Porosity and Pore-Size Distribution of Geomaterials from X-ray CT Scans

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Abstract. Determination of transport properties of geomaterials is an important issue in many fields of engineering analysis and design. For example, in petroleum engineering, permeability of an oil reservoir may be crucial in establishing its viability for exploitation whilst prevention of leakage from underground storage facilities for oils and gas, nuclear waste as well as CO2 crucially depends on its long term values. Permeability is an illusive parameter which is difficult to obtain not only in field situations but also in controlled laboratory environment. Its determination is further complicated by the fact that values are needed for low permeability porous media such as clays/rocks at various degrees of saturation and at elevated temperatures which makes physical experiments not only expensive but also difficult and time-consuming. Permeability is indirectly related to the porosity, pore-size distribution and pore-architecture. A well-known way to obtain this information is through mercury porosimetery but this procedure has safety issues associated with it. Moreover, it is not an easy experiment to conduct. In this paper, we demonstrate the use of micro X-ray CT scanning technique to obtain porosity and its variation in clays. Since the resolution of micro CT equipment is not high enough to be able to observe specific pores in clays, an experimental programme to correlate porosity with data from scanning was undertaken. It consisted of consolidating specimens made from a mixture of kaolinite and bentonite in an oedometer, unloading them and obtaining 32mm samples from various locations, scanning them as well as determining void ratio of these specimens using standard laboratory procedures. It is observed that the Average CT Number (ACTN) for the specimens correlated well with the porosity (void ratio) whilst spatial variation of CT numbers seems to indicate the capability of scanners to capture pore size distribution. This indicates the possibility of computing permeability of low porosity media through CT imaging.

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1 Introduction

In recent years, geotechnical engineering has benefited from advances in various high-technology fields such as computing, instrumentation, electronics and information systems. However, investigation of hydro-mechanical characteristics of the geomaterials has still many limitations. In particular, a reliable estimation of permeability is a matter of the utmost importance in many fields where reliable barriers are required to prevent flow of radioactive contaminants, oil and gas from underground storages.

In this respect, engineers dealing with very fine soil materials like clay have been searching for more advanced techniques for determining porosity, pore-size distribution and permeability[1] under various field conditions. Visualizing and quantifying micro-scale internal structures and physical composition of a material in an undisturbed state is now feasible using 3-dimensional images by X-ray CT (Computed Tomography) methodology.

The advantage of X-ray CT is that it helps engineers to obtain reliable information of the pore and grain structure rapidly in clean and less hazardous environments. They also have a further advantage in that the same specimen can be scanned several times in a variety of mechanical and environmental conditions and interaction between different phases such as solid, water and air can be investigated at any location within the specimen.

In this paper, research on the measurement of porosity[2] (or void ratio), one of the important geotechnical design parameters for fine grained soils, utilizing the micro CT X-ray scanning technique, is presented. In engineering practice indirect methods are used to obtain porosity and permeability. Unlike the case of sandy soils, these parameters are crucial for clays in geotechnical design since they play a key role in deciding consolidation settlement and time (Fox and Berles, 1997). They are also prone to large measurement errors when conventional methods are used. For example the discrepancy between measured and in-situ values of permeability can be of several orders of magnitude. Micro X-ray CT technology seems to be a step forward towards solving this problem.

In the past, Micro X-ray CT research tool has been adopted by Peyton et al. (1992) who tried imaging minute pores of undisturbed sand specimens in the early stages and Zeng et al. (1996) measured the density of specimens based on precision image processing analysis of CT data. Hereafter, the research on the changes of pore-structure was conducted by Wong (2000), Alshibli et al. (2003), and Riyadh et al. (2006). However, all studies have hitherto been restricted to sandy soils. In this paper, we explore if CT technology can be applied to fine cohesive soils whose particles are much smaller than the minimum pixel size of micro X-ray CT. Here we study porosity and pore size characteristics of

[1] This generic term is used to denote inter related terms such as 'absolute' and 'relative' permeability as well.

[2] In this paper, the terms 'porosity' and 'void ratio' have been used inter changeably due to their popularity in petroleum and geotechnical engineering fields respectively.

laboratory prepared artificial clay specimens after subjecting them to various consolidation pressures. Section 2 gives an overview of CT scanning methodology and the facilities available at the Korean Institute of Construction Technology (KICT). Section 3 describes procedure adopted for preparation and testing of clay samples using standard laboratory methods whilst section 4 describes techniques used for calibration and standardization of imaging data. Scanning results are presented and discussed in Section 5 and finally conclusions and recommended guide lines for future research are given in Section 6.

2 Overview of Micro X-ray CT

Devised and commercialized by Hounsfield in 1971, CT (Computed Tomography) is the technology which embodies the analysis of three-dimensional images obtained by seeing through objects using X-rays from various angles, to obtain sectional diagrams of various objects. X-ray CT techniques have been commonly used in the past in the fields of medicine and manufacturing for the detection of fractures and defects in a variety of manufactured consumer goods. In recent years, it is being increasing used in the fields of biotechnology, forensic archaeology and other related areas. Its utilisation in the study of mechanics of geomaterials is, however, recent but growing.

A CT scanner consists of three components viz. a source to generate X-rays, a detector to measure their penetration in the object being scanned and a manipulator to locate and rotate the object through 360°. Fig. 1 shows the equipment used in this study at KICT .

The micro X-ray source in the CT scanner is of the ‘sealed-type’ with radiation power of up to 90kV. Its resolution is 250μA, and the minimum focal spot size is 5 microns (0.005 mm). The scanning chamber can accommodate objects of size up to 300 mm in diameter and 500 mm high.

In general, the factor which governs the capacity of X-ray equipment is the source, but the factor which constraines the use of CT scanner methodology is its resolution. The detector captures the attenuated X-rays passing through the scanned object. It is made by Rad-icon, is 50 mm × 50 mm in size and has a pixel pitch of 48μm, and its limiting resolution is 10Lp/mm (line pair per millimeter).

The manipulator used in the CT scanner at the KICT is of ‘low noise’ type and is used for locating and holding specimen tight for minimizing ‘trembling’ which invariably takes place during rotation of the object. Moreover, there is a discrepancy due to rotating axis not being absolutely vertical which leads to ‘noise’ in CT data. This noise in the KICT scanner used for this study is less than 0.01mm, which, however, has been corrected by a ‘reconstruction’ software.

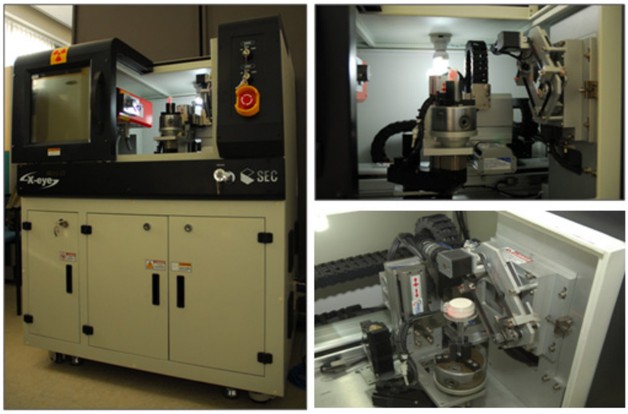

Fig. 1 Micro X-ray CT equipment of KICT (a) view of micro CT scanner (left), (b) arrangement of three major parts (source, manipulator and detector from right hand side, respectively in upper right figure), (c) zoomed manipulator & detector (lower right)

3 Experimental Programme on Artificial Clay

The aim here is to obtain specimens of clay of controlled porosity for scanning and correlating image analysis data with the experiments. With this in view, it was decided to make two sets of samples: one from kaolinite (100 %) and the other from a mixture of kaolinite (90%) and bentonite (10%). It is well known that kaolinite has low volume ion exchange capacity, whilst bentonite has high positive ion exchange capacity. It was therefore expected that pore characteristics of the two sets may be different. In this study, moisture content was set to twice the 'liquid limit' for preparing the clay specimens, A solidifying agent was also added in each case and the resulting slurry was sieved through a No 10 sieve to remove any foreign matter. About 20 specimens of two different compositions were then consolidated in K_0 conditions to a vertical pressure ranging from 150 kPa to 400 kPa. After unloading and removing the samples from the mould, they were horizontally cut into three parts identified as 'upper', 'middle' and 'lower' part. Three cylindrical samples, 32 mm in diameter and 20 mm thick were carefully scooped from each of these parts for scanning using an acrylic sampling ring (see Fig. 2).

Fig. 2 Sampling process: (a) cutting (b) sampling (c) ready for CT scanning

A number of samples got disturbed and these were not scanned. Porosity was subsequently determined for these specimens using conventional methods. The results are presented in Table 1 together with results of CT scanning, which are discussed below.

Table 1 Results of void ratio measurement and the modified CT numbers for certain areas of each specimen (see Fig. 5 for definition of the area)

Vol. fraction (Kaolinite : Bentonite)	Pre-consolidation pressure	Cutting location	Void ratio (e)	Modified CT number (MCT) (-CT no*10)				Average MCT
				Area 1	Area 2	Area 3	Area 4	
9:1	400kPa	Upper Part	1.46	1752	1735	1722	1741	1741
			1.46	1635	1670	1660	1654	1654
			1.46	1584	1584	1621	1655	1611
	350kPa	Upper Part	1.47	1692	1757	1625	1697	1693
			1.47	1653	1720	1668	1635	1669
			1.46	1640	1589	1612	1607	1612
		Middle part	1.63	1960	1947	1898	1951	1939
			1.58	1850	1798	1718	1866	1808
	200kPa	Upper Part	1.59	1882	1911	1783	1911	1872
			1.56	1706	1778	1739	1771	1749
			1.57	1799	1818	1753	1850	1805
		Middle part	1.57	1842	1733	1733	1691	1750
			1.59	1923	1842	1873	1831	1867
			1.48	1692	1724	1682	1696	1699
10:0	350kPa	Upper Part	1.56	1811	1813	1911	1896	1858
			1.55	1849	1794	1778	1846	1817
			1.57	1724	1691	1735	1725	1729
	300kPa	Upper Part	1.69	2208	2095	2106	2170	2145
			1.64	2174	2135	2104	2109	2131
			1.68	2157	2110	2123	2144	2134
		Middle part	1.69	2203	2145	2148	2192	2172
			1.65	2103	2151	2277	2043	2144
	250kPa	Upper Part	1.79	2306	2298	2280	2232	2279
			1.88	2468	2389	2481	2515	2463
			1.82	2258	2393	2334	2305	2323
		Middle part	1.82	2309	2293	2244	2272	2280
			1.82	2258	2226	2179	2185	2212
			1.81	2121	2148	2177	2132	2145
	150kPa	Upper Part	1.61	1924	1895	1850	2001	1918
			1.61	1884	1903	1852	1976	1904
			1.61	1807	1973	1889	1993	1916

4 CT Number: Standardization and Calibration

4.1 Grayscale and CT Number

X-ray CT scan results display grayscale images signifying the intensity of attenuated X-rays represented as contrast ratio ranging from completely white to absolutely black. The corresponding numerical range is $0 \sim 2^{15}$ in 16 bit system. However, the values are dependent on experimental parameters (X-ray machine, distance to the specimen etc.) as well as environment conditions such as room temperature, humidity etc.. In this respect a correction to obtain standard consistent values is required. Unlike the field of medical sciences where a qualitative comparative image is adequate, for engineering analysis the grayscales are converted into a 'CT number' as follows:

$$I = I_0 e^{-\mu x} \quad (1)$$

where I_o is the intensity of the un-attenuated X-ray beam, I is the beam's intensity after it traverses a thickness of x in the material and μ is the linear 'attenuation coefficient' which has a unique value for any material depending on the power of X ray beam and atomic number of constituents of the material being scanned. For improvements in quality of images, some corrections known as 'gain' (normalization) and 'offset' (subtraction) are made as follows.

$$\mu_{corr} = A[\frac{\mu_{org} - \mu_{air}}{\mu_{ref} - \mu_{air}}] - B\mu_{dark} \quad (2)$$

where μ_{mat} , μ_{air} and μ_{ref} are linear attenuation coefficients of the scanned material, air and a very homogeneous reference material, respectively. μ_{dark} is of a dark image taken without X-ray. A and B are constants called 'gain' and 'offset' coefficient, respectively. The reference material chosen in this study is 'water' as it is one of the constituents of our clay samples. Gain and Offset do not affect the final results, but are used to produce a contrast in the images. Then standard CT numbers are determined by:

$$CT = K\left(\frac{\mu_{corr} - \mu_{water}}{\mu_{water}}\right) \quad (3)$$

Where μ_{water} is linear attenuation coefficient of water. K is a constant adopted as '1000' as a standard. Therefore, CT number of water and air always become '0' and '-1000' respectively as μ_{air} =0 in standard ideal conditions. CT numbers for other materials fall between -1000 and 1000 and reflect CT values of constituents like soil, water and air content in them. However, these CT Numbers have to be determined on the particular equipment being used as well as for the environmental conditions prevailing at the time of scanning as described in Section 4.2 below.

4.2 Gain and Offset Correction for CT Number Standardization

The standardization of CT number is an essential task for quantitative evaluation of porosity and pore characterization from CT images. In this study, the apparatus for the ‘gain’ and ‘offset’ correction assembled at the KICT is shown in Fig. 3 and used to obtain CT values independent of X-ray CT scanning equipment and environmental factors. It is a generic setup and is planned to be used for CT scanning studies of other materials such as stones, rocks, ceramics and machine components and manufactured through powder technology.

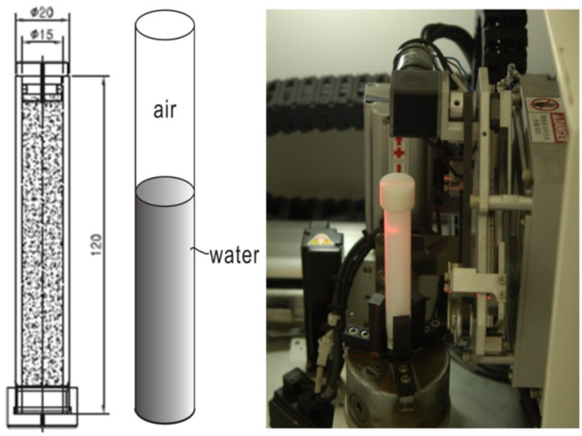

Fig. 3 The apparatus for Gain-Offset correction

After a number of studies, the values of Gain and Offset were selected as 0.2 and 0.1, respectively and were applied to raw data to obtain CT values for all clay specimens and are given in Table 1 above.

5 Porosity and Pore Size Distribution

In general, platelets of clay are smaller than 5 μm in the Unified Soil Classification System. It would, therefore, appear that micro X-ray CT equipment with a resolution of 25 μm would be inadequate to physically view the pores and connecting channels. It is indeed true but our aim here is to assess if the variations in porosity can be picked by the CT scanner as this would lead to a rapid tool to obtain porosity and pore-size distribution which in turn can be adopted to compute permeability. For this reason, even though a number of platelets as well as connecting channels will co-exist in one pixel as shown in Fig. 4, CT numbers will reflect this situation. In general, as the number of platelets (solids) increases in a pixel, CT number should change at the same time (Richard et al., 2001). In the following we will deal with the issue of determination of porosity first, followed by an attempt to identify if the CT scans are capable of detecting possible non-uniformity within the samples.

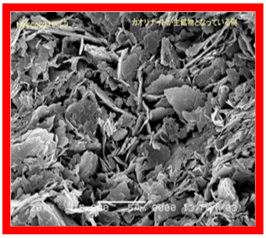

Fig. 4 Clay platelets within a pixel

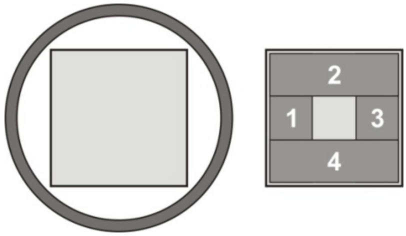

Fig. 5 Areas on which CT values are collected by avoiding the area disturbed by sampling tool

5.1 Estimation of Porosity or Void Ratio

CT numbers for all specimens from four different areas of the specimens (Fig. 5) are shown in Table 1. For estimation of porosity we need an average value of CT numbers for the specimens, which is in the last column of the table 1. Fig. 6 shows a plot of void ratio against average CT number for all cases. A linear relationship is noted. A regression analysis between the average CT number and void ratio leads to Equation (4) below.

$$e = (-0.005) \times (CT\ Number) + 0.6581 \tag{4}$$

The correlation coefficient (R^2) and standard deviation (*SD*) between the void ratio and CT number is 0.91 and 0.38 respectively, which shows high correlation. In particular, it seems that the linear correlation is stronger in the range of smaller void ratios, compared with the range of higher void ratios.

We now focus our attention at the spatial variation of CT numbers at various locations from where the samples for scanning have been extracted. Here we use a Modified CT number (MCT) for the sake of clarity and convenience. It is simply = - CT x 10. Fig. 7 shows the variation of MCT as well as void ratios within the specimens. It is seen that void ratio at the top part of the specimen is higher than the middle part. It is consistent with the loading history in which compression is followed by swelling due to release of stress. It is further noted that the higher void ratio is reflected in the MCT numbers. In this figure, on the left hand side, computed values of void ratio from Equation (4) are also plotted. A close matching is noted indicating that a relationship between CT numbers and porosity may be unique for a clay like material.

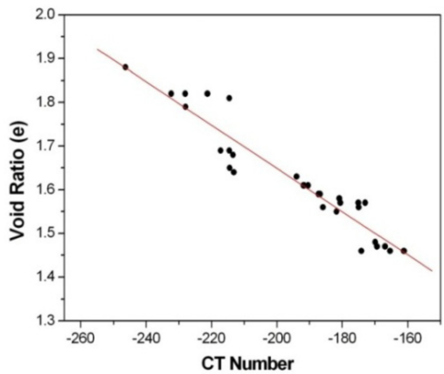

Fig. 6 Relation between void ratio and CT Number

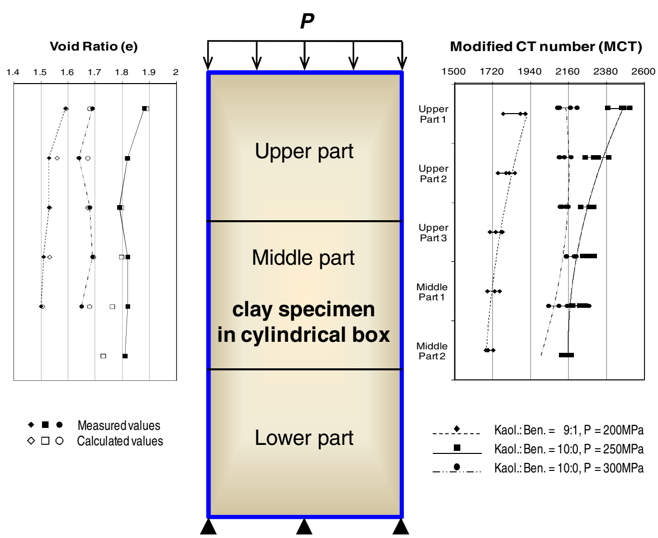

Fig. 7 Distribution of void ratios and MCT values along the thickness of clay specimens

6 Conclusions

In this study CT imaging technique from X ray machines having a resolution of 25 microns has been used to estimate pore characteristics of fine grained soils like clay. The following specific conclusions can be drawn:

(1) For a quantitative analysis of scanning results from micro X-ray CT images, estimation of 'gain' and 'offset' parameters specific to an equipment and environment are required to be determined to obtain accurate CT numbers. This was carefully carried out and seems to have produced consistent set of results.

(2) Although in all specimens more than a one water saturated pores co-exist within a pixel, it is possible to visualize the distribution of pores and pore-structure from the CT numbers. Thus, the concept of estimating porosity from scans for fine grained soils was established.
(3) From the CT numbers of specimens composed of kaolinite and bentonite pre-consolidated to pressures ranging from 150 – 350 kPa inducing different porosities, a correlation between CT number and the porosity was established. The correlation coefficient (R^2) was found as 0.91, indicating a strong linear relationship between the two variables.
(4) CT values in consolidated specimens indicate that there is a clear spatial variation of average values within the specimens which indicates that this methodology can be used not only for rapid determination of porosity but also for pore-size distribution. This will prove to be a major breakthrough as the alternative techniques are expensive, time consuming and have safety issues associated with them.

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