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Transformation of pore structure in consolidated silty clay: New insights from quantitative pore profile analysis

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HIGHLIGHTS

- Sharp BSE images of pores in silty clay with high resolution and low imaging noise.
- 2D pore profiles extracted from the sharp BSE images to characterize pores.
- Transformation of the 2D pore profiles linked with the consolidation process.
- The Washburn's equation was corrected based on circularity of pores.

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ABSTRACT

Pore structure provides essential information for studying the behaviors and properties of silty clay in construction projects. This study uses a developed metal intrusion characterization scheme to investigate the transformation of the pore structure of silty clay in consolidation. Clear pore profile images with nanometer-level resolution are produced by BSE imaging. Comparison between metal intrusion and epoxy impregnation suggests minimal alteration of the pore structure of silty clay with the metal intrusion technique. The pore size redistribution and the transformation of porosity indicate that the pores collapse and form during consolidation process. Solidity is found to decrease as consolidation pressure increases, reflecting the consolidation-induced pore deformation. Aspect ratio is found to be independent of the consolidation pressure, indicating that the pores are likely to shrink evenly in consolidation. Two descriptors, box dimension and probability entropy, are found to decrease as consolidation pressure increases, indicating that the overall pore structure becomes homogenized in consolidation. Washburn's equation is modified based on the area–perimeter relation of pores to provide a more accurate reflection of the pore size measurement by mercury intrusion porosimetry. The results show clear evidence for a distinctive two stages transformation processes during consolidation namely radial compaction and pore segregation.

1. Introduction

Silty clay is widely encountered in the construction of foundations, excavations, and river tunnels, especially in the middle and lower reaches of river basins and in coastal areas. Due to the high sensitivity [1], high compressibility, and low strength [2] of silty clay, construction in a silty clay environment poses problems of consolidation settlement, and stability. The consolidation of silty clay involves transformation of the microstructure, pore pressure, and pore medium, all of which are linked to the pore structure of the silty clay. Better understanding of the changes in pore structure can improve the prediction of consolidation settlement based on poromechanics [3,4].

The pore structure provides essential information for studying the properties and behavior of silty clay, and its correlation with transport, strength, and rheology has been the scope of a large number of investigations in soil mechanics and pedology. For example, Adamids et al. [51] mentioned the term reconsolidation in soil analysis, which is usually used to represent the process of previously-consolidated soil being consolidated again through changed pressure. Zeng et al. [5] investigated the critical yield stress of soil pores by increasing the consolidation pressure. Their

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result indicated that the collapse of pores in the reconsolidation process was caused by the consolidation confining pressure being lower than the critical yield stress. An experimental study by Sridharan et al. demonstrated the relation between the shear strength, pore structure, and pore medium of clay [6]. Griffiths [7,8] studied the pore size distribution due to consolidation and secondary consolidation of clay, and found the pore size redistribution of clay in consolidation. In a pore scale study of soil, Ghassemi and Pak [9] established the relationship between permeability and tortuosity with porosity and other microstructure parameters based on the Lattice Boltzmann method. Sridharan et al. [10] pointed out the relation between larger inter-aggregate pores and the deformation behavior of clay. Understanding of the pore structure of silty clay has particular relevance to geotechnical applications in which silty clay undergoes changes in water content and volume.

Several methods have been commonly used to study the pore structure of silty clay. In the mercury intrusion porosimetry (MIP) technique, an absolute pressure is applied to non-wetting mercury so that it enters interconnected pores [11], from which pore information such as pore size distribution can be deduced [12]. Another technique, based on physical adsorption of gas molecules on the surface of soil pores, such as nitrogen adsorption, enables the evaluation of mesoporosity through interpretation of the capillary condensation processes [13,14]. CT scanning enables direct measurement of the 3D macroporosity of soil through computer-processed combinations of X-ray tomographs [15,16]. Finally, image analysis can quantify the pores in silty clay through direct visualization of the pore structure by microscopy [17] and presents the possibility of quantification of the pore structure. Image analysis is a preferable method because it is one of the most direct ways to analyze the pore structure of soil [18], whereas MIP and gas adsorption acquire pore information indirectly via certain mathematical models, and the resolution of CT scanning is degraded when the size of the feature is less than several microns.

Existing image analysis based studies of the pore structure of silty clay have focused mainly on discerning pore structure transformation and pore size characterization. Only a few have quantitatively studied the shape and homogeneity of pores. Using environmental SEM, Villar et al. observed the evolution of intergrain porosity during dehydration of clay/sand mixtures [19]. Fiès et al. studied epoxy impregnated silty clay samples and derived the lacunar pore volume [20]. Dathe et al. used images of thin section epoxy impregnated samples to calculate the surface fractal dimension of pores [21]. Singh et al. used images of epoxy impregnated soil to characterize macroporosity and pore size distribution [22]. Due to the low contrast between soil and polymer impregnated pore, limited information can be acquired from blurred images of the observed pore profiles, and blurred boundaries induce errors in interpretation. On the other hand, the limited resolution of images of polymer impregnation makes it impossible to separate and quantitatively investigate the micropores embedded in the clay fabric.

It is essential to improve the current image-based techniques to produce a clear view of soil pores for further quantitative image analysis of the pore structure information. However, usage of metal intrusion to replace conventional polymer for pore imaging is limited in the literature due to the highly toxic element in the metal such as mercury, Pb and Cd. The co-authors of this paper have developed the first characterization scheme using non-toxic metal and low voltage small interaction volume backscatter electron imaging to obtain high-quality images in cementitious materials in our previous work [23,24]. To the best of our knowledge, this is the first study using the presented metal intrusion characterization scheme technique to quantify and analyze the pores of silty clay, contributing to the availability of clear images with high-resolution and further possibility of quantitative analysis. This method employs a customized piston cylinder apparatus to effect the intrusion of low-melting-point Field's metal into interconnected pores of silty clay. Compared with conventional intrusion materials such as epoxy resin, Field's metal enhances the contrast and clarity of silty clay pores, by virtue of the significant difference between the chemical composition of the metal and the soil matrix. High-resolution images of soil pore structure are obtained by the combination of metal intrusion and Backscattered Electron imaging.

The new technique enables quantitative analysis of pore profiles, allowing comprehensive investigation of the pore structure of silty clay. In this study, the metal intrusion based characterization scheme is shown to cause minimal alteration to the pore integrity of silty clay, through the comparison to epoxy impregnation, which has been proved to preserve the microstructural integrity of soil [33]. Enhancement of contrast and reduction of imaging noise by metal intrusion are verified. As shown in Fig. 1, images from epoxy impregnation method have limited resolution and blurred boundary, the required BSE images for quantitative analyses are obtained from metal intrusion based method only. Based on high-resolution BSE images, pore volume fraction and pore size distribution are used to describe the transformation of pores in consolidation. Pore shape descriptors are adopted to interpret the pore shape transformation during the consolidation process. Two indicators, namely box- dimension and probability entropy, are used to describe the influence of consolidation on the overall homogeneity of the pore structure. The circularity–pore diameter relation is used to correlate the Washburn's equation and to provide more accurate estimation of the results of MIP. The pore structure information analysis scheme used in this study is beneficial to further study of the consolidation of silty clay and other geomechanical materials.

2. Experimental program

2.1. Materials and instrumentation

The soil investigated in this study is undisturbed silty clay from Wuhan, China. Drilling thin-walled samplers were used to obtain the undisturbed silty clay samples. The samples were packed in airtight containers to avoid evaporation loss after sampling. The properties of the undisturbed silty clay investigated this study are listed in Table 1. The Field's metal was purchased from Rotometals, Inc. The properties of the metal are listed in Table 2. The epoxy resin was purchased from Agar Scientific, Inc.

A Nova 450 SEM was used to conduct BSE imaging of the samples. A custom-designed piston cylinder apparatus [27] was used for the metal intrusion process. A Shimazu AG-X test machine was used for pressurizing. The principle of the instrument is pressurizing the molten low-melting-point metal in the pressure vessel by compression. An AutoPore IV 9505 mercury porosimeter was used to conduct MIP testing. The porosimeter could achieve a maximum pressure of 182 MPa and the corresponding minimum accessible pore diameter was about 8 nm.

2.2. Sample preparation and experimental procedure

In accordance with soil test method standards GB_T50123-1999 [28], uniaxial consolidated silty clay samples with consolidation pressures of 0 kPa (C-0), 600 kPa (C-600), and 3200 kPa (C-3200) were prepared. The samples were then dried in ventilated ovens at 105 ± 1 °C for 24 h. MIP tests were performed for C-0 and C-600 samples using an AutoPore IV 9505 mercury porosimeter.

After the drying process, cubic samples approximately 5 mm × 5 mm × 5 mm in size were taken from the cores of undisturbed