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Research paper

X-ray tomographic method for measuring three-dimensional deformation and water content distribution in swelling clays

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A B S T R A C T

A non-invasive method for simultaneous measurement of the 3D displacement field and the water content distribution of a wetted solid material is developed. The method is based on comparison of X-ray tomographic images of a material sample in the reference state and in the wetted and deformed state. The deformation and water content analyses were successfully compared with numerical results for a cylindrical rubber test sample under axial compression, and with gravimetric results from axially wetted and sliced cylindrical bentonite samples, respectively. The methods were applied in a 4D study (three spatial dimensions and time) of wetting and deformation of purified swelling bentonite doped with glass tracer particles, and wetted with synthetic groundwater. The results obtained for bentonite samples are repeatable and appear qualitatively correct and plausible. They are useful e.g. in validating models involving transport of water and the resulting swelling deformation of bentonite. The method is potentially applicable also in other processes involving liquid transport and deformation such as wetting/swelling and drying/shrinking of heterogeneous materials. A prerequisite for the applicability of the method is that the material contains sufficient amount of local inhomogeneities visible and identifiable in successive tomographic images to facilitate deformation analysis, and that change in water content affects the total density enough to be observable in X-ray images.

1. Introduction

Transport of liquids in partially saturated solid materials and the possible deformation of the material induced by local changes in liquid content are of great interest in many areas of materials science and technology. A number of complex physical and chemical mechanisms contribute to such transport and deformation in processes involving wetting or drying of e.g. soils, building materials, foods and various biological materials (Carmeliet and Roels, 2001; Moldrup et al., 2001; Meinzer, 2002; Saguy et al., 2005). Theoretical approaches based on first principles towards modeling these processes tend to become complicated, and phenomenological input is often required. Measuring the total liquid content and global deformation of a wetting/drying material sample is rather straightforward by conventional gravimetric and morphological methods (Gardner et al., 2000; Orteu, 2009). In their early work, Anderson et al. used medical X-ray tomographic device for rapid non-destructive measurement of bulk density and water content of soil samples (Anderson et al., 1988). At least rough local information can be obtained by destructive segmenting of the sample. Non-invasive techniques based e.g. on nuclear magnetic resonance, electric properties of material, and various modalities of tomography have also been used for measuring the local three-dimensional liquid content distribution (Herrmann et al., 2002; Huisman et al., 2003; Mukhlisin et al., 2012; Aregawi et al., 2013) or the local deformation of material samples in various mechanical conditions (Bart-Smith et al., 1998; Peth et al., 2010). Very few efforts appears to have been made towards simultaneous non-destructive measurement of the evolution of both the liquid content and the local deformation field of a material sample during wetting or drying process. Availability of such a measurement method would be potentially very useful for experimental research of processes involving liquid transport and the resulting deformation, and for development and validation of theoretical models of such processes. In this work, we introduce a method based on X-ray microtomography for non-destructive simultaneous measurement of three-dimensional distribution of local water content and displacement field of a wetted material. The method is applied in monitoring the swelling behavior of a wetting bentonite sample.

With X-ray tomography, the spatial distribution of the linear X-ray attenuation coefficient in the sample is obtained (Stock, 2008). The data is conveniently represented as a three-dimensional grayscale image allowing not only visualization but also quantitative study of the internal structure of many heterogeneous materials. The different material components of a multiphase material can be directly observed provided that the typical size scale of the phase domains is larger than the imaging resolution, that the difference between the values of the attenuation coefficient of various phases is large enough, and that the

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phase configuration remains stable during the time of tomographic imaging. In such a case, the relative amount and distribution of various phases can be found in a straightforward manner using 3D image analysis techniques (Wildenschild et al., 2005; Tippkötter et al., 2009; Riedel et al., 2012). Sammartino et al. have used such an approach together with a relatively fast medical X-ray tomographic scanner in order to characterize flow in macropores of soil samples (Sammartino et al., 2012).

However, although we consider here a solid material partially saturated with a water, i.e. a three-phase system of solid, water and gas, we cannot assume phase separation in the size scale given by the resolution of the X-ray tomographic technique available (~1 μm). Instead, each material volume of the size scale given by the imaging resolution, i.e. the image voxel, can contain all three phases that contribute to the total value of the attenuation constant and thus of the grayscale value of the voxel. A single tomographic image of a material sample can thus not provide direct information on the relative abundances of different phases. Such a case was also considered (Sammartino et al., 2012) in evaluating the amount of water contained in soil matrix regions with small-scale porosity in the vicinity of macropores.

In what follows, we neglect the effect of gas phase, and confine ourselves to cases where the attenuation coefficients of the bulk solid and water are the same order of magnitude such that the changes of water content in the solid material are observable with X-ray tomography. We also expect, that the water transport in the material is slow enough such that the phase configuration can be considered approximately stationary during tomographic imaging. (Depending on the technique used, the typical time required for a single X-ray tomographic scan can vary from a few minutes to several days.) Furthermore, we assume that an experimental correlation formula for the dependence of X-ray attenuation coefficient on solid and water contents can be found using some independent calibration method such as gravimetric measurement. In the case that the solid phase content is known, the water content distribution in the partially saturated state may then be found in a straightforward manner by utilizing the X-ray tomographic image. In many practical cases however, change in water content induces considerable solid phase deformation. Then, the local solid content in the sample is not known making it impossible to utilize the calibration data and find the water content based on the measured total attenuation coefficient only. Solution to the problem can be sought provided that an X-ray tomographic image of the same physical sample is taken in a reference state of known solid density. Using image correlation techniques on the images of the reference state and the partially saturated state of interest, called the 'current state' in what follows, the three-dimensional displacement field of the solid phase may be found. Given the reference state solid density, this deformation information can be used to calculate the local solid density distribution in the current state. Together with the total attenuation coefficient data (the X-ray tomographic image), the calibration correlation thus yields the water density distribution in the current state. A prerequisite for successful analysis of the displacement field is that the material contains local structures visible in tomographic images of both the reference state and the current state. In some cases, this may be achieved by doping the material with suitable marker particles.

The primary motivation for the present work has been the need for developing efficient experimental methods for studying groundwater transport and swelling mechanisms of bentonite. This type of clay can absorb large amount of water, swell multiple times of its original volume, and produce large swelling pressure if wetted confined in a closed space. Fully saturated compacted bentonite is considered as effective barrier for transport of water and various chemicals. Due to its unique properties, bentonite is widely used in many applications of soil mechanics as a buffer and sealing material. It is also planned to be used as buffer material in some repository concepts for used nuclear fuel. The long-term purpose of this work is to utilize the X-ray tomographic techniques in a '4D imaging' sense, i.e. monitoring the evolution of water content and deformation of wetting and swelling bentonite samples in three dimensions and as a function of time, thereby producing detailed experimental data for supporting development and validation of hydromechanical models of bentonite.

2. Methods

2.1. X-ray tomography

X-ray imaging is based on attenuation of X-rays in a material. The intensity I of a narrow monochromatic X-ray beam is attenuated in a material according to the Beer–Lambert law (Hubbell and Seltzer, 1996)

$$I = I_0 \cdot e^{-\int \mu(x)dx}, \tag{1}$$

where μ is the linear attenuation coefficient (LAC) which can depend on position x along the beam path. The detector of a typical X-ray tomographic device consists of a phosphorescent screen that converts X-rays to visible light, and a digital camera. A single X-ray projection image of a sample represents the intensity ratio (I/I_0) detected on such a two-dimensional detector. In a typical X-ray tomographic imaging procedure, of the order of one thousand X-ray projection images of the sample are taken from different directions by rotating the sample in the X-ray beam. The three-dimensional distribution of LAC is then reconstructed from the projection images by a computer. The reconstructed data is represented as a three-dimensional image (stack of two-dimensional cross-sectional images) of the sample. The grayscale 'voxel' values in such an image are linearly correlated with the actual LAC value in the sample.

It can be shown that for a given substance the LAC is proportional to its bulk density (Hubbell and Seltzer, 1996). The mass attenuation coefficient, defined as $\mu_m = (\mu/\rho)$, is thus independent of density but depends on X-ray energy and the atomic number of the substance. For compound material of several substances the LAC is given by

$$\mu = \sum_i \left(\mu_{m,i} \cdot \rho_i\right), \tag{2}$$

where $\mu_{m,i}$ and ρ_i are the mass attenuation coefficient and the partial density of substance i, respectively (Hubbell and Seltzer, 1996). The partial density is defined as $\rho_i = \phi_i\tilde{\rho}_i$, where ϕ_i and $\tilde{\rho}_i$ are the volume fraction and the intrinsic material density of the substance, respectively (Soo, 1990).

The radiation source used in current laboratory scale tomographic devices is X-ray tube which produces a polychromatic X-ray beam. Since the attenuation constant depends on energy, the simple Beer–Lambert law, Eq. (1), does not exactly hold. However, in simple reconstruction algorithms, the effects of the wide X-ray energy spectrum are neglected leading to imaging artifacts such as beam hardening (Stock, 2008). Since the attenuation coefficient typically decreases with increasing energy, beam hardening appears in tomographic images as edges of the sample showing virtually more absorbing (denser) than the interior even for a homogeneous sample. For the present technique, beam hardening poses a challenge since it weakens the linear correlation between the grayscale value and the actual attenuation coefficient on which the method is heavily based on. The effects of beam hardening can be reduced by using metallic filters to cut the low energy part of the spectrum. A potential disadvantage of the filtering technique is that the overall intensity of the beam is decreased leading to longer imaging times. The remaining effects of the beam hardening effect can be corrected for in the reconstruction stage (Zou et al., 2011), whereby the approximate linear dependence of the total absorption coefficient and thus of the grayscale value on the partial densities can be retained. Another imaging defects typical to X-ray tomographic techniques include the ring artifact, which can be caused e.g. by a single