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Engineering properties and microcosmic mechanism of cement stabilized diatomite

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In this study, the engineering properties of remolded diatomite and the effects of cement on the compression characteristic, strength properties and microstructures of cement-stabilized diatomite were investigated. Samples were prepared and stabilized with different cement content ratios, ranging from 0% to 15% by dry mass. Results show that compared with undisturbed diatomite, the compressibility of the remolded diatomite increases while the strength characteristics decrease. With the increase of cement content, the compressibility of cement-stabilized diatomite is significantly reduced and the strength characteristics are improved. Adding cement to diatomite changes the structure of pure diatomite and forms more tiny pores between cement and diatomite, while curing reduces the porosity ratio of samples and enhance the strength of cement-stabilized diatomite, especially for diatomite with higher cement content. The physical-chemical reactions including hydrolysis and hydration between cement and diatomite increase the content of sodium aluminosilicate, calcium aluminosilicate and other minerals in the soil.

KEYWORDS

diatomite, cement, cure time, engineering properties, microcosmic mechanism

Introduction

Diatomite is a kind of biochemical deposition formed by diatom after its death during the accumulation period of 10,000–20,000 years. It belongs to the siliceous rocks, and its chemical composition is mainly SiO_2, which can be expressed by $SiO_2 \cdot nH_2O$.It is mostly deposited in Miocene and Pliocene age (Harben, 2002), China is one of the countries with the most extensive distribution of diatomite in the world and diatomite were found in 18 provinces (Cui, 2008). Diatomite was usually studied as a material for industrial applications in building, bleaching, filtering, and so on (Stoemer and Smoll, 2001; Fragoulis et al., 2005; Van Garderen et al., 2011). In recent decade, with the continuous development of foundation engineering, civil engineering construction is developing towards various complex geological conditions (Yang et al., 2022; Ma et al., 2021; Zhou et al., 2022; Liu et al., 2022; Qiu et al., 2022; Wang et al., 2022). In the construction of railways and highways, diatomite areas are often encountered along the route and the engineering characteristics of diatomite is also beginning to attract researchers' attention (Koizumi et al., 2009; Calvo et al., 2012).

Some scholars have studied the engineering properties and strength characteristics of diatomite, Day (1995) indicated that although diatomite has lower density and higher water content, it has lower compressibility and higher shear strength due to its good biting force and surface roughness of the original soil, the similar results also have been obtained by Tateishi (1997). (Hong et al., 2004a) studied the mechanical properties of natural diatomite in oita prefecture, Japan, providing that before the consolidation pressure reaches the consolidation yield stress, the compression curve is horizontal, but when the consolidation pressure reaches the yield stress, the compressibility will increase sharply, indicating that the original diatomite has a strong structure. The macro-and micro properties also have been investigated by Hong et al. (2006). Zhang et al. (2013) made study the mineralogical compositions, physical property, mechanical and engineering properties of diatomite in Tengchong region of Yunnan Province, Southwest China, results indicated that diatomite is a kind of strong structural soil, both the composition and the structure of diatomite could lead to its different mechanical and engineering properties.

Although the undisturbed diatomite has high structural strength, its mechanical strength will change after its structure is destroyed because of the extremely absorbent clay minerals it contains. However, in many projects, such as the construction of roads and railways, a large amount of backfill will be used, which will inevitably disturb and reshape the local soil, therefore, it is necessary to study the mechanical behavior of reconstructed diatomite.

Meanwhile, in the treatment of soil with poor geotechnical performance, a widespread used technique is to add modifier to improve the engineering properties, and the most commonly used modifier is cement, it has been used for many special soils. Sariosseiri and Muhunthan (2009) investigated the treatment effect of cement on geotechnical properties of Washington State soils; indicating that both the strength characteristics and workability are improved and the cement content should not be higher than 10%. Lemaire et al. (2013) studied the microstructure and macroscopic mechanical properties of plastic silt treated with combined cement and lime, found that the mechanical strength of soil has been significantly improved due to the change of microstructure of treated soil. Ho et al. (2017) investigated the effects of water content, carbonation, and pozzolanic reaction on the strength development of cement-treated soils under the drying condition. And the strength development of sand, sand-loam, and sand-bentonite mixtures treated with cement, founded that carbonation could have both positive and negative impacts on strength development of cement-treated soils were also studied (Ho et al., 2018). Moreover, some scholars have also studied other engineering properties of cement-treated soil, such as the resistance to liquefaction of loess treated with cement (Qian, 2016), the consolidation properties of compacted lateritic soil treated with cement (Mengue et al., 2017), the dynamic response of expansive soil treated with cement (Cai et al., 2020), and the hydraulic conductivity and strength of marine clay treated with foamed cement (Wu et al., 2019). However, the engineering properties of cement treated diatomite have not been studied.

Therefore, in this study, based on geotechnical tests and microstructure tests, the engineering properties, mineral content change and microstructure evolution of remoulded diatomite and cement-treated diatomite were investigated. The influence of cement content and curing period on these properties were obtained and analyzed.

Marerials and methods

Materials

The diatomite used in this investigation is obtained from the City of Shengzhou in Zhejiang province in China. It is grey white and collected at a depth between 3.0 and 5.0 m. The main mineral of diatomite is SiO_2, followed by $CaCO_3$, a small amount of Al_2O_3, Fe_2O_3, CaO, etc., and a certain amount of organic matter, the details of mineral component and the clay mineral composition are shown in Table 1. Table 2 lists the basic physicochemical properties of diatomite in terms. The particle size distribution curve of diatomite is shown in Figure 1, in which fine soil accounts for 98.24%, and part of coarse-grained soil has a particle size less than 0.25 mm.

The cement was 42.5# ordinary Portland cement supplied by Jingyang Cement Company (Shanxi province, China). It is an off-white soil, thin powder with a natural moisture content of 0.60% and a relative density of 2.58.

TABLE 1 Main chemical and clay mineral compositions of diatomite.

Mineral	Content/%
SiO_2	84.43
$CaCO_3$	5.27
Al_2O_3	2.34
Fe_2O_3	1.03
CaO	0.72
MgO	0.69
Loss on ignition	3.56
Other substance	1.96
Illite/Montmorillonite mixed layer (S/I)	47.29
Effective montmorillonite	23.88
Illite (I)	7.69
Kaolinite (K)	14.63

TABLE 2 Basic physicochemical properties of diatomite.

Property	Value
Specific gravity/g/cm^3	2.51
Specific surface area/m^2/g	249.32
Nature water content/%	49.5
Natural density/g/cm^3	1.65
Nature void ratio	1.26
Nature compressibility	0.27
Liquid limit/%	82.3
Plastic limit/%	40.6
Maximum density/g/cm^3	1.15
Optimum water content/%	38.4

Methods

Cement content ratio (C_c) is defined as the ratio of the dry mass of cement to the dry mass of the diatomite-cement mixture. For the sample preparation, the diatomite has an air-dried water content of 32.4%, diatomite is carefully mixed with cement of different content ratios (0%, 3%, 6%, 9%, and 15%) and distilled water to produce mixtures to the natural water content uniformly.

Samples were prepared by statically compacting in a confining ring with an inner diameter of 61.8 mm and a height of 20 mm (for swelling pressure and direct shear test) and a confining ring with an inner diameter of 50 mm and height of 100 mm (for unconfined compressive strength test). The static compaction speed was 5 kN/s and the maximal load was 20 MPa. The sample was pushed out of the apparatus 10 min after the height had reached the predetermined height. A rebound of less than 0.01 mm was observed in the height of the sample after the unloading stage was complete and this was deemed to be negligible. The density of the sample was 1.65 g/cm^3 determined according to the nature density for undisturbed diatomite (Table 1).

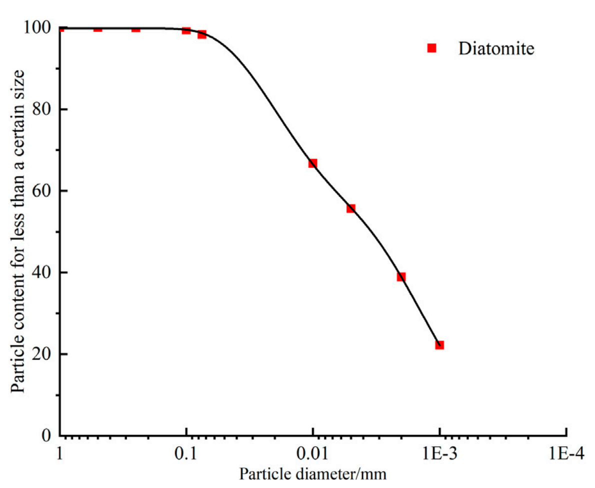

FIGURE 1
Particle size distribution curve.

Swelling pressure tests were performed in a swelling pressure testing apparatus. Direct shear test and unconfined compressive strength test were carried out respectively to determine the cohesion (c), internal friction angle (φ) and unconfined compressive strength (UCS). Direct shear takes the type of fast shear for this study, that is, the shear speed was defined as 0.8 mm/min. And the compression coefficient of soil sample was measured by uniaxial compression test on the consolidation instrument. In addition, in order to investigate the influence of curing age on mechanical behavior of cement-modified diatomite, the samples mixed with different cement ratios were cured for 1 day, 7 days, 14 days and 28 days respectively. Then the direct shear test and unconfined compressive strength test were carried out.

To further investigate the microstructure changes of diatomite stabilized by cement, X-Ray diffraction (XRD), Mercury intrusion porosimetry (MIP) and scanning electron microscope (SEM) were used. MIP used in this study is to determine the pore size distribution in porous materials based on the unique relationship between intrusion pressure and equivalent pore diameter. as proposed by Washburn (1921): $D{=}-(4\gamma\cos\theta)/P$. In this test, θ was 140° and γ was 0.480 N/m, the measured pore sizes ranged from 0.005 μm to 340 μm.

Results

Engineering properties of remolded and undisturbed diatomite

The engineering properties of remolded and undisturbed diatomite are shown in Table 3. Compared with undisturbed diatomite, it can be seen the compression coefficient of the remolded diatomite is increased by 51.2%; while the values of cohesion, internal friction angle and *UCS* (Unconfined Compressive Strength) are reduced by 52.95%, 56.88% and 57.14% respectively. These results indicate that the remolded soil sample destroys the original structure of the undisturbed soil, although the remolded soil has the same initial conditions as the undisturbed soil, its compressibility is increased and strength characteristic is reduced due to the lack of good engineering performance provided by the soil structure. It is worth mentioning that the value of the maximal swelling pressure is very low and basically unchanged, which is attributed to the high porosity of diatomite, most of the expansion is consumed in the reduction of pore volume,

TABLE 3 Engineering properties of remolded and undisturbed diatomite.

Soil type	Maximal swelling pressure/kPa	Compression coefficient $a_{1\text{-}2}$/MPa	Cohesion/kPa	Internal friction angle/°	*UCS*/MPa
Undisturbed diatomite	21.57	0.41	142.6	21.8	0.98
Remolded diatomite	20.68	0.62	67.1	9.4	0.42

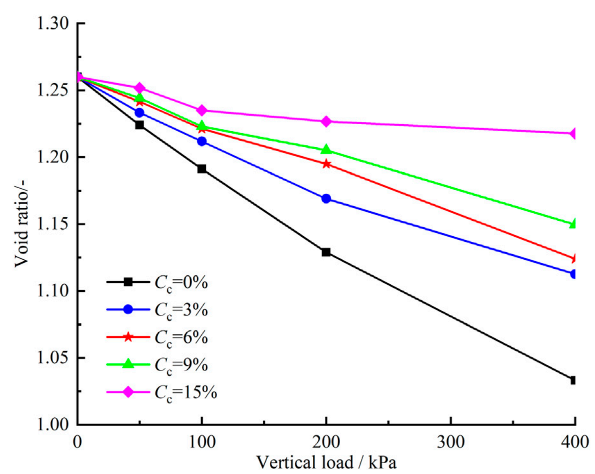

FIGURE 2
e-p curves of cement modified diatomite.

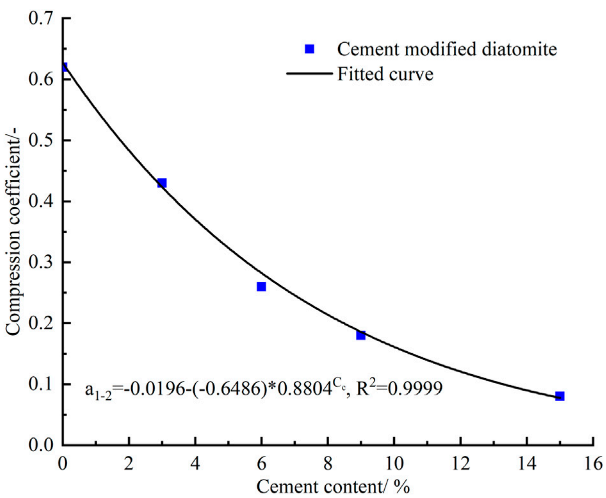

FIGURE 3
Compression coefficient vs. cement content.

which is called "internal expansion" (Zhang et al., 2013), so it cannot form a very high expansion force. Meanwhile, the results also indicate that the damage of soil structure has little effect on the soil expansibility.

Effect of cement content on compressibility of diatomite

Unidirectional compression tests were carried out on diatomite samples with different proportions of cement, and the results are shown in Figure 2, it can be seen that with the increase of cement content, the falling gradient of *e*-p curves of the samples slows down significantly. For remolded pure diatomite sample, with the increase of the load, the compressive deformation of the sample continues to increase, and the void ratio decreases from the initial 1.260 to 1.033 under the loading of 400 kPa, decreasing by 0.227 kPa; when the cement content is 3%, it decreases by 0.147 kPa, which is only 64.8% of pure diatomite. And when the cement content is 15%, the void ratio is 1.128 under the loading of 400 kPa, decreasing by 0.042, which is only 18.5% of pure diatomite sample.

Compressibility coefficients of 100–200 kPa, $a_{1\text{-}2}$(MPa), were calculated, which is a parameter used to judge the compressibility of the sample, and the relationship between $a_{1\text{-}2}$ and cement content is listed in Figure 3. Result shows that $a_{1\text{-}2}$ decreases exponentially with the increase of cement content, and the fitting formula is as follows: $a_{1\text{-}2}$=−0.0196−(−0.6486)*0.8804^{Cc}, R^2=0.9842.

The void ratio of pure diatomite sample is 0.62, and the void ratios of 3% and 15% cement samples are 0.43 and 0.08 respectively. According to the specification, soil with $a_{1\text{-}2}$≥0.5 MPa is defined as high compressibility soil, soil with 0.1 MPa≤$a_{1\text{-}2}$<0.5 MPa is defined as medium compressibility soil, and soil with $a_{1\text{-}2}$<0.1 MPa is defined as low compressibility soil. Therefore, the sample of remolded pure diatomite belongs to high compressibility soil. The sample with 3% cement is reduced to medium compressibility soil, and when the cement content is 15%, it is directly reduced to low compressibility soil. It shows that adding cement to diatomite can significantly reduce the compressibility of soil. It's worth noting that when the cement content is 3%, the value of $a_{1\text{-}2}$ is close to that of undisturbed diatomite (0.41 MPa).

Effect of cement content on strength properties of diatomite

Direct shear tests were conducted on cement-stabilized diatomite and the shear strength parameters were obtained. Figure 4 shows the typical shear stress-shear deformation

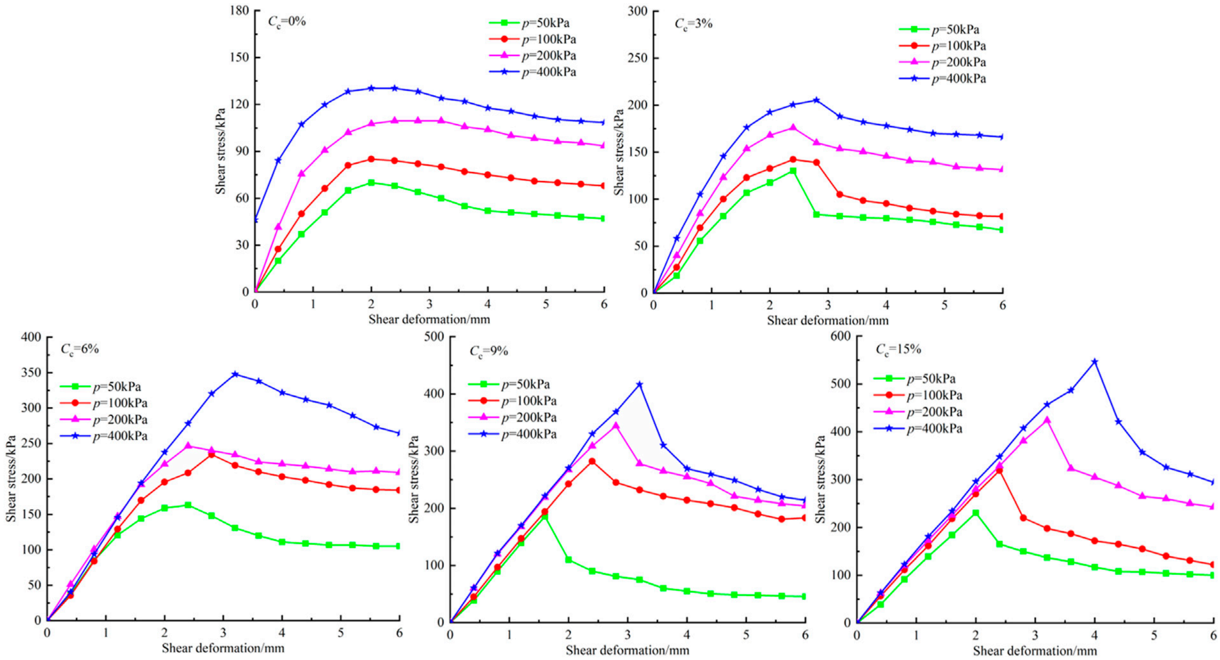

FIGURE 4
Shear stress-shear deformation.

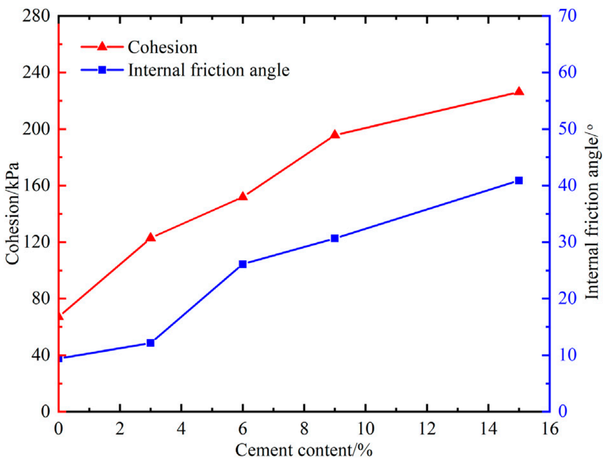

FIGURE 5
Shear strength parameters vs. cement content.

curves for the samples of diatomite with different cement contents, results display that shear strength is enhanced as cement content increases. It's worth noting that with the increase of cement content, the stress-deformation relationship of samples gradually changes from strain hardening to strain softening, when the cement content is 6%, the stress-strain curve shows a very obvious peak strength. These indicate that adding cement to diatomite makes the soil harder and more brittle, which means that the shear behavior is like that of rock and the soil is harder.

The change of cohesion (c) and internal friction angle (φ) with cement content are shown in Figure 5, it shows that with the increase of cement content, both cohesion and internal friction angle increase. Moreover, when the cement content is 6%, the shear strength parameters are close to that of the undisturbed diatomite samples, indicating that cement has an obvious effect on improving the shear strength of diatomite. However, the strength growth rate is not invariable, with the increase of cement content, the increasing effect of cement on soil strength gradually decreases.

Figure 6 shows the result of Unconfined Compressive Strength test, it can be seen after adding cement to diatomite, the soil gradually transforms from strain hardening type to strain softening type.

Figure 7 shows the relationship between *UCS* value and cement content. The *UCS* value increases exponentially with the increase of cement content, similar to the shear strength parameter, when the cement content is 6%, the *UCS* value of the sample exceeds that of undisturbed diatomite, meaning that adding cement significantly improves the strength of soil.

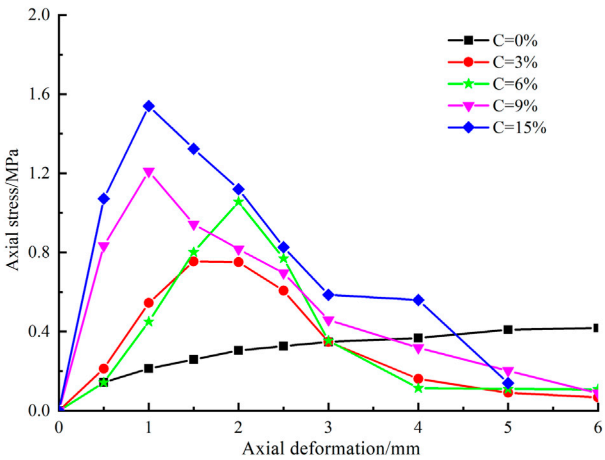

FIGURE 6
Stress-strain relation curve of unconfined compressive strength test.

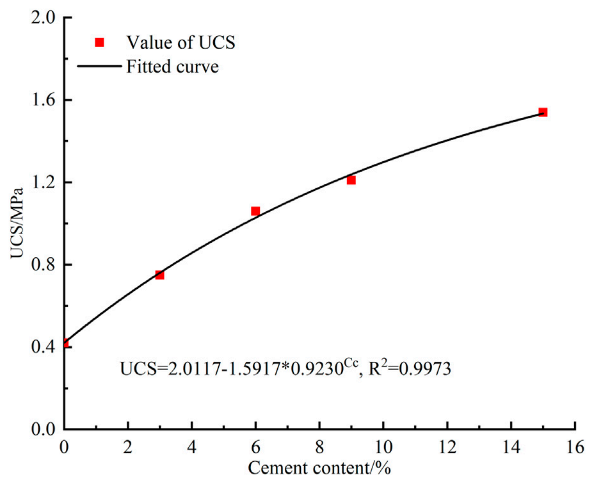

FIGURE 7
Unconfined compressive strength vs. cement content.

Influence of curing period on strength characteristics of cement-stabilized diatomite

In order to clarify the influence of curing period on strength characteristics of cement-modified diatomite, samples with different cement content were cured for 0 days, 7 days, 14 days and 28 days separately, and then direct shear test and Unconfined Compressive Strength tests were conducted on them.

The variation of shear strength parameters with curing period are shown in Figure 8. Curing has almost no effect on pure diatomite. The effect of curing on cohesion and internal friction angle is quite different. For internal friction angle (Figure 8A), the values are increased with the increase of curing time, but the increase rate are different, the value of the sample with 3% cement content increases significantly, while these values of samples with other cement content increase slowly. For cohesion (Figure 8B), curing can significantly improve the value of cohesion, and the strength improvement is mainly reflected in the early curing stage, which has reached more than 85% of the stable value within 14 days. When curing period is more than 14 days, the value of cohesion increases slowly. In addition, results indicate that the value of cohesion of sample with 3% cement content increases from 122.9 kPa to 194.1 kPa after curing for 28 days; While these two values are 226.2 kPa and 648.7 kPa respectively for sample with 15% cement content, which is 5.93 times than that of the sample with 3% cement content, indicating that the curing effect on the sample with higher cement content is more significant.

The UCS value of diatomite with various cement content after different curing period are determined and presented in Figure 9. Results indicate that the value of sample without cement has almost no change with curing time. While the value of other samples with different proportions of cement are increased with curing period and gradually stabilized. And there is little difference in the increase rate of samples with different the cement content.

Microscopic mechanism study

Figure 10 displays the cumulative pore volume curves and the pore size distribution curves of samples with various cements content and samples with 15% cements content after various curing period. It can be seen from Figure 10A, the total pore number of the sample increases with the increase of cement content, but the value of sample with 15% and 9% cement content is almost the same, and from Figure 10C, it can be concluded that the main increased pore size range is 0.01–10 um. While the total pore number of the sample decreases with the growth cure time (Figure 10B), and reduction in pore size is concentrated in 0.1–0.01 μm (Figure 10D).

Meanwhile, the results of both cumulative pore volume curves and pore size distribution curves show that most of the pores in the sample are less than 10um. Furthermore, the results of SEM (Figure 11) also provide sufficient evidence for the phenomenon, indicating that the diameter of diatom remains are mostly less than 10 μm (Figure 11A), and adding cement to diatomite changes the structure of soil and increases the number of pores with diameters ranging from 0.01 um to 0.4 um, (Figure 11B), which are classified as inter-granular pores according to the pore classification proposed by Shear et al. (1993).

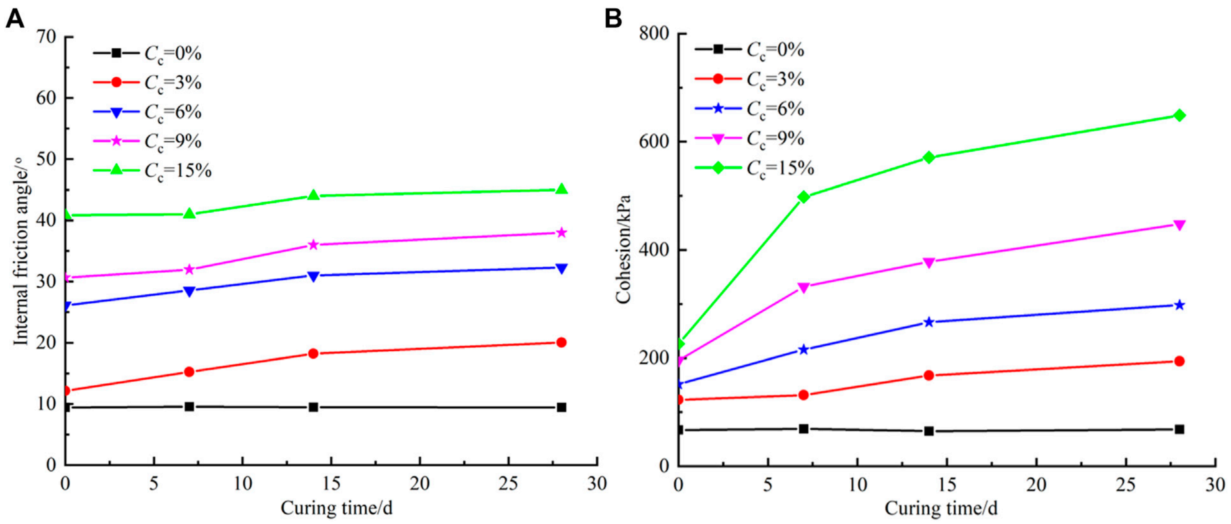

FIGURE 8
Shear strength parameters vs. cure time.

Discussion

The above research results show that adding cement to diatomite can significantly reduce its compressibility and increases its strength. For adding cement can change the structure of pure diatomite and form more tiny pores between cement and diatomite, and the number of pores stabilizes when the cement content reaches 9%, then with the increase of cement content, the total porosity is basically unchanged, but the number of micropores has been increased.

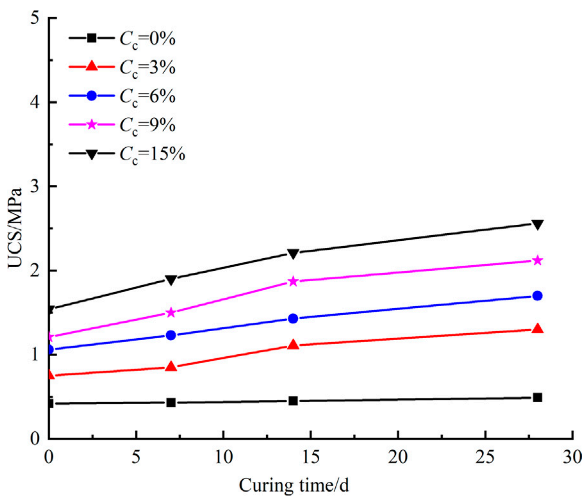

FIGURE 9
Unconfined Compressive Strength vs. cure time.

However, curing can reduce the number of pores, with the growth of cure period, the total pore number of sample decreases (Figure 10B), and at the curing age of 7 days, the void ratio of the sample is 0.59, which is lower than that of pure diatomite (Figure 10A). the missing pores are mainly in the range of 0.01–0.1 um in diameter, and the number of pores with diameters of 0.1um–0.4 um also decreases (Figure 10D). These results also can be verified by SEM (Figure 11C), When the curing period is 28 days, the tiny pores in the sample of cement-modified diatomite are significantly reduced and the sample presents a condensed structure, which is more dense than that of pure diatomite.

Cement can improve soil properties through a series of physical-chemical reactions including hydrolysis and hydration. The main composition of cement is silicate, including tricalcium silicate ($3CaO{\cdot}SiO_2$), dicalcium silicate ($2CaO{\cdot}SiO_2$), tricalcium aluminate ($3CaOAl_2O_3$), etc. When diatomite is mixed with cement and water, the hydration reaction occurs rapidly, forming hydrates such as hydrated calcium silicate ($xCaOSiO_2{\cdot}nH_2O$) and calcium hydroxide ($Ca(OH)_2$). Then $Ca(HO)_2$ diffuses to the surface of diatomite particles and gradually eroded the active CaO, Al_2O_3 and Fe_2O_3 to generate hydrated calcium silicate ($xCaOAl_2O_3nH_2O$), hydrated calcium aluminate ($xCaOAl_2O_3yCaO_3nH_2O$) and hydrated calcium ferrite ($xCaOFe_2O_3nH_2O$).

It can be seen from the XRD spectrums of diatomite and 15% cement-stabilized diatomite with curing period of 0 and 28 days (Figure 12), after the addition of 15% cement, calcite content as well as sodium aluminosilicate and calcium aluminosilicate minerals increased significantly. At the curing period of 28 days, the contents of odium aluminosilicate and calcium aluminosilicate minerals in diatomite with cement content of 15% are increased, indicating that with the growth of curing

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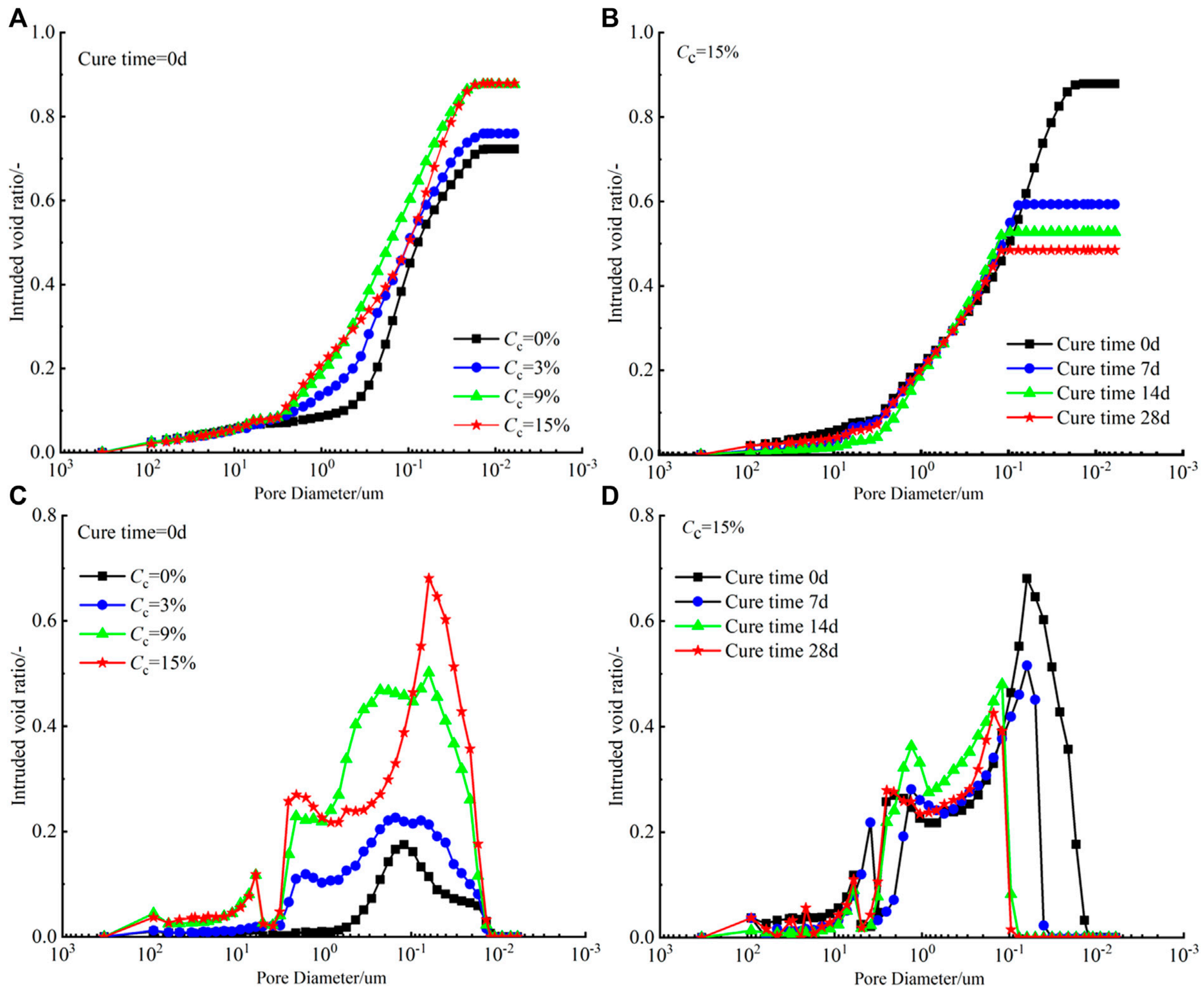

FIGURE 10
Cumulative and pore size distribution curves of cement modified diatomite.

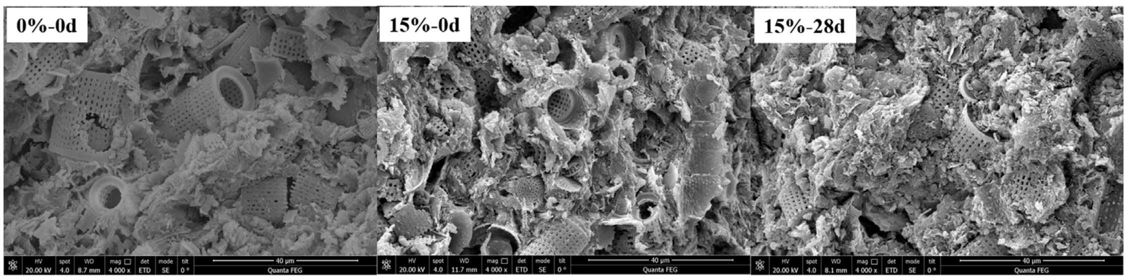

FIGURE 11
SEM of cement modified diatomite.

period, hydration reactions in cement-stabilized diatomite soils develop continuously and more hydrates are generated. These hydrates connect to the particles and filled the void and continues to harden to form the skeleton of cement stone with high strength, while the aggregate of soil is similar to the concrete, which increases the strength of diatomite.

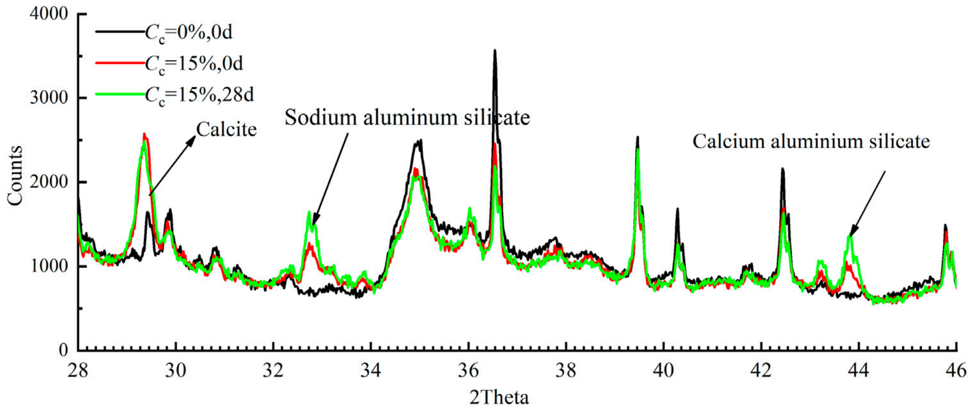

FIGURE 12
XRD spectrums of cement modified diatomite.

Conclusion

The microstructure of remolded diatomite is changed, and thus its engineering properties is affected. The purpose of this paper is to find out the changes of engineering properties of remolded diatomite compared with undisturbed diatomite, study the engineering properties and microstructure of cement modified diatomite by adding different proportions of cement into diatomite. The following conclusions are drawn from the present study.

1 Compared with undisturbed diatomite, the compression coefficient of remolded diatomite is increased by 51.2%; while the values of cohesion, internal friction angle and UCS are reduced by 52.94%, 56.74% and 57.14% respectively; the value of the maximal expansion force is very low and basically unchanged.
2 Adding cement to diatomite makes the pore ratio no longer decrease obviously with the increase of load, and makes the stress-strain curve of samples gradually change from strain hardening to strain softening. With the increase of cement content, both cohesion and internal friction angle increase, and the value of *UCS* increases exponentially while compression coefficient $a_{1\text{-}2}$ decreases exponentially.
3 Curing can improve the internal friction angle of cement stabilized diatomite, but the effect is not very obvious, only the sample with cement content of 3% has a relatively obvious improvement. However, the values of cohesion and *UCS* are significantly improved with the increase of curing time, and the strength improvement is mainly reflected in the early curing stage, the curing effect on the sample with higher cement content is more significant.
4 Adding cement to diatomite changes the structure of pure diatomite, forms more tiny pores between cement and diatomite and increases the total pore number of the sample; while with the increase of curing period, the total pore number of sample decreases.
5 Adding cement to diatomite increases the amount of calcite content, sodium aluminosilicate and calcium aluminosilicate minerals, and curing also increases the contents of odium aluminosilicate and calcium aluminosilicate minerals in diatomite with 15% cement content.

Data availability statement

The original contributions presented in the study are included in the article/supplementary material, further inquiries can be directed to the corresponding author.

Author contributions

SC, ZT, and YZ contributed to conception and design of the study. ZT, YZ, HS, and YJ performed the experiments and statistical analysis. SC performed the theoretical analysis, ZT wrote the first draft of the manuscript. YZ, HS, and YJ wrote sections of the manuscript. All authors contributed to manuscript revision, read, and approved the submitted version.

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Conflict of interest

ZT was employed by the Xi'an Aerospace Construction Supervision Limited Company.

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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