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Soil-water characteristic of biochar-clay mixture in the full suction range

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Abstract: Soil-water characteristic curve (SWCC) plays an important role in defining the hydro-mechanical behavior of unsaturated soils. Biochar has the properties of porous structure, high specific surface area and high adsorption. The hydraulic characteristics of biochar-amended soil may change due to the influence of natural environmental factors when applied as the cover layer of landfills. In order to study the effect of biochar content on the water retention behavior of biochar-clay mixture in full suction range, the suction of samples was controlled by vapor equilibrium technique (suction range 3–368 MPa), filter paper method (suction range 0–40 MPa) and pressure plate method (suction range 0–1.5 MPa), and the water content and saturation degree of samples after suction equilibrium were determined. The soil-water characteristic curve of biochar-clay mixtures was obtained in the full suction range. The results showed that: (1) The soil-water characteristic curve in the full suction range of biochar-clay mixtures was effectively expressed by the three suction testing methods. (2) Biochar can affect the water retention behavior of clay, but within a certain range of suction, the water retention behavior of biochar-clay mixtures was effectively expressed by the three suction testing methods. (2) Biochar can affect the water retention behavior of clay, but within a certain range of suction, the water retention behavior of biochar-clay mixtures was also related to the pore structure and the morphology of water in the pores. (3) As measured by the pressure plate method, the air intake value of samples decreased as the biochar content increased. When the suction value was less than the air intake value, a horizontal section appeared in the curve, and the samples were always in the saturated state. The greater the content of biochar, the better the water retention of the sample. (4) The relationship between the water retention capacity of biochar-amended clay and biochar content was explained by the microscopic

Keywords: biochar; vapor equilibrium technique; pressure plate method; filter paper method; soil-water characteristic curve; microstructure

1 Introduction

Sanitary landfills are still the preferred method of municipal solid waste disposal in many countries^[1–2]. A final cover layer is usually installed on top of the landfill, whose function is mainly to retard the infiltration of external rainwater into the waste layer, thus generating leachate, and to control the escape of internal hazardous gases to the atmosphere. In traditional landfills, compacted clay is mostly used as the barrier layer of the final cover layer of landfills^[3–4]. Due to the seasonal alternation of climate, soil in the cover layer of landfills is in the dry and wet cycle all year round, or due to the influence of differential settlement caused by the waste degradation, the pore structure of the soil in the cover layer of landfills may change, further affecting the water retention of the soil.

In recent years, biochar has gained wide attention as a sustainable and environmentally friendly material for soil improvement in the cover layer of landfills^[9–10]. Biochar is a black insoluble solid substance rich in carbon obtained from biomass (e.g., peanut shell, wheat straw, wood chips and rice straw) by pyrolysis in an oxygen-limited environment^[11–13]. Biochar has a well-developed pore structure, high chemical stability, and high cation exchange capacity (CEC)^[14-15], and its porous structure provides a suitable space for the survival of methane-oxidizing bacteria and promotes microbial oxidation to reduce methane gas^[16–17]. However, after biochar is mixed into clay, the physical and mechanical properties of clay will be affected. The main manifestations are density reduction, porosity increase and pore distribution adjustment^[18-20], which in turn affect the water retention characteristics of clav^[21-22]. The addition of biochar can change the water retention capacity of soil^[23].

Currently, studies have shown the effects of biochar on the physicochemical properties and hydraulic characteristics of soil^[24–25]. And it is generally accepted

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that the addition of biochar will improve the water retention capacity of soil, and the degree of improvement is related to the biochar raw material, addition amount, pyrolysis temperature, pyrolysis time, and soil type^[26]. However, most studies on soil-water characteristic curves of biochar-amended soils have been considered only in a single suction range. For example, Or et al.^[27], Hardia et al.^[28], and Ojeda et al.^[29] studied the water retention capacity of different soils in the suction range from 0 to 1.5 MPa, and they found that the addition of biomass could improve the water retention of soil samples to varying degrees. Arthur et al.^[30] investigated the water retention characteristics of biochar-amended non-compacted sandy soil in the high suction range (10-480 MPa), and found that the addition of biochar could improve the water retention capacity of the soil. Most of the above studies are based on the soil-water characteristic curves of biochar-amended soils under a specific suction range, the content and particle size of biochar, and the biochar type. Therefore, it is necessary to conduct experimental studies on the soil-water characteristic curves of biochar-amended clay in the full suction range.

This paper was intended to study the soil-water characteristic curve of biochar-clay mixtures in the full suction range (0–1000 MPa). Suction tests were conducted on biochar-clay mixtures with different biochar dosages (0%, 5%, 10% and 15%) using three test methods: pressure plate, filter paper method and vapor equilibrium method. The soil-water characteristics of biochar-clay mixtures in the full suction range were studied, i.e., the relationship between suction and water content and the relationship between suction and degree of saturation were analyzed to derive the interrelationship among the water content, degree of saturation and suction. The research results can provide a reference basis for the design of biochar addition schemes for clay cover of landfills.

2 Test summaries

2.1 Test materials

The testing soil was soft silty clay, and the biochar used rice straw as biomass raw material and was obtained by high-temperature pyrolysis under an oxygen-limited condition. Fig. 1 shows the particle distribution curves of the clay and biochar used in this paper. By comparing with the classification plastic diagram of fine-grained soil, it could be concluded that the soil used in this test was low liquid limit clay.



Fig. 1 Grain-size distributions of the clay and biochar

The test method for the ash content of biochar was based on the ASTM D 1762-84 standard^[31] test method. During the test, 20 g of biochar was weighed in a crucible, and the temperature of the muffle furnace was set at 800 °C. After sintering for 4 h, the weight of the crucible was weighed to obtain the mass loss rate of biochar, which was the ash content. The basic physicochemical indexes of the testing clay and biochar are shown in Table 1.

 Table 1 Physical and chemical indexes of the clay and biochar

Clay								
Liquid limit /%	Plastic limit /%	Plasticity index	Maximum density /(g • cm ⁻³	lry Opti)	mum water content /%	Specific gravity	pH value	
35.98	22.20	13.78	1.65		22.50	1.67	7.7	
Biochar								
Density /(g • cm ⁻³)		Specific surface area $/(m^2 \cdot g)$		Specif gravit	ic y pH val	ue Ash	e Ash content /%	
0.55		385.60		1.99	10	10 18		

The damage to the skeleton structure of the original biomass was weak during high-temperature pyrolysis, and the morphological structure of the biochar could be well preserved. The biomass structure of the biochar is shown in Fig. 2. It could be seen that the micromorphology of rice straw biochar had a good porous structure.

2.2 Test method and sample preparation

2.2.1 Plate pressure method

In the pressure plate method test with a testing range from 0 to 1500 kPa, compacted samples with a diameter of 5 cm and a height of 2 cm were prepared, and the percentages of biochar in the mass of the mixture α were 0%, 5%, 10% and 15%, respectively. The prepared compacted cutting ring samples were saturated by pumping and put into the GCTS unsaturated consolidation apparatus with a preset suction value applied. The detailed experimental procedure can refer to in the literature^[32].



Fig. 2 Morphological characteristics of the biochar

2.2.2 Filter paper method

The filter paper method is an indirect method for measuring suction. The method is low-cost and straightforward to operate and can measure a wide range of suction values, including the total suction and the matrix suction. The biochar and clay were dried before the test. Compacted samples with a diameter of 5 cm and a height of 1 cm were prepared, including upper and lower samples. After the sample preparation, a testing filter papers were placed on the top of the container and between the upper and lower samples. After about two weeks, the filter papers and the water in the soil sample reached equilibrium. The filter papers were removed and quickly weighed on an electronic balance. Finally, the soil volume, water content and filter paper water content were measured and the corresponding soil-water characteristic curves were derived by combining the rating curves^[33]. 2.2.3 Vapor equilibrium method

The vapor equilibrium method was used for the dewetting test with controlling suction. Compacted samples with a diameter of 6.18 cm and a height of 2.00 cm were prepared in advance. In the dewetting test, the samples were first saturated by vacuumizing and then cut into pieces and placed on the separator in the dryer. During the test, the mass of the samples in the dryer should be measured regularly. When the mass change of the samples within 3 days was less than 0.01 g, the equilibrium state was considered to be reached. After equilibrium, the volume of the equilibrium samples was measured by Archimedes buoyancy method^[34–35], and then the void ratio and degree of saturation of the samples were calculated, from which the relationship

curves among the suction, water content and degree of saturation was derived.

2.2.4 Microstructure test

To investigate the variation of the pore size and pore content of biochar-amended clay with biochar content, microscopic pore structure tests were conducted using scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP). Prior to the SEM and MIP tests, the samples were required to be immersed in liquid nitrogen (-195 °C) for 5 min for freezing, and then placed in a Labconco freeze dryer for vacuumizing and cold drying^[36].

The pore size distribution of biochar-clay mixtures with different biochar contents was analyzed using a MacroMR12-110H-1 NMR instrument, and the samples were saturated by vacuumizing prior to the NMR test. In the NMR technique, T_2 describes how fast the transverse magnetization decays. The NMR test signal of the soil sample was converted into a T_2 time distribution curve of the pore water by the Fourier transform in the test data system. The area below the distribution curve represented the NMR signal in the corresponding T_2 range, i.e., water content, and then the T_2 value of pore water in the soil sample was directly related to the internal pore structure of the soil sample. Finally, the assumption of pore space in the soil sample yielded a transverse relaxation time T_2 proportional to the pore radius r. Thus, the T_2 value can be used to reflect the pore size of soil.

3 Test results and analysis

3.1 Pressure plate method test

Figure 3 shows the soil-water characteristic curves of the biochar-clay mixture using the pressure plate method.

As shown in Fig. 3(a), the water content of the samples gradually decreased with the increase of suction. Before conducting the pressure plate test, the samples were required to be saturated by vacuuming, so that the initial water content of the samples was saturated water content, and the degree of saturation was close to 100%. The saturated water content of the samples increased with the increase of biochar content, and the magnitude of the increase was more pronounced at 10% and 15% biochar contents. This was because biochar had the large specific surface area and porous structure, which could increase the porosity of the samples when added into the soil^[20]. Therefore, the greater the dosage of biochar was, the higher the saturated water content of the sample would be.

From Fig. 3(b), it could be seen that the suctiondegree of saturation curve of the samples showed a

trend of flattening followed by a sharp decrease as the suction increased. When the suction value was small and did not reach the air entry value of the sample, the soil was always in a saturated state. When the suction value was greater than the air entry value of the sample, some water was discharged from the soil and air entered into the soil pores, and the soil became unsaturated. In addition, when the suction value was greater than the air entry value, the curve decreased sharply with the further increase of the suction value. The slope of biochar-doped soil samples was smaller compared with the pure clay ($\alpha = 0\%$), and the curve was distributed below the clay, indicating that the water retention of the pure clay sample was better than the biochar-doped soil at this time. The reasons might be due to the fact that the water retention of clay particles was strong^[20], so the water discharge in pure clay pores was relatively slow with the increase of suction. In the soil mixed with biochar, the biochar particles filled the pores between soil particles, and the pore size of the sample was more uniform because the biochar was a porous structure and a water-repellent material^[20, 37], so the drainage rate of the sample was faster in the initial stage when the suction value was greater than the air entry value.



Fig. 3 Soil-water characteristic curves of biochar-clay mixtures by pressure plate method

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3.2 Filter paper method test

Figure 4 shows the soil-water characteristic curves of the biochar-clay mixture determined by the filter paper method.

It could be seen from the figure that with the increase of suction, both the water content and the degree of saturation gradually decreased. When the suction was small, the soil-water characteristic curves of the biochar-clay mixtures varied greatly with different biochar contents. The s-w and s- S_r curves of the mixtures tended to coincide as the suction increased. The curves of the biochar-doped soil samples were distributed below the pure clay, indicating that the water retention capacity of the biochar-doped soil samples became worse. With the increase of suction value, the curve distributions of different biochar contents were gradually close to each other, indicating that the effect of biochar content on the water retention characteristics of the mixed soil was gradually weakened.



Fig. 4 Soil-water characteristic curves of biochar-clay mixtures by filter paper method

As displayed in Fig. 4, the addition of biochar deteriorated the water retention of the mixed soil at the same suction value. The reasons might be related to the pore structure characteristics of the soil samples and the forms of water in the pores. The pure clay contained large pores between the clay agglomerates and small pores in the clay agglomerates. The water in the large pores included free water and bound water, and the water in the small pores primarily existed in the form of bound water, and the clay particles in the clay itself had strong water retention. Therefore, as the suction value increased, the free water in the bio-mixed soil was discharged first and the water retention became poor.

3.3 Vapor equilibrium method test

Figure 5 shows the suction versus water content and degree of saturation curves of the biochar-clay mixtures during the de-wetting process.



Fig. 5 Soil-water characteristic curves of biochar-clay mixtures by vapor equilibrium method

It can be observed from Fig. 5 that the water content and degree of saturation of the biochar-clay mixture with different biochar contents decreased with the increase of suction during the dewetting process, and the suction value of the biochar-clay mixture was close to 10⁶ kPa when the water content of the sample was 0%. This trend was consistent with the findings in the literature^[25, 38]. The soil-water characteristic curves of pure clay were distributed beyond the soil mixed with biochar. This was because the pores in the compacted clay were mostly dominated by clay agglomerates, the water in the pores mostly existed in the form of bound water, and the clay particles themselves had strong water retention, the pure clay samples had relatively good water retention performance at this suction value. With the further increase of the suction value, the s-wand $s-S_r$ relationship curves of the biochar-clay mixture with different biochar contents basically overlapped. It indicated that at the high suction stage, the content of biochar had almost no effect on the water retention characteristics of clay.

3.4 Soil-water characteristic curves in the full suction range

Figure 6 illustrates the soil-water characteristic curves of the biochar-clay mixtures with different biochar contents measured by three test methods in the full suction range. Fig. 6(a) shows the relationship curve between the water content and the suction. Fig. 6(b) shows the relationship curve between the degree of saturation and the suction.



Fig. 6 Soil-water characteristic curves of biochar-clay mixtures in the full suction range

The results in Fig. 6 indicated that the combination of the three methods could well measure the soil-water characteristic curve in the full suction range of biochar-clay mixtures. Vanapalli et al.^[39] divided the desaturation process of soil into three stages: i) the

boundary effect stage, ii) the transition stage, and iii) the unsaturated residual stage. From Fig. 6(a), it could be seen that at the low suction stage, before the suction value reached the air entry value of the sample, the sample was always in a saturated state, which corresponds to the boundary effect stage in the typical soil-water characteristic curve. When the suction value gradually increased and exceeded the air entry value, air entered the soil and water was discharged, the soil became unsaturated. At this time, the curve distribution was dispersed and the biochar affected the water retention of clay. In the full suction range, the water retention of pure clay was optimal, and after adding biochar, the water retention of mixture decreased. This was because the pores in the clay mostly appeared as pores between the clay agglomerates, and the water mainly existed in the form of bound water, which was not easily discharged. When the suction value was small, the curve distribution was dispersed; when it entered the high suction stage, the soil-water characteristic curves of the biochar-clay mixture basically overlapped with the increase of the suction value.

4 Microstructure of biochar-clay mixture

Figure 7 illustrates the T_2 distribution curves of saturated soil samples with different biochar contents. It could be seen from the figure that after adding the biochar to the clay, the T_2 distribution curves of the samples had a bimodal structure, and the second wave peak was distributed in the range from 10 ms to 100 ms. For the samples with 0%, 5%, 10% and 15% biochar contents, the first peak distribution range of T_2 distribution curve was 0.11-5.17, 0.13-7.80, 0.14-11.89 and 0.16–14.65 ms, respectively. The T_2 values at the corresponding peaks were 1.48, 1.70, 1.83, and 2.09 ms. The minimum value and peak value of T_2 increased with the increase of biochar content. For saturated samples, the magnitude of T_2 values was proportional to the pore radius of the sample, i.e., it was related to the pore size in the samples and could reflect the pore size of the samples using T_2 values^[40-41]. This indicated that the pore size of the samples was increased after adding the biochar. For small pores, the water in the pores was mostly in the form of bound water, and the suction and electrostatic interaction between the pore wall and water were strong. As a result, the pore water molecules in the pores were disorganized and non-uniform, and the water was difficult to be discharged during the dewetting process. While in large pores, water was mostly in free form, making the arrangement of water molecules in the large pore relatively uniform. Under the action of external force, pore water was relatively

https://rocksoilmech.researchcommons.org/journal/vol43/iss10/5 DOI: 10.16285/j.rsm.2021.7077 easier to be discharged. This could explain the test phenomenon in Fig. 6 that with the increase of suction value, the pore size of biochar-doped samples was larger, the free water content in large pores was high, and the drainage rate of samples was faster.



Fig. 7 T₂ distribution curves of biochar-clay mixtures with different biochar contents

Figure 8 shows the SEM images of the biochar-clay mixture with 0% and 10% biochar contents. From Fig. 8(a), it could be seen that when the biochar content was 0%, there were many large pores in the soil, and the pore type mostly belonged to the inter-clay agglomerate pore When the biochar content was 10%, as shown in Fig. 8(b), part of the pores between soil particles were filled by biochar particles, and the loose and porous nature of biochar increased the pore volume of the soil. This explained the intrinsic mechanism that the water retention capacity of biochar-amended clay varied with the biochar content in a certain suction range.

Figure 9 shows the MIP test results of biochar-clay mixtures with 0%, 5%, 10% and 15% biochar contents. Fig. 9(a) shows the cumulative mercury intrusion curves of the biochar-clay mixtures. Fig. 9(b) shows the pore size distribution density curves of the biochar-clay mixtures.

As could be seen in Fig. 9(a), compared to the pure clay ($\alpha = 0\%$), the maximum mercury intrusion e_{MIPmax} of the samples after adding biochar was greater than that of the pure clay. The curve for 10% biochar content was slightly higher than those of the soil samples with 5% and 15% biochar contents, indicating that the pore content of the biochar-amended clay increased with the increase of biochar content. It could be used to explain the macroscopic test results of the filter paper method and the vapor equilibrium method in Fig. 6, i.e., the water retention capacity of the samples with 10% biochar content was better than those of the 5% and 15% test groups.





(b) $\alpha = 10\%$

Fig. 8 Microstructure of different biochar contents



Fig. 9 MIP results of biochar-clay mixtures with different biochar contents

Figure 9(b) shows the pore size distribution (PSD) curves of the biochar-clay mixtures. It could be seen from the figure that the pore size distribution curves of the mixed soil had a single-peak structure with similar peak values. It indicated that the most probable pore size of the soil sample, i.e. the pore size of those pores with the maximum probability did not change significantly with the increase of biochar content.

By comparing with the soil-water characteristic curves of the biochar-clay mixture in the full suction range shown in Fig. 6, it was found that the soil-water characteristic curves of the samples almost overlapped at the high suction stage with the suction value greater than 10^4 kPa., and the biochar had almost no effect on the samples' water retention. From the MIP results of the samples in Fig. 9, it shown that at the high suction stage, the difference between the total pore content was small, the peak values of the pore size distribution curve were close, and the variation of the most probable pore size was not evident, indicating that the deformation of the samples tended to be stable.

5 Conclusion

In this paper, the soil-water characteristic curves of biochar-clay mixtures with different biochar dosages in the full suction range were measured by three suction testing methods, namely, the pressure plate method, filter paper method and vapor equilibrium method. The following conclusions can be drawn:

(1) The vapor equilibrium method, filter paper method and pressure plate method were used to measure the suction of biochar-clay mixtures in different suction ranges, and the combination of the three testing methods could well show the soil-water characteristic curves of the dehumidification process of the biochar-clay mixture in the full suction range.

(2) Biochar could affect the water retention capacity of biochar-clay mixtures, and the effect of biochar dosage on the water retention capacity of biochar-clay mixtures was not constant at different suction stages. When the suction was smaller than the air entry value of the soil sample, adding biochar could improve the water retention capacity of the mixed soil. At the initial stage, when the suction was smaller than the air entry value, the water retention capacity of pure clay samples was better than that of the soil doped with biochar. With the increase of suction, the soil-water characteristic curves of the samples with different biochar dosages almost overlapped when the high suction stage started, and the biochar had almost no effect on the water retention of clay.

(3) Different microstructure testing methods were used to obtain the pore content and pore size distribution characteristics of biochar-clay mixtures. The results suggested that the clay agglomerates were partly filled by biochar. The porous nature of biochar increased the pore volume of soil, thus affecting the water retention capacity of the mixed soil. The addition of biochar increased the content of pores with large sizes in the soil sample, and this part of pore water was easily discharged when the high suction stage started. The water retention capacity of biochar-clay mixtures did not increase with the increase of biochar content, indicating that there existed a biochar dosage that made the water retention of the mixture optimal. The results of this experiment elucidated the mechanism of the water retention characteristics of biochar-clay mixtures in the full suction range from a microscopic level.

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