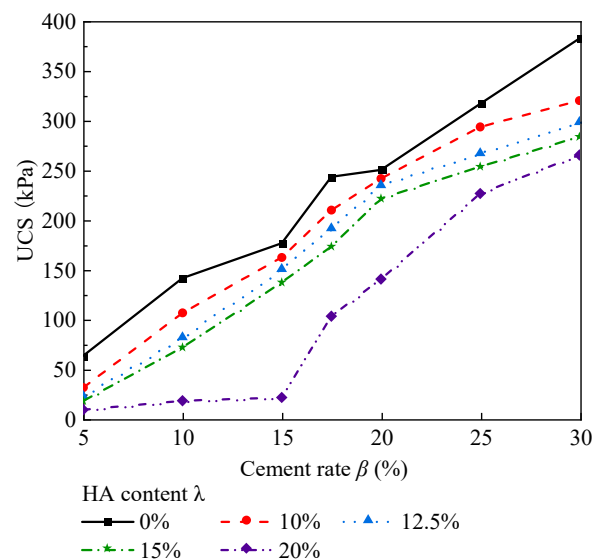


Figure 9. Relation curve between UCS and cement ratio.

Figure 10 shows the relation curve between UCS and humic acid (HA) content of the cemented soil sample. At the same cement ratio, the UCS of the cemented soil sample decreases with increased humic acid content. Especially when the cement ratio is less than 20% and the humic acid content is more than 15%, the UCS of the cemented soil sample decreases significantly.

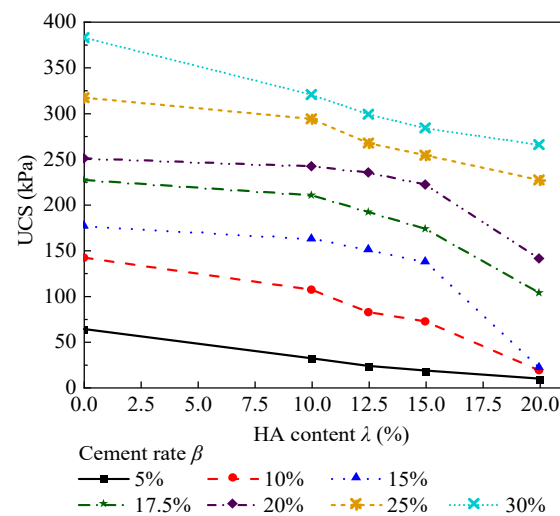


Figure 10. Relation curve between UCS and HA content.

3.2. SEM and EDS Test Results and Analysis of Cemented Soil

Figure 11 shows the cemented soil sample's SEM image ($500\times$, 8000 times). The cement mixing ratio is 17.5%, and the content of HA is 0%, 15%, and 20%. Figure 12 is the EDS image of the fixed-point scanning of the cemented soil sample, and the ratio of elements in the sample is shown in Table 3.

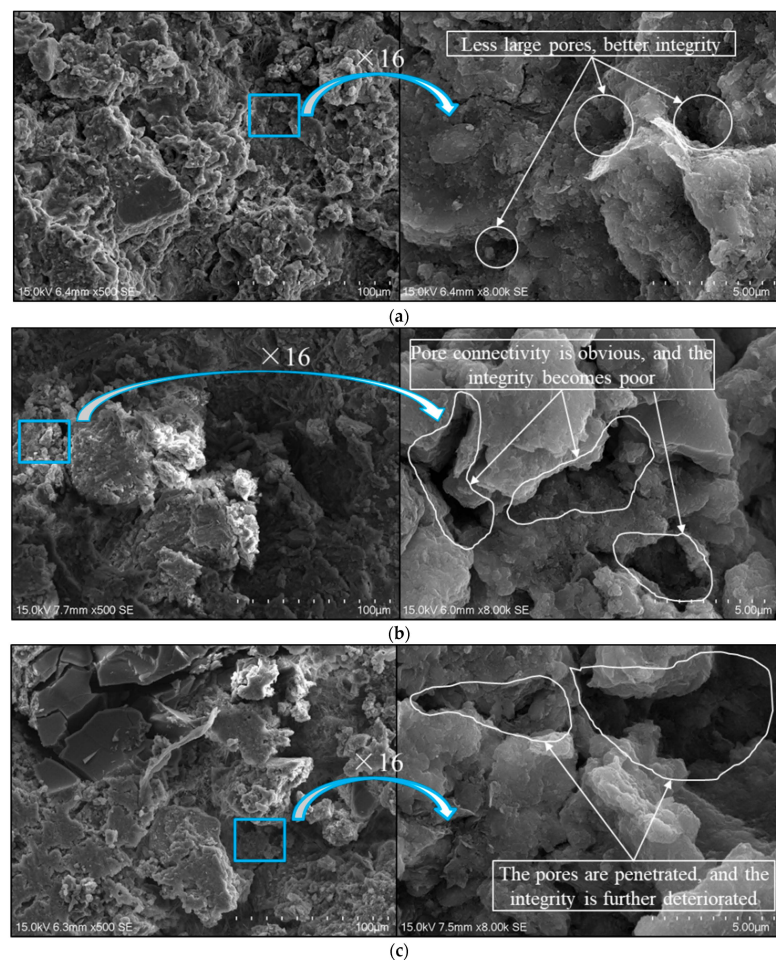


Figure 11. SEM image of cemented soil samples. (a) 0% HA content. (b) 15% HA content. (c) 20% HA content.

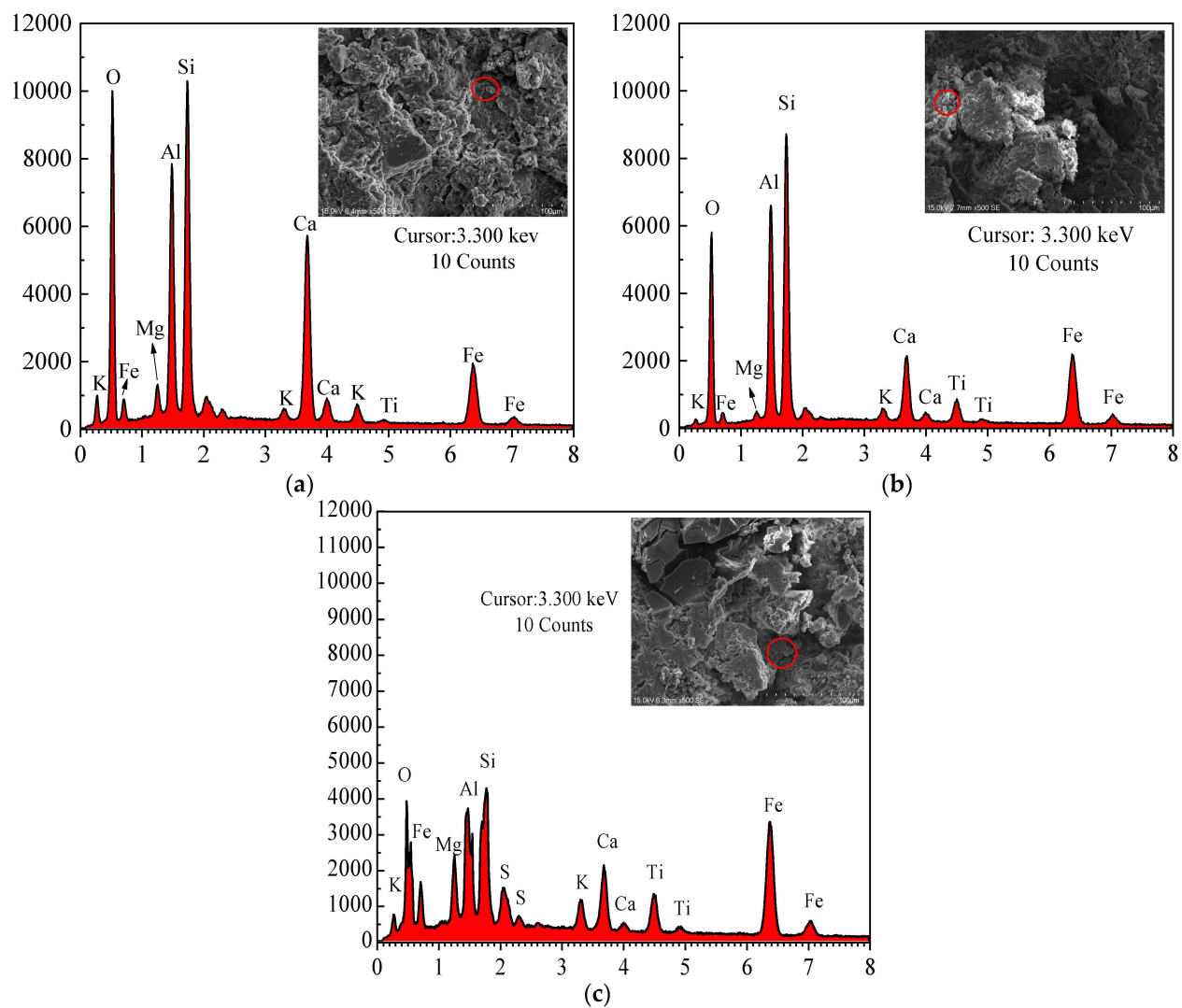


Figure 12. EDS diagram of cemented soil samples. (a) 0% HA content. (b) 15% HA content. (c) 20% HA content.

Table 3. The proportion of elements in samples.

Element	Weight/%			Atomic/%		
	0% HA	15% HA	20% HA	0% HA	15% HA	20% HA
O	44.20	33.36	44.43	63.29	52.68	62.89
Mg	1.18	0.19	2.47	1.11	0.20	2.30
Al	10.68	13.75	13.27	9.07	12.87	11.14
Si	14.80	19.30	16.00	12.07	17.36	12.90
S	0	0	0.65	0	0	0.46
K	0.43	1.03	1.25	0.25	0.67	0.72
Ca	14.43	7.56	3.29	8.25	4.77	1.86
Ti	1.55	3.20	2.52	0.74	1.69	1.19
Fe	12.74	21.60	16.13	5.22	9.77	6.54

Figure 11a shows that compared with the HA–cemented soil samples, the cemented soil without HA has fewer internal macropores. The particles have apparent agglomeration and better integrity. Figure 11b shows that when the content of HA is 15%, the internal pore connectivity of the cemented soil is apparent. The agglomeration effect of soil particles with the content of HA added by cement is not apparent, and the integrity of the cemented soil

becomes poor. Figure 11c shows that when the content of HA is 20%, the cemented soil's internal pores are connected, so there is a large overhead structure. The soil particles are loose, further deteriorating the integrity of the cemented soil. Overall, incorporating HA weakened the cementation effect of hydration products on soil particles. The connection mode between soil particles is gradually transformed from a surface–surface connection to a point–surface connection, point–edge connection, or point–point connection. The internal pore connectivity of cemented soil samples is apparent, and the integrity of cemented soil samples gradually deteriorates. Judging from the chemical properties of HA, the structural formula of HA contains many acidic groups, such as carboxyl and phenolic hydroxyl groups [33–35]. HA is slightly soluble in water and ionizes several hydrogen ions, making the pore water solution weakly acidic. The acidic environment leads to the consumption of alkali metal hydroxides and part of the gelling products in the cemented soil, resulting in poor integrity. Secondly, HA reacts with $\text{Ca}(\text{OH})_2$ and other hydration products in cemented soil, which destroys the alkaline environment required for cement hydration and delays the progress of the cement hydration reaction. The filling effect of hydration products on pores is not apparent [36].

Tiny particles of HA are soluble in an alkaline liquid and insoluble in an acidic liquid. According to the actual measurement, the pH value of the distilled water immersion solution of the cemented soil rises to 9.7–11.0. The alkaline environment can dissolve the HA particles, which directly leads to the structural deterioration of the cemented soil.

Figure 12 shows the main elements in cemented soil. Such as oxygen (O), sulfur (S), silicon (Si), calcium (Ca), aluminum (Al), potassium (K), magnesium (Mg), titanium (Ti), and iron (Fe). After comparison, it is found that the spectral peaks of O, Mg, Al, Si, Fe, and Ca elements are high, and the spectral peaks of K and Ti are also apparent. Ca, Si, and Al elements exist in cement soil. Cementitious products are calcium silicate hydrate, aluminate hydrate, and hydroxide. Compared with the cemented soil without HA, the spectrum peak and atomic mass fraction of Ca element in the cement decreased with the HA content. Although the peaks of Si and Al spectra decreased, the atomic mass fraction and the amount of HA did not show apparent regularity.

3.3. SEM and EDS Test Results and Analysis of Cemented Soil

Figure 13 shows the XRD diffraction patterns of the cemented soil samples with a curing age of 28d, a cement mixing ratio of 17.5%, and a humic acid (HA) content of 0%, 15%, and 20%.

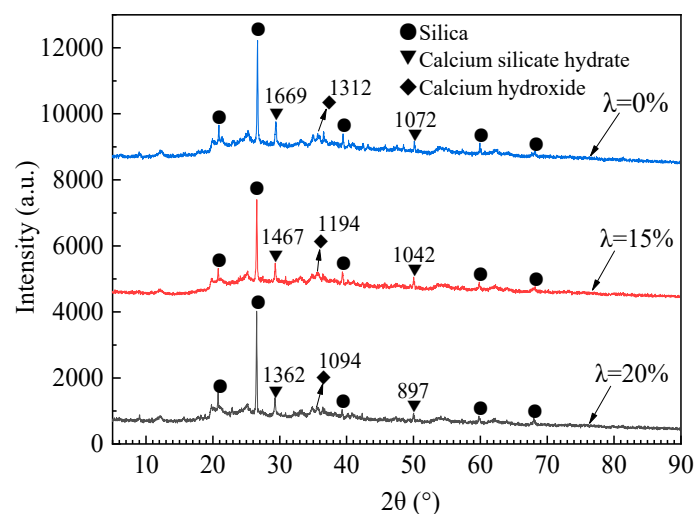


Figure 13. X-ray diffraction spectra of cemented soil samples.

As shown in Figure 13, the main crystalline phases in the cemented soil sample are SiO_2 , calcium silicate hydrate (CSH), and $\text{Ca}(\text{OH})_2$. SiO_2 is the main component of clay

minerals, while CSH and $\text{Ca}(\text{OH})_2$ are both cement hydration products. Under a certain cement mixing ratio, the diffraction peak intensities of CSH and $\text{Ca}(\text{OH})_2$ in cemented soil decreased gradually with increased HA content. This shows that the incorporation of HA hinders the progress of the cement hydration reaction and consumes many hydration products. Therefore, the intensity of the diffraction peaks of CSH ($2\theta = 29.35^\circ$ $2\theta = 50.0^\circ$) and $\text{Ca}(\text{OH})_2$ ($2\theta = 36.528^\circ$) produced by cement hydration decreased.

4. Conclusions

Based on the soil's actual humic acid (HA) content in Dianchi Lake, HA-containing cemented soil samples are prepared by adding HA and cement. After 28 days of curing, unconfined compressive strength (UCS), scanning electron microscopy (SEM), energy-dispersive analysis (EDS), and X-ray diffraction (XRD) tests are conducted on the samples. The strength development and microstructure changes of cemented soil containing HA are studied, and the following conclusions are drawn:

1. With the increase in HA content, the UCS of cemented soil samples decreases to varying degrees. When the content of HA exceeds 15%, HA dramatically influences the UCS of cemented soil. Under the same HA content, the cemented soil UCS increases with the cement ratio.
2. SEM and EDS tests show that the incorporation of HA gradually weakens the cementation effect of hydration products on soil particles. With the increase in HA content, the connection between soil particles gradually changes from a surface–surface connection to a point–surface connection, point–edge connection, or point–point connection. The internal pore connectivity of cemented soil is apparent, and there is a large overhead structure. Its integrity gradually deteriorates. The presence of elements such as Ca, Si, and Al in the cemented soil indicates the existence of hydration products. Ca, Si, and Al spectral peaks all decrease, but their atomic mass fractions and HA content do not show apparent regularity. XRD testing shows that the intensity of the diffraction peaks of CSH and $\text{Ca}(\text{OH})_2$ in cemented soil decreased gradually with increased HA content.
3. It can be seen from the above test that after adding HA, the internal pore connectivity of cemented soil is apparent, the integrity gradually deteriorates, and its strength gradually decreases. It is worth noting that when the HA content is more extensive than 15%, the effect of HA on cemented soil strength is more prominent. However, increasing the cement mixing ratio can weaken HA's negative effect on cemented soil's strength. When the cement mixing ratio is between 15% and 25%, the strength of cemented soil with high HA content can be effectively increased. However, this approach is not conducive to the protection of the environment. The strength of cemented soil is determined by HA content and cement mixing ratio.
4. The above research results show that when HA exceeds 15%, HA dramatically influences the UCS of cemented soil. The reason is that incorporating HA weakens the cementation effect of hydration products on soil particles. Based on previous studies, this paper focuses on the microcosmic study of the impact of humic acid on cemented soil. It more comprehensively demonstrates why humic acid inhibits the development of cemented soil strength. In addition, this paper shows that a 30% cement ratio can improve the mechanical properties of the soil. The technique can thus be promising. However, 30% cement is neither economical nor environmentally friendly. More work is needed to see if a target improvement can be reached using a smaller cement ratio.

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