Effects of calcium carbonate on hydro-mechanical properties of bentonite

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Abstract. To simulate characteristic changes of the bentonite mixing with CaCO₃ crystals that precipitate from groundwater around the high-level radioactive waste repositories, different amounts of CaCO₃ powders were mixed in the bentonite. In this study, four series of bentonite/CaCO₃ mixtures were prepared with different contents of CaCO₃ powders. A series of swelling, compression and direct shear tests were conducted on the bentonite/CaCO₃ samples using deionized water. Terzaghi consolidation theory was adopted to compute the hydraulic conductivity of bentonite/CaCO₃ mixtures. The results demonstrated that the final swelling strain decreased with increasing vertical stress and the content of CaCO₃ powders. The compression index decreased slightly with increasing CaCO₃ content. It was found that as the CaCO₃ content increased, the hydraulic conductivity of bentonite/CaCO₃ mixtures decreased. There was a peak point in the shear strength of unsaturated samples with the differences of the CaCO₃ content. The cohesion of unsaturated samples increased first and then decreased, but saturated samples presented the opposite character. It could be seen from the scanning electron micrograph that the existence of CaCO₃ powders filled pores of bentonite/CaCO₃ mixtures and made the structure denser.

1 Introduction

Deep geological disposal has been widely adopted by many countries as a safe and effective treatment to deal with the long-term radioactive waste. For disposal of the high-level radioactive waste (HLW), the bentonite or bentonite-based materials are generally used as buffer materials due to their favorable properties such as high swelling, high radionuclide retardation and low permeability [1-5].

For the security of repository, the bentonite materials have been widely investigated for their mechanical properties [6-10] and hydraulic behavior [11-14]. Performance of the bentonite in the repository is normally affected by high temperature [15-18], chemical effects [20-23] and gas [24-29], and even the coupling of several situations. Thus, it is necessary to study properties of the bentonite under different surroundings.

With the closure of the repository, groundwater intrudes into the bentonite continuously. This means that many ions which are soluble in the groundwater can be bought into the bentonite, including the lower hydration energy of the exchangeable monovalent cation such as Na⁺ and divalent cation Ca²⁺ [30]. Meanwhile the surrounding environment also has many anions such as bicarbonate ions and carbonate ions. Guo et al. [31-32] investigated the groundwater composition in Beishan (Gansu province of China) and Yumeng Su (Xinjiang

province of China), and analyzed the chemical characteristics. They found that the groundwater in the researched area was mainly saline with high dissolved solids, 7.39-10.14 pH value, 8-17°C temperature, 0.7-19g/L total dissolved solids. It was concluded that the groundwater of Beishan mainly composed of sodium ions, calcium ions, sulfate ions and bicarbonate ions. Bicarbonate ions are likely to be converted to carbonate ions in alkaline conditions (Eqs. (1) and (2)) and most minerals in groundwater had reached saturation in this area. With the increase of salinity, calcium ions in the groundwater combine with carbonate ions to form precipitates.

In addition, the gases such as carbon dioxide, hydrogen and methane can be produced due to the microbial degradation process in the long-term operation of the deep geological repository system. carbon dioxide produced by microorganism can increase the gas pressure in the bentonite and facilitate the formation of bicarbonate ions and carbonate ions, which combine with calcium ions to form precipitates. Meanwhile, Sasaki et al. [33] reported that the microbial respiration can induce calcium carbonate in the soil.

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It can be seen that there are several ways of forming CaCO_3 crystals. Therefore, it is extremely important to study the effect of CaCO_3 crystals on properties of the bentonite. In this paper, experiments were performed on the bentonite with different calcium carbonate mixing ratios (CMR) to study its swelling, compression, and permeability properties. The study of strength characteristics is divided into saturated and unsaturated to simulate the near heat source point and near rock point in the repository respectively. Afterwards, the conclusions were verified based on the analysis of testing results.

2 Materials and experimental methods

2.1 Materials and sample preparation

The bentonite used in the experiment was taken from Qitai County, Xinjiang, which is one of the Chinese nuclear waste buffer materials. The bentonite was crushed through a 0.5 mm sieve before the experiment. Basic properties of the bentonite were shown in Table 1. CaCO_3 powders which were white powders and composed of aragonite and calcite crystals with a density of 2.72 g/cm^3 were produced by Sinopharm Group (No.10005760). In order to avoid the influence of the metal ions on the bentonite properties, the deionized water was used.

Table 1. Basic properties of the bentonite.

Parameter	Value
Specific gravity of soil grain	2.71
Liquid limit w_L (%)	152.3
Plastic limit w_P (%)	52.5
Ion exchange capacity (mol/100g)	94.65
Mainly exchangeable ions (mol/100g)	Na^+ / 65.18 $1/2\text{Ca}^{2+}$ / 21.60 $1/2\text{Mg}^{2+}$ / 6.34 K^+ / 0.52
Main mineral composition	Montmorillonite / 75.9% Quartz / 14.2% Feldspar / 5.9% Gypsum / 4.0%
Na_2O (%)	2.96
CaO (%)	1.2

The bentonite and CaCO_3 powders used in the experiment were placed into a 105°C constant temperature oven to keep them completely dry firstly. 24 hours later, these specimens were removed immediately and then the different amounts of CaCO_3 powders (0%, 3.125%, 6.25% and 12.5%) were mixed with the bentonite samples after cooling down. The water content of the target was 15%. Then these mixtures were maintained 24 hours to ensure that water in the mixed samples was evenly distributed. According to the initial overall dry density (1.6 g/cm^3) of the target and the

suitable volume of different samples, the quantity of mixed samples was calculated. Finally, compressed unsaturated samples were obtained by CTM8050 universal testing machine. The initial state of each sample was shown in Table 2.

2.2. Experimental method

The whole experimental process included four tests: 1) Swelling test, 2) Oedometer test, 3) Scanning electron microscope test and 4) Shear test.

Table 2. Initial states of samples

NO.	CMR (%)	Height (mm)	Diameter (mm)	Bentonite dry density (g/cm^3)	Swelling test	Oedometer test	Direct shear test
1	0	10	61.8	1.6	√	√	
	0	15	61.8	1.6	√		√
	0	15	50	1.6			√
2	3.125	10	61.8	1.55	√	√	
	3.125	15	61.8	1.55	√		√
	3.125	15	50	1.55			√
3	6.25	10	61.8	1.5	√	√	
	6.25	15	61.8	1.5	√		√
	6.25	15	50	1.5			√
4	12.5	10	61.8	1.4	√	√	
	12.5	15	61.8	1.4	√		√
	12.5	15	50	1.4			√

2.2.1. Swelling test. As shown in Figure 1, the compressed samples were placed in the oedometer apparatus. While the vertical prestressing of 1kPa was applied, the initial reading of the dial gauge was recorded. Then the target vertical stress was reached (the samples with the height of 15mm were loaded to 200, 400 and 800kPa, respectively; the samples with the height of 10mm were loaded to 50kPa) through step loading method. The deionized water was not injected into the consolidation cell until the reading of dial indicator remained stable (the reading changed 0.01mm within 1h). Finally, when the change of the reading of dial gauge was less than 0.01mm/24h, the loaded swelling test was terminated.

Figure 1. The oedometer apparatus.

The swelling strain was adopted to describe swelling properties of the bentonite. The swelling strain was defined as:

$$\varepsilon = \frac{\Delta H}{H_0} \times 100\% \quad (3)$$

Where ε is the swelling strain, ΔH is the vertical deformation (mm) and H_0 is the initial height of samples (mm).

2.2.2. Odometer test. After swelling tests were completed, the samples of 10mm height were loaded under prescribed stress paths (50kPa -100kPa -200kPa -400kPa -800kPa -1600kPa). This part mainly focused on the compressive coefficient of samples, and did not take into account the effect of the secondary consolidation deformation during the loading process. So, after 12 hours, the samples were considered to be stable when the vertical deformation was less than 0.01mm per hour. The vertical consolidation coefficient C_v under different vertical stress was measured by the time square root method:

$$C_v = \frac{0.848h^{-2}}{t_{90}} \quad (4)$$

Where C_v is the consolidation coefficient (cm^2/s), $\bar{h}$ is the maximum drainage distance (cm), t_{90} is the time required for consolidation to reach 90% (s).

According to Terzaghi's one-dimensional consolidation theory, the hydraulic conductivity of clay can be calculated (Sun et al. 2015):

$$k = c_v m_v \gamma_w \quad (5)$$

Where k is the hydraulic conductivity (cm/s), m_v is the volume compressibility (m^2/kN), γ_w is the gravity of water (kN/m^3).

2.2.3. Scanning electron microscope test. In order to fully understand profoundly the macro performance changes of samples, the microstructure morphology of samples with the height of 10mm, the CMR of 0% and 12.5% was conducted by the scanning electron microscope. The apparatus used in the test was FEI Nova NanoSEM NPE produced by the USA.

The samples (the CMR of 0% and 12.5%) were prepared after the swelling test (400kPa vertical stress). Then some small elements with $2.5\text{mm} \times 2.5\text{mm} \times 5\text{mm}$ that were cut out of the samples had to be dehydrated by liquid nitrogen. Finally, the microstructure morphology was conducted by the scanning electron microscope.

2.2.4. Direct shear test. In order to fully understand profoundly the macro performance changes of samples, the microstructure morphology of samples with the height of 10mm, the CMR of 0% and 12.5% was conducted by the scanning electron microscope. The apparatus used in the test was FEI Nova NanoSEM NPE produced by the USA.

Figure 2. HUS-25sixty shear direct shear apparatus.

3 Experimental results and analysis

3.1 Swelling test

Figure 3a-3c presented the swelling strain vs. the time for three different vertical stresses (200kPa, 400kPa and

800kPa). As shown in Figure 3a-3c, it could be seen that while the vertical stress increased, the swelling strain of bentonite/CaCO₃ mixtures decreased. And under the same vertical stress, the swelling strain of samples decreases with the increase of the CMR under different vertical stresses. This phenomenon may be explained that the montmorillonite, which was the main swelling mineral in the bentonite, decreased as the content of CaCO₃ powders increased under the same initial dry density. According to diffuse double layer theory [9], the repulsion between the bentonite particles decreased with the reduction of the content of montmorillonite. Especially under the low stress, the effect of CMR on the swelling strain was more obvious. At the same time, Figure 3a-3c also reflected the time required for the samples to stabilize. It could be found that the more the content of CaCO₃ powders, the less the time required for the test. Under a vertical stress of 400kPa, the test used with the pure bentonite took 14 days until the test was completed, and it took 10 days to complete the test with a mixed sample (the CMR was 12.5%).

Sridharan et al. [34] reported three clear phases in swelling, viz., the initial swelling, the primary swelling, and the secondary swelling (Figure 4). The results (Figure 3) were consistent with that of Figure 4 and with the “S” shape.

Figure 5 presented some methods of tests for measuring the swelling pressure [35]. As shown in Figure 5, Gao et al. [35] introduced three methods for calculating the swelling pressure: the free swell and load test, the swell-under-load test and the constant volume test. The sample conducted by swell- under-load test was swelling in the case of one-dimensional, and then a vertical stress was applied to recover the initial void ratio, which was the highest value of the swelling pressure (σ_{s1}). σ_{s2} , which was determined by the constant volume test, was generally considered to yield higher values than that of σ_{s1} and σ_{s3} was determined by the free swell and load test. But the value of σ_{s3} was too large to be used as the swelling pressure of the bentonite due to the compression deformation of the unsaturated bentonite increased with increasing vertical stress before wetting, while σ_{s2} was not unique under different constant vertical stresses. Thus, σ_{s1} was more reasonable to use as the swelling pressure of the bentonite.

Figure 3. Swelling strain developed with time for bentonite/CaCO₃ mixtures under different vertical stress.

Figure 4. Scheme of initial, primary, and secondary swelling stages[34].

Figure 5. Methods of tests for measuring swelling pressure.

As shown in Figure 6, the swelling pressure of mixed samples decreased with the increase of CMR. According to the diffuse double layer theory, the repulsion between the clay particles could decrease with the increase of the content of CaCO_3 powders due to the reduction of the content of montmorillonite per unit volume.

Figure 6. Swelling pressure for bentonite/ CaCO_3 mixtures.

3.2 Oedometer test

The compression curve of bentonite/ CaCO_3 mixtures was shown in Figure 7. The compression curve presented a bilinear characteristic with an obvious inflection point. This phenomenon may be explained that with the increase of vertical stress, the transition of samples was generated from the elastic deformation of the original drainage to the plastic deformation of the structural failure. The compression becomes more difficult due to the further compact contact between particles.

Figure 7. Void ratio vs. vertical pressure.

According to Figure 7, the compression index for bentonite/ CaCO_3 mixtures was obtained in Figure 8. A line was obtained by linear fitting, and the fitting equation was Eq. (6). The compression index decreased slightly with the increase of CaCO_3 content.

$$C_c = -0.273\text{CMR} + 0.467 \quad (6)$$

Where C_c is the compression index. According to Eq. (6), the compression indexes of the bentonite mixed with any CMR can be predicted ($\rho_s = 1.6 \text{ g/cm}^3$).

It has been found that the determination of consolidation coefficient by the square root method was available to deduce the hydraulic conductivity of clay sample [36]. In the paper of Sun et al. [36], this method was used to remove the anomalous points due to the interaction of the volume compression coefficient and the consolidation coefficient. It can be seen from Figure 9 that the hydraulic conductivity of samples decreased with the increase of the content of CaCO_3 powders, as a consequence of the quite small size of the CaCO_3 crystal particles which existed and blocked the pores.

Figure 8. Compression index for bentonite/ CaCO_3 mixtures.

Figure 9. Hydraulic conductivity vs. void ratio.

Figure 10. SEM photomicrographs of bentonite/CaCO₃ mixtures under 400kPa vertical stress (20000*): (a) CMR-0%; and (b) CMR-12.5%.

3.4 Direct shear test

Figure 11, 12 and 13 presented displacement vs. shear stress under 200kPa vertical stress for unsaturated bentonite/CaCO₃ mixtures, shear strength vs. CMR for unsaturated and saturated samples and shear strength vs. vertical stress for unsaturated and saturated samples, respectively. As shown in Figure 11, the unsaturated mixed samples showed the behavior of strain softening under 200kPa vertical stress. In addition, the saturated samples and unsaturated samples under other vertical stresses showed the same strain softening behavior. The peak point of the shear stress-displacement curve was defined as the shear strength of mixed samples. As shown in Figure 12(a), the shear strength of unsaturated samples increased first and then decreased as the CMR increased, and the peak point occurred when the CMR was 3.125%. From Figure 12(b), it could be seen that the shear strength of saturated samples increased at first and then decreased under the low stress, but the shear

3.3 Microstructure

Figure 10 presented the images of bentonite/CaCO₃ mixtures which were obtained at a magnification of 20000 times under the scanning electron microscope. It could be clearly seen from Figure 10 that the surface structure of samples mixed with CaCO₃ crystals was denser because the presence of CaCO₃ crystals filled the pores of the clay sample to make the surface smoother and the structure denser. So, it resulted in the decrease of the hydraulic conductivity, which was consistent with the above macro-trait analysis.

strength decreased at first and then increased under the high stress, as consequences of the interaction of cohesion and internal friction angle.

According to the relationship between the shear strength and vertical stress (Figure 13), the parameters of mixed samples were shown in Table 3. For unsaturated samples, the two parameters firstly increased and then decreased with the increase of CMR. When the CMR was sufficiently small, the particles of the bentonite were bound tightly due to the cementation of CaCO₃ crystals, resulting in the increase of cohesion. Meanwhile, a small amount of the CaCO₃ crystals bonded with near the bentonite particles could increase the frictional resistance. However, with the increase of CaCO₃ content, the relative content of the bentonite particles decreased so that the cohesion decreased due to the reduction of total van der Waals force. The higher the content of CaCO₃ crystals, the smaller the friction and internal friction angle of mixed samples. For saturated mixed samples, the cohesion of mixed samples showed the trend of decreasing firstly and then increasing with the CMR.

Figure 11. Shear stress vs. displacement for unsaturated bentonite/CaCO₃ mixtures under vertical stress 200kPa.

Figure 12. Shear strength vs. CMR.

Figure 13. Shear strength vs. vertical stress.

As shown in Table 3, the cohesion between unsaturated and saturated samples was of significant difference because the cohesion of unsaturated samples was not only derived from Van der Waals forces, the gravitational or the repulsive force of the electric double layer, the cementation caused by the solute precipitation, etc., but also from the capillary effect caused by the unique surface tension of unsaturated clay.

Table 3. Parameter for the bentonite/CaCO₃ mixtures

Sample	CMR (%)	Cohesion (kPa)	Internal friction angle (°)
Unsaturated	0	752.9	25.50
	3.125	792.1	26.52
	6.25	760.7	26.12
	12.5	699.4	22.81
Saturated	0	143.8	14.07
	3.125	81.9	18.50
	6.25	87.6	18.76
	12.5	126.8	14.37

4 Conclusion

In this paper, some experiments were conducted to study the effects of different CaCO₃ contents on the swelling, compression, permeability and shear strength of the bentonite. The results were as follows:

The swelling properties of bentonite/CaCO₃ mixtures varied with the CMR. With the increase of the CaCO₃ content, the swelling strain and swelling pressure of bentonite/CaCO₃ mixtures decreased. While the vertical stress increased, the swelling strain of bentonite/CaCO₃ mixtures decreased.

While the content of CaCO₃ powders was less, the CaCO₃ powders had little effects on the compressive properties of bentonite/CaCO₃ mixtures. The existence of CaCO₃ crystals would lead to the change of the microstructure. The CaCO₃ crystals blocked the pores of bentonite to narrow the permeate channel of the liquid medium. While the content of CaCO₃ powders increased, the permeability of mixtures decreased.

While the increase of CMR, the cohesion of unsaturated samples first increased and then decreased, but saturated samples presented the opposite character. As for the internal friction angle of saturated and unsaturated samples, it first increased and then decreased.

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