Testing sensitive clays through time and length scales

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Abstract. In the Nordics, engineering of sensitive soils is of vital importance for realising the urban environment and the infrastructure connecting and underpinning it. The hydro-mechanical behaviour of sensitive clays is strongly affected by the geological deposition history in the area and subsequent human activities resulting in changes in stress and environmental loading. Finally, the characteristic physico-chemical identity of the clay-water system is expressed by the sensitivity of the material. A combination of classic geotechnical tests in the laboratory needs to be complemented with state-of-the-art technologies for material analyses, in order to deepen our understanding of the interaction between the colloidal nature of the clay and the observed response at the engineering scale. Until now these activities have been performed separately. The microstructural observations using various microscopy techniques were not directly combined with classic geotechnical tests. In contrast, the work presented herein showcases methodologies for simultaneous monitoring of fabric and mechanical probing under controlled conditions on samples of sensitive clay. In order to enable real-time fabric measurements of samples of sensitive clay two non-invasive techniques are utilised: X-ray Scattering (XS) and X-ray Computed Tomography (XCT). Furthermore, a bespoke apparatus for sample probing is designed and built at Chalmers University of Technology. The design of the apparatus is adapted to the special challenges of very soft samples and addresses issues of sample quality in soft soil testing (i.e., sample mounting, membraneless configuration). The intention of this work is to demonstrate the feasibility of expanding geotechnical testing outside the limits of the traditional geotechnical laboratory, combining geotechnics with state-of-the-art technologies for material analysis. This, in the end, provides a critical view on the perception of the material and its constituents that aims to contribute in improvement in geotechnical laboratory testing and the development of advanced constitutive models for sensitive clays.

1. Introduction
Sensitive clay is a common type of soil in the Nordic countries with large consequences on construction projects, due to the occurrence of these problematic soils near the coast and rivers where the majority of the population lives. Thus, it concentrates the attention of geotechnical practice and research. In particular, sensitive clays exhibit anisotropy, rate & temperature dependency and degradation of strength and stiffness when hydromechanically loaded -sheared to failure-. Sensitivity is used to describe the change in undrained shear strength between the intact and remoulded state of the clay. This large strength difference is attributed to the microstructural changes of its constituents during shearing and is defined as:

\[ S_t = \frac{S_u}{S_{ur}} \]  

(1)
where $S_u$ is the undrained shear strength of the intact soil and $S_{ur}$ is the undrained shear strength of the remoulded soil [1]. Sensitivity is a result of thixotropy of the polydisperse colloidal system. In engineering this can be expressed in its extreme form as events of catastrophic quick-landslides (e.g. Surte 1952; Tuve 1977; Smáröd 2006). The sensitive macroscopic behaviour can be explained by the microstructure of the clay. Sensitivity often is linked to high void ratio and high natural water content values (water contents usually higher than those measured in the laboratory for the liquid limit of the remoulded material). These are indications for an open structure of clay aggregates. The microstructure of fine-grained soils [2, 3, 4] as well as sensitive soils [5, 6] is well studied (eg. Figure 1).

Most studies employ Scanning Electron Microscopy (SEM) and Mercury Intrusion Porosimetry (MIP). These methods have a great contribution on the understanding of the microstructure of fine-grained soils, within the experimental constraints of the method, due to the intrusive preparation techniques required, commonly involving the replacement of the pore water. Contemporary studies demonstrate that those treatment methods can influence the resulting microstructure [7]. It is of great importance to employ real-time monitoring of the material through several length scales, in order to link the macroscopic behaviour usually measured in the laboratory by element testing with the changes in material structure. Non-invasive technologies can provide such continuous monitoring during mechanical probing.

2. Methodology

In order to span the observations through time and length scales, two non-invasive techniques are selected to study the material in nano- and meso- scale:

- Small Angle X-ray Scattering (SAXS)
- X-ray Computed Tomography (XCT)

Small Angle X-ray Scattering (SAXS) is a non-destructive technique, which utilises the change of direction of X-rays (scattering) due to interaction with matter (particles) to provide bulk measurements of the mechanical structure of matter in the Å to nm range. In this way, we can observe changes in distances between basal units in the clay particle and the change of orientation of the clay particles. Basic requirement of the method is the miniature size of the sample (1 mm). In order to create such a small sample, a thin-wall capillary tube was used to contain the soil sample in its natural water content. The soil was captured by penetrating
Figure 2. (a) Sampling procedure of clay in capillary tubes; (b) the capillary tube is used to form a plane strain oedometric cell.

Figure 3. The specially designed oedometric cell mounted in the experimental hutch of SAXS beamline in MAXLAB.

Another non-invasive method which allows the study of the composition and internal structure of bulk objects using X-ray radiation is X-ray Computed Tomography (XCT). This method provides 3D images of the object scanned with X-rays, based on the measurement of transmitted radiation using a reconstruction algorithm. The most common X-ray tomography method is based on the measurement of the attenuation of X-rays. This tomography is absorption-based
and works as an extension of radiography. The use of CT has been an important tool in medical imaging since the 1970s. Tomography can resolve structures with inherent density contrast. For the case of fine-grained soils (clays) the natural contrast is only available between the colloidal (clay) and granular (silt, sand) fractions.

Tomographic 3D images can be used to calculate the strain distribution through the sample by the use of Digital Image Correlation (DIC) techniques. Digital images are arrays of integer or float numbers. Therefore, they can be manipulated and compared in the digital domain. The method used here, digital image Correlation, involves statistical comparison of two subsequent images in different deformation stages. This provides the displacement field of a grid of points in the two images. Once the displacement field is obtained, the strain field can be calculated.

Figure 4. Schematic layout of the XCLAY miniature triaxial

XCT is a very promising experimental technique to acquire the internal strain field of clay specimens during a geotechnical test. Nevertheless, for such a test to be realised there are major modifications that need to be made on the triaxial apparatus. A bespoke apparatus is developed in order to combine tomography and in-situ mechanical probing of soft sensitive clays. The miniature hydraulically controlled Bishop-Wesley cell (Figure 4) can test miniature samples (10 mm diameter, 20 mm height) inside the tomograph, while it rotates 360° for the tomographic scan to be acquired. Two requirements mandate a small sample size for tomographic X-ray imaging: (1) the high degree of attenuation of X-rays by wet clay (and the fluid in the cell)
and (2) the low hydraulic conductivity of clay samples, which results in long test duration for
drained stress/strain paths. The sample size must be sufficiently large to ensure that the sample
contains a representative distribution of particle and void sizes. In the case of fine-grained soils
this requirement is usually met with ease. Sample mounting without any additional disturbance
turns out to be a special challenge, due to the required small size of the sample. An **untouched by hand** mounting methodology is applied. Soil is first sub-sampled from a bigger sample in
a cylindrical tube of 10 mm internal diameter with steady sampling rate. Then the miniature
sample is extruded from the tube by sliding the tube downwards on the pedestal. After this
the sample is exposed and mounted on the pedestal while the sampling tube remains trapped
around the bottom part of the pedestal during the test. The sample size also makes any folding
of a membrane a rather difficult task. Therefore, a membrane-less configuration is selected.
The Norwegian Geotechnical Institute (NGI) presented a method to perform bare sample tests
in clays, using the paraffin method [8]. Paraffin is immiscible with the pore-water of the sample.
Also, the contact surface between the cell liquid and the soil can sustain the maximum pressure
without penetration of the paraffin oil in the fine pore system. The critical connection for sealing
is at the point of contact between the sample and the mounting ends on the top and the bottom
of the sample. A sharp edge which penetrates the sample assures that there is no leakage at the
early stage of loading. An extra reason to avoid the use of a membrane, is to avoid the need to
support the weight of the top cap and possible bending moments created during the mounting of
the sample. The stresses that would result from an improper mounting procedure, could reach
half the undrained strength for a soft clay.

### 3. Results

Results from the two methods are presented in this section. A first piece of information obtained
by SAXs is the composition of the soil minerals. In Figure 5, the two peaks that can be
distinguished in the signal correspond to the two minerals: illite and montmorillonite.

If the peaks evolution is followed during the compression stages changes can be recorded. For
instance, in Figure 6 a new peak is observed only for one of the minerals. It is worth noticing
that the change in the mineral is observed only after the monotonic loading has finished.

For the case of miniature triaxial testing the performance of the test needs to be correlated
to results of a standard size triaxial specimen test (Figure 7a & 7b). Preliminary comparative
results between a standard size (diameter 50 mm x height 100 mm) and a miniature test are
presented for samples from the Utby test site (Gothenburg, Sweden) retrieved from 9 m depth.
Both tests are undrained compression tests of anisotropically consolidated samples, close to in-
situ conditions. Shearing was conducted with a constant rate of strain, 0.01%/min and 0.1%/min
Figure 6. A new peak arises for one of the minerals only after the monotonic loading is finished [9].

Figure 7. (a) Stress paths comparison for miniature and standard sized specimens for undrained triaxial compression.; (b) the capillary tube is used to form a plane strain oedometric cell.

for the standard and miniature specimens respectively. The results of the miniature test seem to relate remarkably well to the standard size test in terms of peak deviatoric stress. This peak, however, mobilises at larger strain levels for the miniature test. Furthermore, softening response is also different from the standard test. It is important to consider that different strain rates were applied in the two different tests, as well as the absence of an membrane in the miniature tests. Nevertheless, a complete series of comparative testing is necessary to demonstrate the extent of influence of the size effects on test results.
4. Conclusions
It is demonstrated that advanced non-invasive techniques can be successfully combined with hydro-mechanical testing of clay samples. This will yield information on the structural evolution of sensitive clays in a wide range of length scales. Necessary procedure is the miniaturisation of element tests. New sampling methodologies are specially designed for each non-invasive technique used. The miniature sample size is not only focused on the use of XCT, but can tackle sample quality concerns. Subsampling from the core of a 50 mm tube eliminated the effect of the oxidization that usually starts radially in the tube. Additionally, the membraneless configuration reduces sample disturbance during mounting. The preliminary results show that sample miniaturisation does not affect the outcome of the test in terms of shear strength. Finally, new insight on the nanoscale response of the material is provided by the use of real-time SAXS measurement during in-situ testing of sensitive clay.

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