Advances in **Multiphysical Testing** of **Soils and Shales**

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

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- 1. Background & motivation
- 2. Overview of micro X-ray CT scanning
- 3. Calibration of CT data
- 4. Experimental programme (coarse and fine soils)
- 5. Prediction of porosity & pore-size distribution
- 6. Concluding remarks





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<u>Transport properties of geomaterials</u> are an important issue in many fields of engineering analysis and design, which are mainly governed by <u>porosity as well as micro-structural</u> <u>characteristics</u> such as pore-size distribution, etc.

 Porosity and pore-size distribution can be measured by well known techniques such as MIP, Nitrogen adsorption test, etc. but it is not easy experiment to conduct especially <u>for fine</u> <u>cohesive materials.</u>

 Try to directly calculate porosities of the chosen volumes at certain positions in a specimen by using Micro X-ray CT
 porosities at certain regions as well as spatial distribution of the porosities of a specimen



- 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 <u>sandy soils</u> whose pore or particle shape can be captured directly.



 In this study, we explore if CT technology can be applied to <u>fine</u> cohesive soils whose particles are much smaller than the <u>minimum pixel size</u> of micro X-ray CT.



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Micro X-ray CT scanner (90kV) in KICT

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Sauraa	■Max output: 0 kV to 90kV
Source	Max definition : 5microns (at 4W)
Comoro	■Max photo size: 49.2mm x 49.2mm
Camera	Critical resolution : 10Lp/mm
Drive	Rotating resolution: 2.5/1000deg
shaft	■Wobble: About 0.01mm





Basic form of computed tomography(CT)

The **attenuation of x-rays** of wavelength λ is

$$I = I_0 e^{-\mu x}$$

where I_0 is the intensity of the unattenuated x-ray beam, I is the beam's intensity after traversing, x is thickness of (homogeneous) material with *µ* being the linear attenuation coefficient



Inverse calculation (back-projection) to obtain (μ_i) at voxels





Calibration of image data (μ_i)

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

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CONSTRUCTION TECHNOLOGY

Standardization to CT values

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



Keys to obtain reliable images

- Two concepts are important in image analysis: spatial resolution and contrast resolution.
 - Spatial resolution (size) is the ability to resolve close and high contrast features in the image. Two objects will not be separable in an image if the spatial resolution of the image is larger than the distance between them.
 - Contrast resolution (intensity) is the ability to distinguish between objects with small contrast difference (attenuation coefficient).
- In general, <u>there are many inherent noises</u> on CT image data coming from many different resources: mechanical inconsistency, sensitive scanning environments, etc., which can not be avoided completely.
- Necessarily, <u>all the noises (artifacts) should be aware especially</u> <u>in quantitative use of CT data</u>. In general, <u>an industrial CT</u> <u>machine only provides raw linear attenuation values which need</u> <u>to be calibrated externally for each kind of noises</u>.





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Calibration for Beam hardening

Solutions

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- Use higher-energy x-rays
- Pre-harden the x-rays
- Take wedge through similar material (wedge calibration)
- Software correction during reconstruction









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Ring artifact reduction



No correction

9-point smooth

21-point smooth





 Calibration procedures for physical errors from source, detector and drive shaft arrangement depending on assembling and installing condition

	Parameter		Spec.	Measured
	Pixel size	[mm]	0.048	0.048
Detector	Width	[mm]	1024	824
	Height	[mm]	1024	824
	Vertical	[Pixel]	0	+38.1133
Offset	Horizontal	[Pixel]	0	+1.1659
	Tilted angle	[degree]	0	-0.0029
Oriente	SDD	[mm]	343.5	358.2094
Geometry	SOD	[mm]	241.1	272.3499







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Raw image

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Scanning for fine grained soils

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 Image for coarse grained soils (Jumunjin sands)

Image for fine grained soils







Basic concept for measuring porosity

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For fine grained soils



		Coarse	Fine sc	il (mm)
		soil(mm)	Silt	Cohesive soil
	AASHTO	0.074	0.002 ~ 0.074	0.002 or less
	USCS	0.05 or more	0.005 ~ 0.05	0.005 or less



X Key hypothesis:

the pixel occupied by more solid particles has larger CT value



Sampling and scanning mould

- Prepare a sampling mould to minimizes the disturbance and stabilizes the sample in scanning
- The mould material should not have effect on scanning







ID	OD	Acrylic thick	Sample thick	Material
(mm)	(mm)	(mm)		
32	40	4	20	Acrylic





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Testing of Soils and Shales

Making of artificial clayey soil samples

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Material : Kaolinite, Bentonite

※ Void ratio to be adjusted using swelling of bentonite

- Mix ratio : 9:1, 100% Kaolinite
- Specific gravity : 2.68
- Pre-consolidation load : 150 kPa ~ 400kPa
- Initial void ratio: 1.4 ~ 1.72



Experimental processes (sampling & scannings and Shales



Cutting



Before inserting mould



After inserting moolds



Sample moulding



After sample moulding



connecting to a rod for scanning



Scanning



Scanning for gain calibration





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relationship

The area to be avoided

- 1. Sampling disturbance
- 2. Cupping error on boundary
- 3. Rotation axis



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Experimental results

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Vol. fraction	/ol. fraction Pre- Cutting		Pre-			Modified CT number (MCT)				Average
(Kaolinite	Consolidation	location	ratio (e)		(-CT no^10)			MCT		
: Bentonite)	pressure			Area 1	Area 2	Area 3	Area 4			
			1.46	1752	1735	1722	1741	1741		
	400kPa	Upper Part	1.46	1635	1670	1660	1654	1654		
			1.46	1584	1584	1621	1655	1611		
			1.47	1692	1757	1625	1697	1693		
		Upper Part	1.47	1653	1720	1668	1635	1669		
	350kPa		1.46	1640	1589	1612	1607	1612		
0.1		Middle part	1.63	1960	1947	1898	1951	1939		
5.1			1.58	1850	1798	1718	1866	1808		
			1.59	1882	1911	1783	1911	1872		
		Upper Part	1.56	1706	1778	1739	1771	1749		
	200kBa		1.57	1799	1818	1753	1850	1805		
	200KFa		1.57	1842	1733	1733	1691	1750		
		Middle part	1.59	1923	1842	1873	1831	1867		
			1.48	1692	1724	1682	1696	1699		
-	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		
10:0			1.65	2103	2151	2277	2043	2144		
		Upper Part	1.79	2306	2298	2280	2232	2279		
			1.88	2468	2389	2481	2515	2463		
			1.82	2258	2393	2334	2305	2323		
	250KPa		1.82	2309	2293	2244	2272	2280		
		Middle part	1.82	2258	2226	2179	2185	2212		
			1.81	2121	2148	2177	2132	2145		
			1.61	1924	1895	1850	2001	1918		
	150kPa	Upper Part	1.61	1884	1903	1852	1976	1904		
			1.61	1807	1973	1889	1993	1916		





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Linear relationship (CT-e)

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 The correlation coefficient (R²) and standard deviation (SD) between the void ratio and CT number is <u>0.91 and 0.38</u> respectively



Validation and void-ratio distribution in V







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Pore distribution in fine soils

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- Hypothesis: a voxel contains a pore in different size
- Void voxels are combined, thereby leading a bigger pore





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- CT imaging technique has been used to estimate pore characteristics of fine grained soils.
- A correlation between CT number and the porosity was established. The correlation coefficient (*R*²) was found as 0.91, indicating <u>a strong linear relationship between the</u> <u>two variables.</u>
- Although in all specimens more than one of water saturated pores co-exist within a pixel, it is possible to visualize the distribution of pores and pore-structure from the CT numbers.
- There is a clear spatial variation of porosities within the specimens which indicates that this methodology can be used not only for rapid determination of porosity but also for pore-size distribution
- <u>A formulation has been made for identifying different</u> phases in micro-pores which will be reported shortly





Concluding remarks

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<u>New high-capacity industrial CT facility for 'live' testing</u> <u>available in KICT now</u> which is <u>open for any research</u> <u>group. Please contact us for further information</u>





Switchable hige-power multi tube type

1) High power target X-ray tube (320kV)

 > Closed High power tube (FSS: 0.4mm)
 2) Directional target X-ray tube (225kV)
 > Micro focus open high power tube (FSS: 6μm)

- 3) Transmission target X-ray Tube (120kV)
 - > Nano Focus Open Tube (FSS: 400nm)

Dimension	3000 x 1850 x 2700 (mm)
Weight	20,000 kg

- Object Loading Size: max. ø500mm x 1000mm(h)
- Work Table Withstand load: max. 100kg
- 3DCT area: ø300mm x 900mm(h)







"Being safe is risky, and being risky is safe!"

Thank You.

Examples for spatial resolution calculation

Tube Type	Specification	Sample size (ø mm)	R FOV (ø mm)	Mag(x)	Spatial Resolution (µm³)	Voxel Resolution (µm³)	Remark
		5	1.12	360	0.68	0.55	FDD: 900mm
Turnensierien	✤Voltage :	15	3.34	120	1.71	1.63	
	120KVp	30	6.66	60	3.36	3.25	
target X-ray	♦Current : 200uA	45	10	40	5.02	4.88	
Tube	♦Focus > 0.4um	60	13.32	30	6.68	6.50	
		60	60	6.67	30	29.26	FOV Max
Directional target X-ray Tube	 ♦Voltage : 225KVp ♦Current : 3.0mA ♦Focus > 6um 	34	6.84	58.8	6.81	3.34	FDD: 1000mm
		70	14.06	28.6	9.08	6.87	
		100	20.08	20	11.51	9.80	
		150	30.12	13.33	15.99	14.71	
		180	36.14	11.11	18.81	17.65	
		180	180	2.22	90.06	88.22	FOV Max
	. √oltage .	150	150	2.68	261.7	73.3	
High Power 3 target X-ray • Tube •	 vonage : 320KVp ♦Current : 5.6mA ♦Focus > 	220	220	1.83	212	107.29	FDD: 1100mm
		250	250	1.61	195	122.48	
		300	300	1.34	180.54	146.62	
	400um	340	340	1.18	180.04	166.29	





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System configuration







Dimension	3000 x 1850 x 2700 (mm)
Weight	20,000 kg
Power	220VAC, 50A







- X-ray source specifications
- Nano focus & high voltage multi tube type
- Source 1) Max voltage : 20 to 320kv
- Source 2) Max voltage : 30 to 225kv
- Source 3) Max voltage : 30 to 120kv
- Focal spot size :

0.4mm • 6µm • 400nm

- Specification of characteristic
 - High power target X-ray tube

 Closed High power tube

 Directional target X-ray tube

 Micro focus open high power tube
 Transmission target X-ray Tube
 Nano Focus Open Tube







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- X-ray detector specifications
- Type : Digital Flat Panel Detector
- Radiation Energy: 40~320kV
- Active Area : 400(h) x400mm(v)
- Pixel Matrix : 1,024(h) x 1,024(v)
- Pixel Pitch : 200 µm
- Resolution;
 - 2.5lp• mm @15FPS (1x1)
 - 1.25lp•mm @ 30FPS(2x2)
- A/D Conversion:

16-bits (65,536 gray levels)







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- Work table · manipulator
- Object Loading Size: max. ø500mm x 1000mm(h)
- Work Table Withstand load: max. 100kg
- 3DCT area:
 - ø300mm x 900mm(h)
- Repetition accuracy : 0.004°
- High Precision Granite Surface Plate Anti-magnetic High shock tolerance
 - Free from vibration







Servo axis: 6 axis

- X-ray Source Traverse: 900mm
- Detector Traverse(Top Down movement) > 900mm
- Detector Traverse (Left-Right) : 500mm
- Work table Traverse (Left-Right) : 500mm
- Work table Traverse (Top-Down) : 100mm
- Work table Rotation : 360°
- Work table withstand load : Max. 100kg





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- Radiation safety
 - Shield Cabinet : Pb + Fe
 - Survey window :
 - Pb Windows
 - Door Safety Interlock
 - Emergency switch
 - Master key switch
 - X-ray warning indication
 - Equipment Surface of Leaking
 - Dose 1µSv/h less than Radiation Producer Design Approval





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