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Pore-size changes and responses of kaolinite with different structures subject to consolidation and shearing

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ABSTRACT

In this paper, the responses of kaolinite samples with two different soil structures, i.e., unwashed and pH 7.8 samples, under 1-D consolidation, isotropic loading–unloading and triaxial shearing are examined. The focus is on the associated changes in the pore-size distribution. During isotropic consolidation, the unwashed sample, with an open, aggregated and flocculated structure, exhibits higher compressibility than the pH 7.8 sample, which has a densely packed aggregated structure. The associated deformation in both samples primarily arises from the compression of the large inter-aggregate pores, which are not controlled by the suppression of the electrical double layer, and the intra-aggregate pores remain almost unchanged during consolidation. Similar behavior is observed in clay samples with different structures during 1-D consolidation and triaxial shearing that only the inter-aggregate pores are affected and the intra-aggregate pores stay almost the same. All of these findings suggest that the mechanical responses of kaolinite clay depend on aggregate-to-aggregate interactions rather than on particle-to-particle interactions. The aggregates of kaolinite clay can be considered analogous to sand particles.

1. Introduction

The features of dual porosity in clay, i.e., intra- and inter-aggregate pores, and the associated influence on soil behavior have long been identified by an early study (e.g., Olsen, 1962). Subsequent efforts have been devoted to gain insights into this topic by means of different methodologies and measurement techniques. The salient findings can be briefly summarized as follows. During 1-D consolidation, deformation is primarily the result of the compression of inter-aggregate (or inter-cluster) pores (Delage and Lefebvre, 1984; Griffiths and Joshi, 1989; Wang and Xu, 2007; Kanayama et al., 2009) and the intra-aggregate pores remain almost unchanged even after secondary consolidation (Wang and Xu, 2007). In a triaxial test, a clay sample that is anisotropic with respect to particle orientation behaves isotropically in regard to macro-scale responses, such as the stress–strain behavior, suggesting that aggregate-to-aggregate (or cluster-to-cluster) orientation rather than particle-to-particle orientation within the aggregates controls the behavior (Anandarajah et al., 1996; Kuganenthira et al., 1996). At the critical state, clay samples with different fabric associations have similar values in the q–p' plane where q and p' are the deviatoric and mean effective stresses, respectively; however, they have different values in the e–lnp' plane where e is the void ratio (Wheeler and Sivakumar, 2000; Wang and Siu, 2006b). This suggests that although the macrofabric of the samples is sheared to the critical state, the relatively strong microfabric within the individual clay packets (or aggregate) may not reach the critical state, i.e., the initial fabric is not completely erased, causing different values in the e–lnp' plane (Wheeler and Sivakumar, 2000). In summary, these findings lead to a conclusion that aggregate-to-aggregate (or cluster-to-cluster) interactions rather than particle-to-particle interactions within the aggregates govern the mechanical responses of clay. Nevertheless, further experimental evidence is required for verification and this is the main objective of this study.

This paper begins with a review of the surface charges, fabric associations and associated pore-size distributions in kaolinite clay. Then, the features of inter- and intra-aggregate pores in kaolinite samples are described to facilitate the discussion of the experimental findings. In the experimental section, the deformation characteristics of kaolinite samples with known fabric associations under isotropic loading–unloading and 1-D consolidation are presented first. Then, the pore-size evolution of kaolinite samples subjected to isotropic loading–unloading, 1-D consolidation and drained triaxial shearing are examined. The investigation focuses on the individual changes in intra- and inter-aggregate pores,

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which aims to provide the experimental evidence to support the assertion that the mechanical response of the kaolinite clay is dominated by aggregate-to-aggregate interactions rather than by particle-to-particle interactions.

2. Testing materials and sample preparations

The Speswhite kaolin, purchased from Imerys Minerals Ltd., UK, was used in this study. It contains 47% SiO_2 and 38% Al_2O_3, and most of the particles have sizes smaller than 2 μm. The specific gravity and surface area [Brunauer–Emmett–Tell (BET)] of Speswhite kaolin are 2.6 and 14 m^2/g, respectively. Further information such as isoelectric point (IEP) of Speswhite kaolin will be discussed in Section 4.1. Samples with two different fabric associations, i.e., unwashed and pH 7.8, were prepared for the tests. To avoid the presence of other clay minerals in the testing samples to bias the experimental results and associated discussions as pointed out by Hammad et al. (2013), the commercial kaolinite instead of the natural clay was used in this study.

The clay samples were prepared from a slurry state, following the method described by Wang and Siu (2006a). In preparing the kaolinite slurry for the unwashed sample, the kaolinite powder was first mixed with deionized water. The amount of deionized water required was about two times the liquid limit (LL) of the clay sample (LL = 65% as measured in this study), i.e., ~1.3 kg of water was mixed with 1 kg of dry kaolinite powder. The pH value and conductivity of the unwashed kaolinite slurry were 5.5 and ~350 μS/cm, respectively. To prepare the kaolinite slurry for the pH 7.8 sample, the kaolinite powder was first washed thoroughly by deionized water to remove excess salts. The washed kaolinite slurry was then adjusted to the designated pore fluid conditions, i.e., pH 7.8 by adding sodium hydroxide (NaOH) solution (i.e., 0.5% by mass). The targeted pH value was verified using OAKION Benchtop pH/Conductivity/TDS 510 m (manufactured by Cole-Parmer Instrument Company, USA).

The prepared kaolinite slurry was then poured into a tailor-made consolidometer of 110 mm in diameter and 400 mm in height for slurry-consolidation using the stress of ~50 kPa. The consolidation process was considered completed when there was no apparent settlement observed. This slurry-consolidation stage lasted for ~10 days for the unwashed sample and about one month for the pH 7.8 sample. Following slurry-consolidation, the kaolinite sediment was carefully trimmed into various sample sizes for different tests.

3. Experimental details

Three different tests were conducted: 1-D consolidation, drained isotropic loading–unloading and drained triaxial shearing tests. The measured water contents before the tests (i.e., the initial state) and after the tests are summarized in Table 1. Pore-size measurements of selected samples of these three tests were done using Mercury Intrusion Porosimetry (MIP). The details of each experiment are described below.

3.1. Oedometer tests

The oedometer tests were carried out on both unwashed and pH 7.8 samples according to ASTM D2435, with deionized water used as the surrounding fluid in the test. The size of the kaolinite sample was 70 mm in diameter and 20 mm in height. The loading stages were 12.5 kPa, 25 kPa, 50 kPa and 100 kPa. Each loading stage lasted for at least 24 h to ensure that the clay samples had reached the end of the primary consolidation, which was also checked from the deformation–log(time) curve.

3.2. Tests using the triaxial device

The CKC triaxial system was used to perform the isotropic loading–unloading and drained triaxial compression tests; the details of this system can be found in Li et al. (1988). The testing sample had the size of 50 mm in diameter and 100 mm in height. To minimize the effect of end friction, lubricated end platens were used. A back pressure of about 200–250 kPa was applied to increase the degree of saturation in the clay samples up to a B-value of ~0.98.

Table 1
Comparisons of the total intrusion volume per gram in the MIP tests and the measured water content.

Tests and samples	Total intrusion volume in the MIP tests (ml/g)	Measured water content (%)
Isotropic consolidation tests (unwashed sample)		
Initial state	0.640	64.1
After consolidation by 100 kPa	0.515	51.6
After consolidation by 400 kPa	0.462	47.3
Isotropic consolidation tests (pH 7.8 sample)		
Initial state	0.431	40.6
After consolidation by 400 kPa	0.365	34.0
1-D consolidation tests (unwashed sample)		
Initial state	0.600	63.3
After consolidation by 100 kPa	0.550	55.7
Drained triaxial compression tests (unwashed sample after the test)		
NC sample — upper	0.418	
NC sample — middle	0.427	43.5
NC sample — lower	0.433	
OC sample — upper	0.425	
OC sample — middle	0.409	41.5
OC sample — lower	0.400	

The isotropic loading–unloading tests were performed on both unwashed and pH 7.8 samples. During the loading stage, a confining pressure from 20 kPa to 400 kPa was applied in a constant rate of 3 kPa/h. This loading rate was slow enough to ensure the complete dissipation of excess pore water pressure at each loading stage. The same rate was also applied during the unloading stage.

The drained triaxial compression tests were performed only on the unwashed samples with overconsolidation ratio (OCR) of 1 and 4. For normally consolidated (NC) sample (OCR = 1), the sample was subjected to a confining pressure of 100 kPa before shearing. For overconsolidated (OC) sample (OCR = 4), the sample was isotropically consolidated to 400 kPa and then unloaded to 100 kPa. The confining pressure used was 100 kPa, and the adopted loading rate was 0.005% of the axial strain per minute (during the shearing stage), which was determined using the method suggested by Head (1994). All the tests were terminated at a large axial strain of 35% to enhance the shearing influence on the pore-size change.

3.3. Measurement of pore-size distributions

At the end of the previously described tests, selected samples were fully unloaded and taken out from the testing device. Small cubes of ~10 mm × 10 mm × 5 mm (length × width × height), used for the MIP tests to investigate the pore-size changes, were carefully cut from these selected samples using a wire saw. Note that the specimens in the drained triaxial compression tests were divided into three different sections, i.e., upper, middle and lower sections for the MIP analyses.

Before the MIP analyses, water in the test samples has to be completely removed. To minimize the fabric alterations resulting from the capillary effect during the drying process, the freeze-drying method was adopted. The small-cube samples were first submerged into liquid nitrogen for quick freezing of the pore water immediately after cutting. Then, these frozen cubes were put into a freeze-dryer (Edwards Super Modulyo, Edwards Limited, UK), operating at the temperature of ~− 50 °C for more than 48 h. This process allowed the water to evaporate by sublimation; the capillary effect was minimized therefore the fabric was preserved. In addition to the freeze-drying method, some of