Research Article

Microstructure of Compacted Loess and Its Influence on the Soil-Water Characteristic Curve

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Soil-water characteristic curve (SWCC) is a key constitutive relationship for studying unsaturated soil, and as is known, microstructure of the soil has great influence on the mechanical behaviour of the soil. In this study, the wetting and drying soil-water characteristic curves (SWCCs) of loess compacted at three different water contents were measured using the filter paper method. And microproperties of compacted loess were obtained by the mercury intrusion method (MIP) and scanning electron microscope (SEM). Results show that the compaction water contents have significant influence on the SWCC and microstructure. The pore size distribution (PSD) curves have great differences in macropore range and are similar in micropore range. Loess compacted at optimum and dry of optimum are generally connected, while there are certain number of nonintruded pores in loess compacted at wet of optimum. The SWCC curves vary significantly in low suction ($u_a - u_w < 1000$ kPa) and tend to converge together in high suction ($u_a - u_w \geq 1000$ kPa). Hysteresis in the SWCCs is more obvious for loess compacted at optimum and dry of optimum in the matric suction of 0∼100 kPa; however, there is a pronounced hysteretic for loess compacted at wet of optimum in full matric suction range. The characteristic of the SWCCs including their hysteresis can be well interpreted from the loess microstructure.

1. Introduction

In northwestern China, loess is widely distributed, and the compacted loess is always used to construct loess embankment, dam foundation et al. [1]. Because of the arid and semi-arid climate, deep groundwater, and other conditions, the loess is always unsaturated. For unsaturated soil, the engineering properties are much more complicated than saturated soil and poorly understood. This is primarily because the existence of the third phase, the gas phase. Soil-water characteristic curve (SWCC) is a crucial tool to understand behavior of unsaturated soil and is closely related to the mechanical properties of unsaturated soils, for example, the unsaturated hydraulic conductivity [2], unsaturated shear strength parameters [3–6], suction stress [7], the stress-strain relationship [8], and migration of pollutant in the soil [9].

The loess has a metastable and porous structure and is sensitive to water and force. Both evaporation and infiltration of water will cause changes in the matric suction in the loess, which will in turn cause changes in the mechanical properties of the loess. Thus, it is of significant importance to study the SWCC of the loess.

The soil-water characteristic is influenced by many factors [10, 11], such as mineral composition, soil structure, dry density, stress history, the drying, and wetting circulation. The microstructure is the most fundamental factor while other factors work mainly by changing the microstructure of the soil. In unsaturated soil, the water and air conversion occurs at the microscopic range [12]. In engineering practice, loess is always compacted, and because of the uncertainty of external conditions such as climate and rainfall, the compacted water content is not easy to be
controlled [13]. Previous studies have shown that fine-grained soils have different soil structure and further different soil-water characteristics and mechanical properties if they are compacted at different initial water contents. It is poorly understood how much influence the different initial compacted water contents will have on the soil structure and then the hydraulic and mechanic properties.

The aim of this paper is to investigate the microstructure, SWCC of loess compacted at different initial water contents and the influence of the microstructure on the soil-water characteristic. The MIP, SEM and filter paper methods were conducted on the compacted loess to provide essential data.

2. Soil Samples and Testing Producers

2.1. Testing Materials and Preparation of Compact Loess. The tested material is a loess taken from Yanguoxia Town, Yongjing County, which is located in the west of Lanzhou City, Gansu Province. The sampling depth is 20 m. The basic properties of the soil sample were tested in the laboratory. The particle size distribution is determined by the Better-size2000 laser particle analyser and is shown in Figure 1. It shows that there are two peaks in the particle size distribution curve of the tested loess. And the peak values are near 0.9 \( \mu m \) and 30 \( \mu m \), respectively. Some basic geotechnical properties are summarized in Table 1.

The soil was air-dried, pulverized by a wooden mallet, and then its mineral component was tested using X-ray diffraction method (Table 2). Results show that the nonclay mineral in the tested soil are mainly quartz, feldspar, and calcite, of which quartz is the most abundant, accounting for about half of the total minerals in the soil, and clay minerals are only illite and chlorite.

Compaction test was conducted according to the Unified Soil Classification System (USCS; ASTM 2011) [14]. The compacting curve (Figure 2) shows that the optimum water content of the tested soil is 17.0\%, and the corresponding maximum dry density is 1.71 g/cm\(^3\).

In the preparation of compacted loess, three different water contents were selected: dry of optimum (8\%), optimum (17\%), and wet of optimum (19\%), and their dry densities are consistent with the compaction curve. The loess was air-dried, pulverized by a wooden mallet, passed through a 2-mm sieve. A predetermined amount of distilled water was sprayed on the air-dried soil in several layers and left overnight in a platter sealed by fresh-keeping film in a cool room. The soil was then well-mixed with a soil cutter, placed in plastic bags, and kept in a moist chamber for at least 48 h for water moisture equilibration.

Cutting ring compacted loess for testing programs with 20 mm in height and 61.8 mm in diameter was prepared using a static sampler. The error of the dry density is controlled within 0.03 g/cm\(^3\). Basic physical properties of compacted loess are shown in Table 3.

2.2. Microstructural Investigation. The soil can be divided into four types according to its pore distribution characteristics [15], namely: (i) isobaric-pored soil, (ii) uniform-pored soil, (iii) single-pored soil, and (iv) double- or multi-pored soil. At present, the techniques for testing soil microscopic are mainly gas absorption method (BET method), mercury intrusion method (MIP method), microscopic observation and statistical method, X-ray tomography (photographic) method, and nuclear magnetic resonance method (NMR). In this paper, to have a better understanding of the microstructure of the compacted loess, the scanning electron microscopy and mercury intrusion porosimetry were used. The microstructural analysis of compacted specimens was performed using an MIP (AutoPore IV 9500) at Cold and Arid Regions Environmental and Engineering Research Institute, Chinese Academy of Sciences, and a SEM.
2.3. SWCC Test by Filter Paper Method. 
The filter paper method was conducted on compacted loess to obtain their drying and wetting soil water characteristic curves. The Whatman No. 42 filter paper is used to measure the matric suction of the loess sample, and the contact filter paper method is selected. The test procedures were mainly following the guidance of ASTM Standard D5298-03 [16] and summarized as the following:

2.3.1. Specimens Preparation before the Experiment. Each compacted loess with different initial water contents was divided into 2 groups. One was oven-dried at 105°C for at least 8 hours, and the dried cutting ring loess samples were weighed and recorded in pairs to carry out the wetting SWCC test; the other group was saturated with a vacuum saturator, weighed in a fresh-keeping bag, and placed in a moisturizer for the drying SWCC test.

2.3.2. Aluminum Box and Filter Paper Preparation. The aluminum boxes were washed with distilled water, oven-dried at 110°C for at least 8 hours, and then the aluminum boxes were allowed to cool to room temperature; soaked the protective filter papers and the test filter papers with 2% formalin (preventing them mildew and affecting the test results), placed them in the aluminum boxes, oven-dried at 110°C for more than 16 hours, and placed in a desiccator for use.

2.3.3. Wetting SWCC Test. The compacted loess was dripped to different estimated moisture contents, then wrapped in a fresh-keeping bag and placed in the moisturizer for more than three days. The test filter paper (Whatman No. 42 type) was sandwiched between two protective filter papers and placed between the two cutting ring soil samples that had been weighed. The protective filter papers should be larger than the test filter paper to ensure that the test filter paper does not touch the soil samples. Bonded the upper and lower soil samples with waterproof tape to fix the soil samples and filter papers together. The prepared soil samples are completely wrapped in a foil paper, try to keep them flat and then wrapped them with a layer of wax for seal. Finally, the sealed soil samples were labeled and placed in a thermo tank which was set 20°C for reducing the influence of evaporation and temperature. This procedure is shown in Figure 3.

Left the soil samples for 15 days to ensure thorough equilibrium. After the equilibrium time was reached, the soil samples and the test filter paper were carefully and quickly weighed within 5 seconds with a high-precision analytical balance (0.0001 g) to obtain their water contents. Finally, the suction was calculated using the suction calculation equation of the filter paper recommended by the ASTM.

2.3.4. Drying SWCC Test. The saturated soil samples were in pairs naturally air-dried to the estimated moisture contents, and the rest of procedures were the same as the wetting SWCC test.

3. Results and Interpretation

3.1. Microstructure Characteristics of Compacted Loess Specimens. The SEM and MIP are two complementary techniques to study the microstructure of the soil [12, 17, 18]. The SEM is a qualitative observation, and the MIP is a quantitative description of pores in the soil. These
two methods have been widely used by researchers to study the microstructure of intact or compacted loess [19–21] and other research field [22]. Figure 4 shows the MIP test results. Figure 4(a) shows the cumulative mercury intrusion curves of the specimens, and Figure 4(b) shows the pore-size density distribution. Figures 5–7 show SEM photographs of loess compacted at dry of optimum (8%), optimum (17%), and wet of optimum water contents. (a), (b), (c), and (d) in these Figures are photographs at different amplification factors.

Cumulative mercury intrusion curves (Figure 4(a)) show that pore distributions of the three compacted loess are quite different when the pore size is smaller than 10 μm. The total mercury intrusion volume of loess compacted at dry of optimum is 0.2382 ml/g, which is the highest in the three loess specimens. Then is the loess compacted at optimum (0.2286 ml/g), and the loess compacted at wet of optimum is the lowest (0.2024 ml/g). Theoretically, the tested total mercury intrusion volume should be consistent with the void ratio of the specimens. The void ratio order of the specimens is loess compacted at dry of optimum > loess compacted at wet of optimum > loess compacted at optimum. However, the total mercury intrusion of loess compacted at wet of optimum is less than the loess compacted at optimum. This phenomenon may be caused by inhomogeneity of the specimens and randomness of loess sample selected for mercury intrusion test. Or there may be many pores in the loess compacted at wet of optimum that cannot be intruded by the mercury.

The delimiting line in Figure 4(b) was utilized to separate micro pores and macropores. The derivative of PSD is zero at the delimiting line [23]. As presented in Figure 4(b), the delimiting line of the three compacted loess is at about 0.2 μm. Microstructure of the three loess is basically similar. There are no obvious dominating pores in the microscopic range of specimens compacted at optimum and wet of optimum, and the loess compacted at dry of optimum have a nonsignificant dominating pore in the microscopic range, the corresponding pore diameter is 0.095 μm. In macroscopic range, with the increase of the dry density, the dominating pores of the three compacted loess shift left, and the corresponding pore size density is reduced significantly. For the loess compacted at dry of optimum and wet of optimum, the dominating pore sizes are 3.6 μm and 1.3 μm,
respectively. The dominating pore size of soil compacted at optimum water content ranges from 1.8 μm to 6 μm.

The PSD curves of the specimens (Figure 4(b)) show that the microstructure of the three loess is basically similar, and there are big differences in the macrostructure. In other words, what different compaction conditions mainly change is the macroscopic PSD, and they have limited influence on the microscopic PSD. Tan et al. [24] proposed the concept of sensitive pores and inert pores. Under external loading, if the structural strength of a certain microstructure level is destroyed, it can be considered that the mechanical properties of the soil are controlled by the pores at this structural level, such pores are called sensitive pores. The pores of the other layer may maintain the original state and have no significant influence on the mechanical properties of the soil, namely inert pores. It can be seen that for the Heifangtai loess, the micropores belong to the inert pores, while the macropores belong to the sensitive pores. Therefore, intra-aggregate pores in loess are relatively stable, which is difficult to change under external force. Nevertheless, the stability of interaggregate pores is poor, and easy to change under external loading. Similar conclusion can be found in Romero et al. [25].

In general, the pore structure of compacted loess is more homogeneous (Figures 5–7). The particles are mainly surface to surface contact. There are generally no pores larger than 10 μm in the compacted specimens, and the interspaces between skeleton particles are mainly filled with clay and colloidal particles. For specimen compacted at dry of optimum (Figure 5), the specimen becomes denser, but the particle contour of the specimen is still distinct. The pore size of the soil is mostly around 5 μm, and the overhead pores with poor stability are less. In the SEM photograph with 1000 and 2000 magnifications (Figures 5(c) and 5(d)), the skeleton particles can be seen clearly, with a little clay and colloidal particles attached to them. These particles are in a relatively dispersed state. For specimens compacted at optimum and wet of optimum (Figures 6 and 7), the number of intra-aggregate pores increases, and soil density increases significantly. Soil particles are densely arranged, and it is difficult to see pores larger than 10 μm in diameter. The contour of skeleton particles is clearly even when the magnification reached 2000 (Figures 6(d) and 7(d)), because of the existence of clay and colloidal fractions on the particle surface and between the interspace. It can be seen that there are still very little overhead pores in the SEM photograph of
the specimen compacted at wet of optimum (Figure 7(a)), while there are almost no overhead pores observed in the specimens compacted at optimum. For specimen compacted at optimum, clay and colloidal fractions exist on the surface and fill in the interspace of the skeleton particles. The pores are in a relatively connected state. Clay and colloidal fractions in the specimen compacted at wet of optimum form pastes that attach to the particles surface and fill the interspace of the skeleton particles. These pastes are close to skeleton particles in size which may form pores with small access. In the previous description, we can see that tested total mercury intrusion volume of specimen compacted at optimum is higher than that of the specimen compacted at wet of optimum. This phenomenon can also be explained from the SEM of the two specimens: There are more pores with small access in the specimen compacted at wet of optimum. There are two kind of pores that cannot be intruded by the mercury, namely the nondetected pores and the nonintruded pores [23]. The former are the pores with a large entrance diameter and the nonintruded pores are the pores with a very minimal access. Owing to the limitation of minimum and maximum pressure of the apparatus, the two kinds of pores cannot be measured by the MIP device. In the SEM photographs (Figure 7), we cannot see pores with large entrance diameter, thus we can say there are more nonintruded pores in the specimen compacted at wet of optimum.

It is obvious that the initial compaction water content has significant influence on the structure of the compacted loess. Some aggregates formed in the process of soil-water mixed [12]. As supported by Delage et al. [12], at lower water content \( w \leq 8\% \), dry of optimum, with high suction, internal cohesion in aggregates is strong, so it is difficult to get a relatively high dry density. As the water content increases and is close to the plastic limit \( w = 17\% \), optimum, the original aggregates become deformable and easy to break down, and it is easy to achieve a dense state during compaction. When the water content is greater than the optimum water content, it is easy to form clay paste. The soil permeability then becomes bad, pore water pressure may partially become positive, the compaction effort is dispelled in plastic deformation of the clay paste. In addition, when compaction stress is released, the specimen will rebound significantly, thus the dry density will not reach the maximum.
3.2. Soil-Water Characteristic Curve. Matric suction-volumetric water content data points of compacted loess tested by the filter paper method were fitted by the Gardner model [26], Van Genuchten model [27], and Fredlund and Xing model [28], which are fitted well for loess. Selected results are presented in Figure 8. Results show that Fredlund and Xing equation is the best-fit, and the SWCCs fitted by Fredlund and Xing equation were used in the following description. It should be pointed out that SWCCs of loess compacted at dry of optimum were piecewise fitted because it consists of two consecutive inverse sigmoid functions.

3.2.1. Soil-Water Characteristic Curve of Loess Compacted at Different Water Contents. Wetting and drying soil-water characteristic curves are shown in Figures 9 and 10, respectively. According to the test results, the minimum matric suction obtained by the filter paper method is 3.5 kPa, and the maximum is 44 090 kPa. It is difficult for the filter paper method to obtain matric suction within 10 kPa. Therefore, the matric suction data at this stage are limited, which affects the

Figure 7: SEM photographs of loess compacted at wet of optimum under different magnification: (a), (b), (c), and (d) are under magnification of 200, 400, 1000, and 2000, respectively.

Figure 8: Typical result for the loess compacted at optimum water content (wetting SWCC).
character in this section, and the rate of water adsorption/dehydration of these two specimens is smaller compared to the specimen compacted at dry of optimum. The SWCCs have an intersection point at matric suction of about 40 kPa, and the corresponding volumetric water content is about 26.0%. Corresponding gravimetric water contents at the intersection point are respectively 16.6% (dry of optimum), 15.3% (optimum), and 15.6% (wet of optimum), which is close to the optimum water content (17.1%), determined by the compaction curve. The concept of critical water content/matric suction was proposed to describe this intersection [29]. It means that regardless of dry density of the compacted loess, there is always a state in which the water content and the matric suction are the same, and it is influenced by the particle size rather than the particle spacing. However, whether the statement is true remains to be investigated. (3) Matric suction in this stage is greater than 1000 kPa, the rate specimens adsorption/dehydration become slowly again. Soil-water characteristic curves are similar, and this trend of the wetting curve is more obvious. It indicates that the initial compaction water contents or the internal structure have no significant influence on the soil-water characteristic curve of the high suction stage. It is the adsorption that governs high suction value of the SWCC [25].

On the left side of the intersection point of the wetting curves, it can be found that the soil-water characteristic curve of loess compacted at optimum is higher than the other two soil samples. However, for the drying curves, the loess compacted at wet of optimum is at the top.

### 3.2.2. Hysteresis of Soil-Water Characteristic Curve

Because the SWCC test procedure is tedious and time-consuming, it is usually assumed to be a single-valued function for convenience of study. However, water content corresponding to a certain matric suction of drying and wetting curve is typically different. Generally, the drying curve is above the wetting curve. This is the hysteresis of the SWCC.

Drying and wetting SWCCs of the three compacted loess were plotted respectively in Figures 11, 12, and 13. According to the results of Figures 11 and 12, hysteresis of specimens compacted at dry of optimum and optimum is more obvious in matric suction between 0~100 kPa. However, when the matric suction is greater than 100 kPa, there is little difference between the drying and wetting SWCC. For specimen compacted at wet of optimum, the hysteresis is obvious in the full suction range (Figure 13). From the pore size distribution testing results, there are many nonintruded pores in the specimen compacted at wet of optimum, and the pore connectivity is poor. Therefore, it is difficult for water to enter the nonintruded pores during wetting. On the other hand, the water in the pores with poor connectivity is not easy to discharge during drying. It can be speculated that hysteresis in the specimen is not only caused by the ink-bottle effect, but also the existence of large amount nonintruded pores. This can also interpretate the result in Section 3.2.1 which the drying curve of specimen compacted at wet of optimum is above it of specimen compacted at...
optimum. The dry density of specimen compacted at wet of optimum and optimum is 1.67 and 1.71 g/cm$^3$, respectively, a little different from each other. But, there are more nonintruded pores in the specimen compacted at wet of optimum. Therefore, it is more difficult for the water to discharge during drying.

3.3. Effect of Microstructure on SWCCs. The microstructure of soil has significant influence on the shape and hysteresis of the SWCC [23, 30–32]. By using equation (1) [33], the pore diameter can be converted to equivalent matric suction, then the SWCC and pore size (converted to matric suction) density function can be plotted in one Figure (Figure 14) to better explain the effect of microstructure on SWCC. Considering the same logic in drying and wetting curve, the wetting SWCC is presented.

It can be seen from Figure 14 that the soil-water characteristic curves and the pore distribution curves of the three compacted samples have a good correspondence relation. When the pore size distribution density on the pore distribution curve increases, the slope of the corresponding section on the SWCC also increases. For loess compacted at dry of optimum (Figure 14(a)), it can be seen that (i) in the matric suction of 0–15 kPa, the soil sample is almost saturated, and the data obtained by the filter paper test at this stage are limited. (ii) The matric suction of 15–70 kPa is the section with the largest slope of the soil-water characteristic curve. The pore distribution curve in this section has macroscopic peak point, which is coincident with the inflection point on the SWCC. (iii) In the matric suction of 70–300 kPa, the soil-water characteristic curve becomes gentle again as the pore size density is low, and the delimiting line of macro and micropores points on the PSD exists in this stage. (iv) In the matric suction larger than 300 kPa, the slope of the soil-water characteristic curve increases compared with the previous part, the micropore peak point of the pore distribution curve exists in this section, and the corresponding inflection point can be seen clearly. Pore size distribution density of micropore peak point is significantly smaller than it of the macropeak point. Accordingly, the soil-water characteristic curve in this section is also gentler than the matric suction of 15–70 kPa.

Macropores in loess compacted at optimum and wet of optimum are little, the PSD is more uniform, and the soil-water characteristic curve is also gentler than loess compacted at dry of optimum. For loess compacted at optimum (Figure 14(b)), the pore size distribution density is the highest in the intermediate section (matric suction of 15–1000 kPa), in which the SWCC is the steepest. As shown
in Figure 14(c), the same law can be observed in the SWCC-PSD of loess compacted at wet of optimum.

From Section 3.2, it can be seen that the SWCCs of loess compacted at different water contents have definite similarities and differences. The PSD curve has great influence on the characteristics of the SWCC. Therefore, the differences between the three compacted SWCCs can be interpreted through their pore size distribution curves. The SWCCs and PSD curves of three compacted loess were plotted together in Figure 15, and divided into three main parts based on their characters. In the first section (0–15 kPa), data points measured by the filter paper method are limited. Macropores in this section is difficult to be detected because of the limitation of the minimum pressure of the mercury intrusion test [25]. But, it can be seen from the SEM photographs of the three specimens in Section 3.1, that the loess compacted at optimum has the least macropores, which also corresponds to the characteristics of the SWCCs in this section. The PSD curves of the three specimens in the third section (>1000 kPa) tend to converge. Correspondingly, the SWCCs have the same trend. The second section (15 kPa–1000 kPa) of Figure 15 is the section with the largest differences in the pore distribution curves. In this part, pore size density of the loess compacted at dry of optimum is obviously higher than that of optimum and wet of optimum. Therefore, the slope of the SWCC in the second part of dry of optimum is the steepest.

4. Conclusions

This paper aims to understand the microstructure and soil-water characteristic curve of loess compacted at three different initial water contents. A series of laboratory tests such
as the MIP, SEM, and filter paper method were conducted and the following conclusions can be drawn:

(1) The PSD curves of the three loess are basically similar in the micropores, and there are big differences in the macropores. For loess compacted at dry of optimum and optimum, pores are in a relatively connected state. There are a certain amount of nonintruded pores that cannot be detected by the MIP device.

(2) The drying and wetting soil-water characteristic curves of the three compacted samples vary from each other greatly in the low matric suction range ($\mu_a - \mu_w < 1000$ kPa) and tend to be converged while the matric suction is greater than 1000 kPa. Hysteresis is more obvious for loess compacted at optimum and dry of optimum in the matric suction of 0–100 kPa, however there is a pronounced hysteresis for loess compacted at wet of optimum in the full matric suction range.

(3) The characteristics of SWCCs of loess compacted at different water contents can be interpreted through their microstructure. The higher the pore size distribution density of the specimen, the stronger its water holding capacity, the smaller the dehydration or water absorption rate and the more gentle the corresponding SWCC. For loess compacted at optimum and dry of optimum, hysteresis is presumed to be caused by the ink-bottle effect. Hysteresis for loess compacted at wet of optimum is relatively obvious throughout the whole test range, and the reason should be that there are more nonintruded pores in this specimen.

**Conflicts of Interest**

The authors declare that they have no conflicts of interest regarding the publication of this paper.

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