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Identifying porosity distribution and swell anisotropy of laboratory sedimented high plasticity clay through NMR imaging

Identification de la distribution de la porosité et de l'anisotropie du gonflement de l'argile à haute plasticité sédimentée en laboratoire grâce à l'imagerie RMN

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ABSTRACT: When studying fundamental features of soil behaviour it is common to resediment or remould “artificial” soil samples under well-defined conditions to limit uncertainties caused by variability in soil structure. Therefore, the laboratory sedimented sample is a useful reference state for a given soil. However, the sedimentation process inevitably causes anisotropy which is previously linked to sedimentation structure. This is related to the anisotropic stress state of oedometric confinement used in laboratory sedimentation. As such anisotropy is unavoidable it is necessary to identify it. More troublesome is the potential unwanted heterogeneity caused by the separation of constituents during the sedimentation process. To investigate these two features, large (Ø20cm x 6cm) laboratory sedimented high plasticity clay discs were prepared. On both vertically and horizontally oriented subsamples at different locations in these discs, the porosity distribution was mapped through low field Nuclear Magnetic Resonance (NMR) imaging. This allowed verification of the level of homogeneity achieved by the laboratory sedimentation procedure. Further, the anisotropy of the discs was studied by measuring the free swell of the subsamples at the two orientations. The obtained results provide insights into the effectiveness of laboratory sedimentation for creating homogeneous reference soil samples and further exemplify the usefulness of NMR imaging in identifying initial porosity distribution and porosity evolution during swelling.

RÉSUMÉ: Lors de l’étude des caractéristiques fondamentales du comportement du sol, il est courant de resédimenter ou de remouler des échantillons de sol « artificiels » dans des conditions bien définies afin de limiter les incertitudes causées par la variabilité de la structure du sol. Par conséquent, l’échantillon sédimenté en laboratoire constitue un état de référence utile pour un sol donné. Cependant, le processus de sédimentation provoque inévitablement une anisotropie qui est auparavant liée à la structure de la sédimentation. Ceci est lié à l’état de contrainte anisotrope du confinement oedométrique utilisé en sédimentation en laboratoire. Une telle anisotropie étant inévitable, il est nécessaire de l’identifier. Plus gênante est l’hétérogénéité potentiellement indésirable provoquée par la séparation des constituants au cours du processus de sédimentation. Pour étudier ces deux caractéristiques, de grands disques d'argile sédimentés en laboratoire (Ø20 cm x 6 cm) à haute plasticité ont été préparés. Sur des sous-échantillons orientés verticalement et horizontalement à différents endroits de ces disques, la distribution de la porosité a été cartographiée par imagerie par résonance magnétique nucléaire (RMN) à faible champ. Cela a permis de vérifier le niveau d’homogénéité atteint par la procédure de sédimentation en laboratoire. De plus, l'anisotropie des disques a été étudiée en mesurant le gonflement libre des sous-échantillons aux deux orientations. Les résultats obtenus donnent un aperçu de l'efficacité de la sédimentation en laboratoire pour créer des échantillons de sol de référence homogènes et illustrent en outre l'utilité de l'imagerie RMN pour identifier la distribution initiale de la porosité et l'évolution de la porosité au cours du gonflement.

Keywords: Resedimentation; sample homogeneity; swelling anisotropy; nuclear magnetic resonance (NMR).

1 INTRODUCTION

Soil behaviour depends both on the material properties (i.e. mineralogical and particle size distribution, and particle size and morphology) as well as its state (porosity and its distribution, particle arrangement, bonding/cementation, etc.). Burland (1990) devised a reference state, namely a remoulded conditions, which represents the intrinsic material behaviour by providing a term of comparison and thereby allowing to distinguish between behavioural features linked to material properties and those linked to soil “structure”, defined by Cotecchia and Chandler (2000), as the combination of fabric (i.e. particle orientation) and bonding. Although the remoulded sample starts from an isotropic state (no preferential particle orientation), the anisotropic stress state experienced during oedometric compression, which is used for laboratory preparation of fine-grained samples, induces a preferred particle orientation. Furthermore, when using resedimentation rather than remoulding as a preparation method, Stallebrass et al. (2007) observed particle sorting caused by the sedimentation process (similar to the effects utilized when determining particle size distributions, see e.g. ASTM D7928). This led to a parallel shift of the compression line relative to the intrinsic one, based on the reconstitution method proposed by Burland (1990).

It is apparent, that laboratory produced soil samples will inevitably contain some degree of anisotropy and potentially some unwanted heterogeneity. A large laboratory resedimented disc (D=20cm x H=6cm) of Røsnæs Clay was created and several small (D=2cm x H=2cm) specimens were taken at different locations to investigate these aspects. The degree of heterogeneity was determined through measurements of the pore size distribution and porosity at these varying positions through Nuclear Magnetic Resonance (NMR). The degree of anisotropy was estimated by exposing both vertical and horizontal plugs to demineralised water and monitoring the free swell process also through NMR measurements.

The combination of these observations provides an assessment of laboratory resedimentation procedures for creating relevant reference soil samples.

2 METHODS AND MATERIALS

The material used as the basis of the study is the already mentioned disc of resedimented Røsnæs Clay. The details of the resedimentation procedure are described by Kinslev et al. (2022) and are briefly summarised as: The soil is mixed into suspension with artificial pore water (composition) ensuring a water content of 500%, after which it is sedimented in a purpose build consolidometer and compressed to 1000 kPa. The final product (the disc) is removed and stored in several layers of waxed cling film while not being sampled.

The specimens are sampled in plastic confining rings to enable NMR measurements since metal rings would interfere with the magnetic field. These confining rings have a perforated base, on which is placed a filter paper, resulting in uptake of water from both ends but one-sided swelling once exposed to demineralised water. In total seven specimens were sampled from the disc, positioned as illustrated in Figure 1.

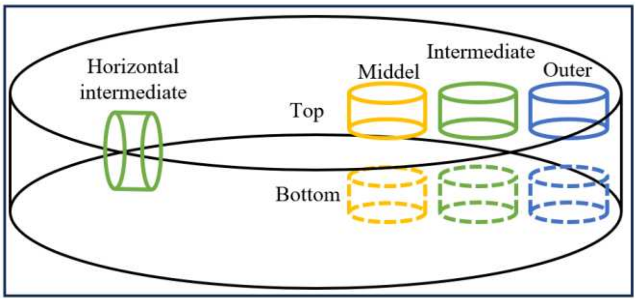

Figure 1. Position of specimens in the resedimented disc.

The Røsnæs Clay used in this study originates from core samples taken during the ground investigation campaign performed for the Fehmarn Belt tunnel project and has properties as listed in Table 1.

Table 1. Selected properties of Røsnæs Clay. Data from Awadalkarim 2014, Di Remigio 2021 and Kinslev 2022.

Grain density	[-]	2.75-2.85
Liquid limit	[%]	93-181
Plastic limit	[%]	26-39
Clay mineral content	[%]	64-68
Kaolinite	[%]	11-18
Illite	[%]	13-15
Smectite	[%]	33-43
Specific surface area	[m^2/g]	56-68

The Røsnæs Clay formation was deposited in the North Sea during the Palaeogene period. In Table 1 only the clay mineralogy is detailed but it should be added, that the remaining mineralogy is dominated by very fine particles (primarily quartz) as nothing is retained by a 63 micron sieve.

The NMR measurements are based on T_2 relaxation; hence the exponentially decaying relaxation of the nuclear angular momentum of hydrogen nuclei to an external magnetic field after tilting by 90 degrees via a timed magnetic pulse. This relaxation is governed by three mechanisms: Bulk relaxation, diffusion and surface relaxation. At the fast diffusion limit (almost all soils), relaxation is dominated by the surface relaxation and T_2 relaxation times correlate linearly to pore size.

Bulk NMR measurements were taken for each specimen to determine the relative pore size distribution as well as total porosity. Subsequently the specimens were exposed to a 0.18M NaCl solution and allowed to free swell for 800 min, while NMR bulk measurements were taken at relevant time intervals (more closely spaced initially, when the free swell process is fastest).

3 RESULTS AND DISCUSSION

The homogeneity of the produced disc is evaluated comparing porosity distributions. The total porosity in Figure 2 is obtained as the area under the curve (also reported in legend), while the T_2 relaxation time distribution is a proxy for pore size distribution. Figure 2 shows the values for each of the vertical specimens (see Figure 1).

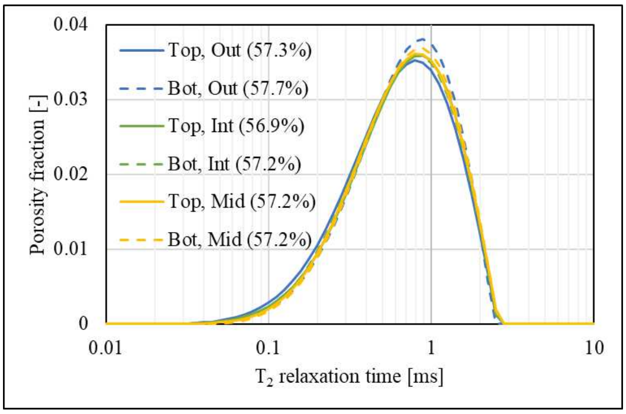

Figure 2. Distribution of porosity fractions with T_2 relaxation times for the six vertical specimens with their total porosity noted in the legend.

From Figure 2 it may be seen that the porosity varies between 56.9 and 57.7% across the disc, which is deemed to be minimal. Further, since there is no consistent trend in the total porosity distribution between the positions in the disc (from middle to outer or top to bottom) the variations in porosity are more likely related to the accuracy of the NMR measurements as well as potential small disturbances during the sampling process. In addition to the consistent total porosity between the specimens, Figure 2 also shows that the pore size distribution is almost identical between them. They all display a similar build up from 0,05 ms to a single peak at 8-9 ms and a maximum relaxation time of approx. 2.5 ms. Altogether this indicates that particle segregation was minimal and that the applied sedimentation procedure resulted in a homogenious sample. This sucess is of course related to the particular soil in this study as another attempt of laboratory sedimentation by Stallebrass et al. (2007) was visibly heterogenous. However, it is worth noting that Røsnæs Clay is a natural material with a heterogenious mineralogy albeit with limited partice size varriation.

In the assessment of sample anisotropy generated by the oedometric compression the development of total porosity with time during free swell is plotted in Figure 3.

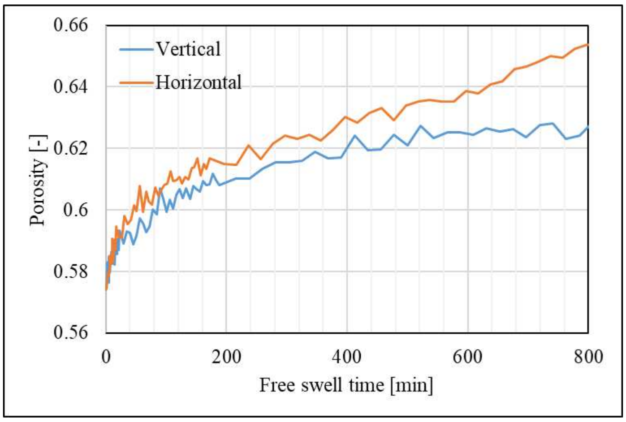

Figure 3. Time development of porosity in the vertical and horizontal specimens subjected to free swell.

From Figure 3 it may be seen that the horizontal specimen swells faster and more compared to the vertical one. At the 800 min mark the vertical specimen has a porosity of 62.8% relative to the 65.4% for the horizontal one, resulting in roughly 50% more swelling (relative to the starting point at 57.4%) of the later. Despite the horizontal specimen swelling slightly faster than the vertical one, the porosity development between the two is very comparable up to the 600 min mark, where the vertical plug plateaus while the horizontal one keeps swelling at a close-to-constant rate. This indicates, that the free swell should have been allowed to continue for a longer period of time to ensure stability of the results. However the overall trend is still clearly evident from the tests.

The clear swelling anisotropy of the sample shows that the anisotropic stress state during compression caused particle rearrangement, which is retained after release of this stress state (during dismantling where the stress state becomes isotropic). The NMR free swell tests can be used to quantify the degree of anisotropy, however for a more direct assessment of the particle arrangement, these results must be compared to e.g. molecular dynamic modelling or scanning electron microscopy performed on planes positioned at different height along the specimens.

While Figure 3 shows the development of porosity during free swell, no information is gained about how the pore space changes to accommodate this increase of porosity. To illustrate this, the pore size distribution before and after free swell are compared in Figure 4. Here it may be seen, that the pore size distribution retains its general shape during the free swell regardless of the specimen orientation. The curves expand in the direction of the largest pores, such that the initial build up of the porosity distribution is completely unchanged, while the front moves to larges relaxation times.

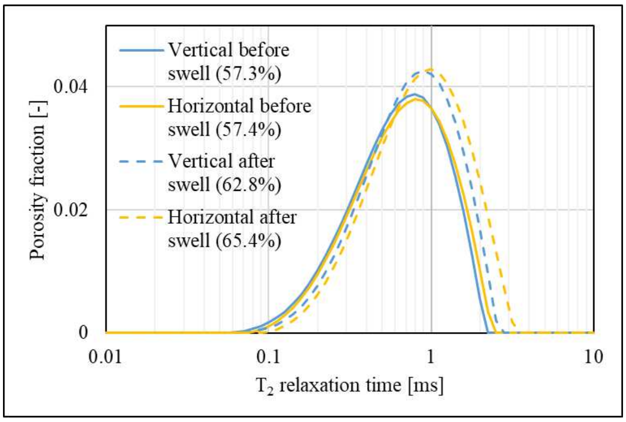

Figure 4. Bulk porosity before and after free swelling with their total porosity noted in the legend.

Because the size of the smallest pores are almost unchanged, the particle interactions occur at the same positions (hence the electro-static repulsion is unchanged) and the stress release causes “only” an elastic rebound of the entire soil skeleton. Since this process is the same in both the horizontal and vertical directions, the anisotropy must be related to boundary conditions of the plugs hence which plane (vertical or horizontal) allowed to deform.

4 CONCLUSIONS

The presented investigations confirm the usefulness of laboratory resedimentation for creating representative reference samples with minimal heterogeneity, provided particle size is below 63 microns. After laboratory sedimentation and compression of a high plasticity clay (Røsnæs Clay) a homogeneous pore size distribution, as measured through NMR T_2 relaxation, was observed throughout the sample. Only minimal variations occurred, which fall within the accuracy of the measurement method. In addition to the particular presented case, the study illustrates the usefulness of NMR measurements in evaluating sample homogeneity. Although applied destructively in this study (by sub-sampling), the method also has potential for being applied non-destructively through imaging or slice selective bulk measurements. Beyond the evaluation of the homogeneity, the investigations showed that significant sample anisotropy emerges from the sedimentation and compression process as a 4% increase of free swell is observed in the horizontal relative to vertical direction. This quantification represents a key step in understanding the development of stress induced anisotropy.

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