The impact of overburden pressure on unsaturated shrinkage behaviour of fine-grained soils

A.C. Smoor
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by

A.C. Smoor

to obtain the degree of Master of Science
at the Delft University of Technology.

Student number: 1528475
Project duration: April, 2015 – December, 2015
Thesis committee: Dr. ir. P. J. Vardon Geo Engineering
Prof. ir. A. F. van Tol Geo Engineering
Dr. ir. L. A. Van Paassen Geo Engineering
Dr. ir. K. J. Bakker Hydraulic Engineering

An electronic version of this thesis is available at http://repository.tudelft.nl/.
Hereby I would very much like to thank everyone who has stood beside me during the process of this MSc research project and helped me, for better or for worse. This help at times has been of vital importance. First of all, I would like to thank the members of my thesis committee for their patience, guidance and constructive feedback. They have kept me on my toes and never failed to ask the annoying little questions that make all the difference. Phil, Leon, Frits and Klaas Jan, I am very grateful for your time and efforts. Secondly, I must express my gratitude to Arno and Han for helping me get started in the unfamiliar territory of the laboratory. My thanks also go out to Ellen and Joost, who in spend many hours helping me scan my samples and process the data. Special mention must be made of Eliza, who ever so patiently helped me set up the hyprop test. I would also like to thank my fellow students for taking the time to discuss an issue or provide much needed distraction. Thanks in particular to Sander and Nadine, who selflessly took measurements in my absence. Thanks also to Bernardien, for accompanying me during the summer at a practically deserted university. Finally, I want to thank my family and Hugo for their wonderful support and unwavering trust that I would make it to this very day. I appreciate it immensely.

A.C. Smoor
Amsterdam, December 2015
Abstract

The impact of overburden pressure on unsaturated shrinkage behaviour of a fine-grained soil is assessed by means of a shrinkage curve based on experimental data. The research is relevant for engineering practices where slurried fine-grained soils are deposited until the material has gained enough strength (e.g. for land reclamation). The deposited material requires containment measures and monitoring due to the high initial water content, slow settlement of fines and low consolidation rate. Extended knowledge on shrinkage behaviour as the material dries helps understand and predict the deposition process.

A promising sludge deposition method is referred to as Atmospheric Fines Drying. To substantiate the method, experimental tests have been performed under atmospheric pressure and a numerical model is developed. In the Sludge Ripening Model by Vardon and Van Tol (2014) a relationship was proposed between shrinkage behaviour of soil and applied overburden pressure. This research is used to verify the existence and extent of this relationship.

To obtain a relevant shrinkage curve, dry oedometer tests and a hyprop test have been performed on a fine-grained material with a high initial water content (near the liquid limit). The samples are subjected to a range of overburden pressures. During the test run, the samples are monitored and analysed by frequent weighings and CT scanning.

Essential for assessing shrinkage is the sample volume (and thereby void ratio) that is taken into account. In this thesis, three definitions for sample volume are used. The first and most general definition accounts for sample volume that changes due to vertical settlement only ($V_{settlement}$). The second definition describes sample volume change due horizontal and vertical shrinkage ($V_{hkv-shrinkage}$). The third volume definition refers to the soil matrix alone, $V_{soil matrix}$. For each of these definitions a separate shrinkage curve type is found. For the shrinkage curves related to $V_{settlement}$ and $V_{hkv-shrinkage}$, a range of solutions is found for the samples subjected to different loads. This indicates the existence of a relationship between minimal void ratio and overburden pressure. The shrinkage curve based on the soil matrix volume is similar for the range of overburden pressures. On soil matrix level, the shrinkage curve may be perceived as unique for a material.

In the aforementioned numerical model, macroscopic soil deposition is simulated. The appropriate volume definitions are ($V_{settlement}$ and $V_{hkv-shrinkage}$). According to the thesis results, it is therefore justified to take into account a minimal void ratio that is dependent on applied overburden pressure. The proposed logarithmic mathematical formulation based on applied pressure and a compression constant can be used to predict the decrease in minimal void ratio.
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1.1. Introduction to the research
In this thesis, the impact of applied overburden pressure on the unsaturated shrinkage behaviour of fine-grained soils is investigated. In this chapter, first the context and relevance for this particular topic are addressed. Subsequently the objectives of the research project are formulated. In the subsequent chapters, the theoretical background to the subject and the approach taken to meet the objective are discussed. In the final section of this chapter, a brief overview of the content of the report is given.

1.2. Context of the research
Knowledge on unsaturated shrinkage behaviour of fine-grained soils is relevant for several disciplines within the engineering practice, varying from dredging activities to land reclamation to mine tailing storage. In these practices, one often deals with slurried fine-grained soils. In fine-grained soft soils, the consistency (e.g. the amount of water) is a major influence on the strength and stiffness of the material. A slurried soft soil has a low shear strength and is unfit for further processing (e.g. land reclamation). To increase the shear strength, the water content and the void ratio of the soil can be reduced. To this end, the material is often temporarily deposited. Due to consolidation, creep and dessication the material dewateres. Depending on the rate of each process, measures (such as costly storage dams) must be taken to contain the material. Often the measures remain necessary for extended periods of time due to the typically low consolidation rate.

Many deposited fine-grained materials undergo volumetric strains of over 50 % due to self-weight consolidation. If surface water is removed and drying is allowed, strains may become even greater (Stark et al. (2005)). This process is referred to as unsaturated shrinkage. The effect of surcharge induced consolidation (e.g. by placement of a less compressible cohesionless soil cover) on the settlement process needs to be assessed. An increased understanding of the development in strength and volume of the deposited material helps to deal with the material appropriately and may lead to the technologies to accelerate the process.

A typical practice where research on unsaturated behaviour of fine-grained soils is relevant, is the exploitation of the Canadian oil sand depositions and the subsequent storage of the tailings. In the next section, this example is expanded on.

1.2.1. Oil sand tailing storage
In Canada large deposits of tarsand, or oil sands, are found and exploited. Oil sands are a mixed deposit of sand, water, clay, bitumen and other minerals. The main oil sand reserves are found in the province of Alberta, Canada. In this province an area of 142,300 km² is underlain with oil sand deposits.

As it is, only a limited portion of the deposition is accessible for open mining, the surplus is exploited using in situ methods. For this research, the open pit mining method is most relevant, for this method generates large tailing ponds and consequential challenges.

After the oil sand ore is mined, the material is processed in order to obtain crude oil. Bitumen is too viscous to flow unless heated or diluted, which is why large quantities of hot water are added. The residual by-products...
of processing oil sand ore are referred to as tailings. Tailings consist of a mixture of sand, clay, water, heavy metals and residual bitumen. The tailings are stored in large ponds until the material meets the set requirements for reclamation and further permanent storage (Shell Canada (2013)).

Various companies exploit the oil sands in Alberta Canada, among which is global energy company Shell. In 2012 1.9 million barrels per day were produced of Alberta's oil sands. According to the Alberta government in 2013 approximately 180 km$^2$ of land were covered with tailing ponds (Alberta Government (2013)). The tailings are stored in large ponds. Over time the material settles and the water can be recycled in the production process. Eventually also the occupied land can be reclaimed. The settlement process, due to the high quantity of 'fines', silt and clay particles, in the material, can take up to 25 years. Over the past 5 years, increasingly research is performed on whether this process can be accelerated (Shell Canada (2013)). In figure 1.1 the process of extracting bitumen from the oil sand ore is illustrated. Large quantities of tailings must be stored in order to gain a unity of product.

![Diagram of the oil sand exploitation process and tailing storage (after BGC Engineering inc. (2010))](image)

As the by-product is deposited in a tailing pond, larger particles settle relatively quickly. The product segregates and a layer of stagnant water is formed on top (figure 1.1). The water can be reused in the extraction process. What remains is a suspension with a high water content c.q. low solids content, Mature Fine Tailings (MFT) (Yao et al. (2012)).

The substance of mature fine tailings is an inconvenient by-product in multiple aspects, such as toxicity caused by residual bitumen and high water content and therefore large volume. The excessive drying time augments the impact of the negative material properties. The problem concerning MFT can thus be summarized:

Due to the high water content and large settlement time of the material,...

- ... a large area must be reserved for tailing ponds. This land must be deforested and can not be reclaimed for as long as the MFT strength is below the set norm.
- ... a delay exists in the water recycle process and fresh water is used for the extraction process, increasing the risk of pollution.
- ... large quantities of the toxic tailings are exposed to the environment. Consequences include groundwater pollution and risk for waterfowl.
- ... large quantities of a material with shear low strength (before consolidation) must be stored in a stable situation, requiring (geo)technical structures.
1.3. Deposition of tailings

In the previous section, the context and relevance of the thesis objective are illustrated by the example of the mature fine tailings storage. Typical problems and challenges related to the deposition of soft soils and tailings have been addressed. The issue of tailings storage has generated many research initiatives both by industry and the academic world. In this section, relevant findings are presented.

1.3.1. Deposition methods

BGC engineering published a report in 2010 in an attempt to identify the largest challenges and to map possible methods of improving dewatering time and reducing volume. According to BGC, the water holding capacity of the MFT and its large consolidation time are accounted for by the surface properties of the clay minerals such as negative charge promoting dispersion of particles and interparticle forces. As a result, MFT have a mean particle size between 5 and 10 \( \mu \)m, a hydraulic conductivity in the range of \( 10^{-6} \) to \( 10^{-9} \) m/s, an average void ratio of 5 and a shear strength (much) less than 1 kPa (causing it to act like a fluid) (BGC Engineering inc. (2010)). Expanding the available knowledge on MFT characteristics will speed up the process of adequately solving the slurry disposal problem.

BGC employs a division to categorize the presented methods. The following treatment technologies are recognized: mechanical processes, natural processes, chemical processes, mixtures and permanent storage of MFT. At the time, they concluded that best results should be obtained by combining elements from multiple categories in a complex solution (BGC Engineering inc. (2010)).

1.3.2. Thin lift deposition

A technology that is the object of interest for further research for Shell Canada and Delft University of Technology is referred to as Atmospheric Fines Drying (AFD). AFD is an example of a complex solution, in which chemical, (soil) mechanical and natural processes are combined. A polymer flocculent is used to bind the MFT particles. Subsequently, the material is dried in thin lifts under atmospheric conditions. The deposed layer is subjected to drying and rewetting cycles due to precipitation and the addition of a new layer as the former has dried. The process has been simulated in both numerical work and experimental testing and AFD is currently implemented on commercial scale by Shell Canada. (Vardon et al. (2014))

Optimised Seasonal Deposition (OSD) is comparable to AFD, but takes into account the local conditions (in this case for Alberta, Canada) and proposes an optimised system of tailing lift deposition. The main spring deposition dewaters due to drying, cracking and consolidation. The fall toplayer gains strength due to the process of freeze and thaw. In figure 1.2 tailing deposition using thin lifts is illustrated.

![Figure 1.2: Flocculated MFT is deposited in thin layers, in which (a.o) self-weight consolidation and desiccation take place (after Caldwell et al. (2014))](image_url)

Caldwell et al. state that the OSD concept is a means for depositing a larger quantity of processed MFT on a fixed area (Caldwell et al. (2014)). The objectives for the method can thus be formulated as strength regain and volume reduction. As the material dewatered, a certain volume reduction is established. During consolidation of a soil, water is driven out. During the desiccation process, water evaporates from the material. It eventually becomes un-
saturated and suction develops due to negative pore water pressure, causing the material to shrink until a minimum void ratio is reached.

1.3.3. Previous research at Delft University of Technology
Quite some work has been done in order to understand and predict the characteristics and behaviour of flocculated MFT in the context of atmospheric fine drying. Properties of the material were determined experimentally. Shrinkage and swelling behaviour was evaluated in terms of void ratio change under atmospheric conditions. This behaviour is captured in the Soil Shrinkage Characteristics Curve (SSCC) (Yao et al. (2014)). A numerical model was developed to simulate the sub-aerial drying process in order to proof that AFD is a adequate method of depositing oil sand tailings. In this model, the Sludge Ripening Model, volume change behaviour during desiccation and rewetting is taken into account, as well as self-weight consolidation (Vardon et al. (2014)).

1.3.4. Influence of overburden pressure on shrinkage behaviour
As the flocculated MFT is deposited in thin lifts, multiple processes may be at work simultaneously. The desiccation process can start whilst the consolidation process has not yet come to an end. In the numerical work developed by Vardon and Van Tol (2014) a relationship has been proposed on the impact of these combined processes on the shrinkage (i.e. decrease of void ratio to a minimal value) of the material, which governs the minimal volume of the deposition. In order to understand and predict the settlement behaviour of the deposited material more accurately, additional research must be done on the relationship of overburden pressure and unsaturated shrinkage.

1.4. Positioning the research
In this thesis, the work performed at Delft University of Technology on the volume change behaviour of (f)MFT during Atmospheric fine drying is continued. In the previous sections, the motivation for assessing the impact of overburden pressure on unsaturated shrinkage is addressed. Performing the assessment is the main objective in this research project.

1.4.1. Context and motivation
In the engineering practice, deposition of fine-grained soft soils (such as oil sand tailings) causes a technical challenge due to its large water content, low consolidation rate and chemical composition. Steps have been taken to predict accurately the settlement behaviour of deposited material (e.g. the sludge ripening model has been developed). The question is raised whether the application of overburden pressure influences the volume change behaviour of deposited slurries due to the contribution of consolidation in combination with unsaturated shrinkage.

1.4.2. Main Research Question
Does applied overburden pressure impact unsaturated shrinkage behaviour of a fine-grained soil with a high initial water content? If so, what is the impact and how can the impact be quantified?

1.4.3. Hypothesis
The application of the overburden pressure does influence the unsaturated shrinkage behaviour of a fine-grained soil as captured in a shrinkage curve. Due to the elevated pressure, the material is further consolidated, delaying the air entry point. Due to further compaction (relative to atmospheric pressure), the minimal void ratio ($e_{\text{min}}$) (governing the shrinkage curve) will decrease. The impact can be quantified by means of experimental tests and numerical modelling (e.g. sludge ripening model).

1.4.4. Additional remarks
In this research, the main material used is not MFT, but a somewhat similar yet more general slurried clay. This is done to ensure the reproducibility and therefore scientific value of the research. Also, the use of MFT would limit the available experimental testing equipment, due to the residual bitumen in the composition.
1.5. Content of the report

In the previous section, the main research question and hypothesis are defined. In the upcoming chapters, the research, performed in order to assess whether the hypothesis is correct, is presented.

In chapter 2 a theoretical background for the main research is given. After a brief introduction of basic soil theory (e.g. the definition of soil phases), the focus of the chapter will lay on the aspired change in volume such as has been put forward in section 1.3.4. Main drivers for volume change are identified and different types of behaviour are elaborated. The information provided will be called upon to support steps taken in the research process and interpret and verify results.

In the subsequent chapter 3, the methodology utilized in the research is discussed. Considerations and steps taken for both the experimental and the numerical parts of the research listed, as well as the motivation with respect to the hypothesis.

The obtained results are presented in chapter 4. Verification is essential for correct interpretation of the results. From the process of validation, more clarity should be obtained on the actual significance of the research results.

In the final chapters, the results are discussed in a critical manner and conclusions may be drawn. In the final stage of the research, recommendations related to the process and results can be made for potential future research.
2.1. Introduction
In the previous chapter, the motivation and objectives of this research were elaborated. The practical issue of oil sand tailing storage is taken as a start off point for a more fundamental research on the impact of elevated pressure on void ratio change during the consolidation and drying of a fine grained soil. A brief literature review is presented here on relevant soil characteristics and engineering mechanics that may help understand the research question and either challenge or strengthen the formulated hypothesis. In short, this chapter shall function as a theoretical basis for the research.

2.2. Phases in a soil
A soil is not a solid mass but a mixture of elements. These element-groups are commonly addressed as phases of a soil. In this section, a short review of the various soil phases and related basic properties is done. In nature one generally encounters a solid phase, a water phase and an air phase. A fourth phase can be identified as the contractile phase, or the air water interface (Fredlund and Rahardjo (1993)). In figure 2.1 two phases diagrams are given and explained in more detail below.

2.2.1. Solid phase
A soil contains soil particles. The combined volume, or mass, of these particles in a soil constitute the solid phase. The mineralogy, size, shape, diversity and structure of the particle(group)s influence strongly the engineering properties, and thereby the mechanical behaviour, of the material. The compressibility of (inorganic) soil particles is often considered negligible (relative to the stress range). A volume change in the soil phase therefore cannot occur. Rearrangements of soil particles may result in a change in the volume of the water and/or the air phase.

2.2.2. Water and air phases
In between the soil particles voids or pores are found. The size, shape and content of these pores are relevant for the material behaviour. The size and shape are dependent on structure, stress (history) and properties of the soil particles. The pore content defines the classification the soil as dry (all pores are filled with air), saturated (all pores are filled with water) or unsaturated (pores are filled with both air and water). Each of these soil types show different behavioural responses to external stimuli.

2.2.3. Air water interface
The thickness of the interface is in the order of a few molecular layers and can therefore be neglected when looking into the volume-mass relationship of a soil. The air water interface does have an important property, surface tension $T_s$, which results from intermolecule forces acting on molecules in the contractile skin. In order to be in equilibrium, a tensile pull is generated along the interface. The surface tension causes the interface to behave like an curved elastic membrane with a radius $R_s$. In an unsaturated soil, the air water interface is subjected to a pressure difference, for the pore air pressure
2. Literature review

Figure 2.1: Phase diagrams for an unsaturated soil. In the second diagram, the fourth phase is added. Mostly, the first, simplified, diagram is used (Fredlund and Rahardjo (1993)).

is larger than the pore water pressure. The pressure difference \( u_a - u_w \) is referred to as matric suction. The relationship between matric suction and surface tension is given in equation 2.1, which is referred to as Kelvin's capillary model equation (Fredlund and Rahardjo (1993)).

\[
(u_a - u_w) = \frac{2 T_s}{R_s}
\]  

(2.1)

where

- \( u_a \) pore air pressure
- \( u_w \) pore water pressure
- \( u_a - u_w \) matric suction
- \( T_s \) surface tension
- \( R_s \) radius to curved membrane (interface)

In section 2.3.3, the relevance of surface tension and capillarity for this research will be further discussed.

2.2.4. Volume mass relation

Volume mass relations are relevant properties in engineering practice. Environmental properties such as porosity, void ratio, degree of saturation, water content and density are used frequently to describe the relationship between and ratio of phases in a soil and help clarify and predict soil behaviour. The basic volume mass relationship is grasped in the formula 2.2 for the total density of a soil. If the mass of air is assumed negligible the formula applies for both saturated and unsaturated soils.

\[
\rho = \frac{M_{\text{soil}} + M_{\text{water}}}{V_{\text{soil}} + V_{\text{voids}}}
\]  

(2.2)

2.2.5. Phase properties

In order to interpret and predict soil behaviour, knowledge of engineering soil properties is essential. These properties depend on the composite effects of two factors; compositional factors and environmental factors. The first factor determines the potential range of values for a property. Examples are the type and amount of minerals, shape and size distributions of the particles, pore water composition and other possible constituents in the soil. The second factor determines the actual value of a property. Examples are water content, (bulk)density, confining pressure, temperature, fabric and availability of water. In appendix A the subjects of soil mineralogy and fabric are further addressed.

Behaviour of cohesionless soil is often determined by applied confining pressure and the void ratio in relation to the maximum or minimum value. A cohesive soil is characterized by values of stiffness and strength and properties defined as the Atterberg limits.

2.3. Description of the stress state

The mechanical behaviour of a soil, such as volume change behaviour and (changing) shear strength can be captured in constitutive relations. As this research is mainly a study on resulting volume change, the former
is our soil response of interest. Volume changes in a soil can be induced by changes in applied stresses, chemical and moisture environments, and temperature. The effects of stress state change are generally most important and have been most studied (Mitchell and Soga (2005)). Therefore, often the constitutive relation for volume change relates deformation state variables to stress state variables (Fredlund and Rahardjo (1993)).

2.3.1. Terzaghi’s principle of effective stress

The state of stress in a soil is brought about by various combinations of stress variables. The stress state variable should be independent of physical soil properties. The number of stress variables necessary to describe the stress state depends on the number of phases involved (Fredlund and Rahardjo (1993)). Simply put, the total stress state is a function of the stresses and forces within each of the soil phases. Karl Terzaghi first developed a stress state variable with which volume change behaviour and shear strength behaviour of a saturated soil can be described (Terzaghi (1936)). The single-valued stress state variable, effective stress, is captured in the equation 2.3. The principle of effective stress has become the keystone in modern soil mechanics.

\[
\sigma' = \sigma - u_w
\]  

(2.3)

where \(\sigma'\) effective normal stress  
\(\sigma\) total normal stress  
\(u_w\) pore water pressure

In Terzaghi’s view, forces in a soil propagate though the soil skeleton, from one grain to another. Pore pressure \(u_w\) is considered a neutral stress, isotropic, invariant in direction and without a shear component, and acts over the entire surface of the soil grain. Assessments of Terzaghi’s work led to the conclusion that equation 2.3 does not capture true effective stress, but, if soil grain compressibility is assumed negligible, is useful for assessing the soil response during changing stress conditions in engineering applications (Skempton (1960)).

2.3.2. Influence of interparticle forces

The term intergranular stress is often considered equal to Terzaghi’s macroscopic principle of effective stress. Equation 2.3 however does not capture all the forces at work in a soil mass. Microscopic interparticle forces alter the soil response related to deformation and strength and should be evaluated for their contribution in the total stress state.

At a microscale, interparticle stresses can be separated in three categories (Santamarina (2003)):

1. **Skeletal forces due to external loading**, transmitted through particles.

2. **Particle level forces**, such as weight forces, buoyancy force, hydrodynamic and seepage forces. These forces serve as counterbalance for skeletal and contact level forces (Lu and Likos (2006)).

3. **Contact level force**, e.g. physicochemical forces such as electrical forces and van der Waals forces, and additional attractive forces such as surface tension forces, capillary forces and cementation forces.

Interparticle forces can be both repulsive and attractive forces. In a soil mass at equilibrium, a balance exists among all interparticle forces, water and air pressure and applied stresses. Within a saturated soil, the first two interparticle force categories often suffice to describe the behaviour since the material can be treated as an equivalent continuum medium with macroscopic stresses defined at the boundaries (Lu and Likos (2006)). When a soil desaturates, the pore pressure no longer functions as a macroscopic stress but disintegrates into three forces acting both through the water and the air phase in the system. Air pressure is acting on the dry or hydrated grain surfaces, water pressure acts on the wetted portions in menisci formed near the interparticle contact points and surface tension develops along the air water interfaces. The distribution of these forces alters with changes in the granular fabric of the material and the degree of saturation. (Lu and Likos (2006))

In the next section, the development of capillary forces is discussed in more detail.
2.3.3. Capillarity

Within a multi-phase system, one can speak of a wetting and a non-wetting fluid. Wettability refers to the attraction of a fluid to a solid surface in presence of another fluid and can be measured using contact angle \( \theta \). The interface between the fluids reaches the solid surface with a certain angle. If this angle is less than 90 degrees, the reference fluid is the wetting fluid and vice versa. Within an unsaturated soil, water and air are the wetting and non-wetting fluid, respectively.

In section 2.2.3 mention was made of a surface tension \( T_s \) that exists in the air water interface. As a result of the surface tension and the attraction of water (being the wetting fluid) to the soil particle surface, a suction develops in the pores during desaturation. Matric suction causes porous media to draw in the wetting fluid and repel the non-wetting fluid - hence the negative pore pressure - and is defined as the difference between the non-wetting fluid pressure and the wetting fluid pressure (Mitchell and Soga (2005)). In equation 2.1 the relationship between the matric suction and \( T_s \) was already given.

A soil pore can be perceived as a capillary tube as shown in figure 2.2. The capillary stress in the pore can be estimated with equation 2.4.

\[
P_c = \rho_w g d_c = \frac{2T_s \cos \theta}{R_p}
\]

where

- \( P_c \) capillary pressure
- \( T_s \) air water interfacial tension
- \( R_p \) pore radius

Figure 2.2: The capillary tube concept can be applied for soil pores (after Mitchell and Soga (2005)).

Capillary forces affect strength and deformation behaviour. Due to negative pore pressures, a temporary cohesion and strength increase is witnessed in a soil during drying.

The soil water characteristic curve

A soil type has a particular range of pore sizes. To replace pore water with air, a capillary pressure inversely proportional to the pore size is necessary. For a certain soil, the relationship between the remaining water content and the capillary pressure (or suction) build up is referred to as the soil water characteristic relationship.

In figure 2.3 such a relationship is represented. The soil system starts out as a saturated mass. Only after the matric suction has matched the air entry pressure, the pore water is displaced by air. The air entry pressure needed is dependent on the pore size; in a fine-grained soil the pressure must be larger than in a coarse grained soil. It must be noted that figure 2.3 does not give information on the stress conditions at the time of drainage/drying. The particular relationship may be characteristic to a soil under atmospheric conditions, but change at a different stress range. Also, the figure suggests that the mass remains saturated at \( \theta_s \) until the suction causes the pores to drain. Before the air entry value is reached, however, the de-saturation process may have started due to evaporation (depending on the conditions).

During the initial de-saturation stage, soil particles remain continuously surrounded by water, which is referred to as the funicular regime. The fluid acts like a membrane of negative pressure as discontinuous air
2.3. Description of the stress state

Pores develop. At a certain point, the water phase is disconnected and is left to form small water bridges at the interparticle contacts. The magnitude of the capillary force is now dependent on the size of the water bridge and the distance between the soil particles. The influence of the soil fabric on the contact level force distribution becomes more pronounced. This stage is referred to as the pendular regime. The curved air-water interface generates a pore water tension, which translates to an interparticle compressive force at the contact points (Mitchell and Soga (2005)).

During the pendular regime, evaporation becomes the primary mechanism of water transport (out of the soil).

In figure 2.3, \( \theta_r \) signifies the residual volumetric water content. At this point, the water content can no longer decrease by an increase of matric suction. Since the actual degree of saturation of the material can decrease after this point (depending on the conditions for further evaporation), \( \theta_r \) is foremost a mathematical parameter (Mitchell and Soga (2005)).

![Figure 2.3: A conceptual illustration of the soil water characteristic relationship for a fine-grained soil (after Lu and Likos (2006)). The stress conditions of the material are not taken into account.](image)

2.3.4. The stress state in unsaturated conditions

A principle constitutive relationship of volume change in saturated soils can be captured in void ratio versus effective stress. In the case of an unsaturated soil, the description of the behaviour by means of effective stress becomes more complex. The altered air-water relationship must be captured in a stress state variable. In Figure 2.4 a set of constitutive curves for the unsaturated situation is shown. In the case of a constant total stress, the behaviour represented by the shaded constitutive surface is in fact the soil water characteristic curve (Figure 2.4 (b)) (Barbour (1998)).

![Figure 2.4: Constitutive relationships for unsaturated soils: (a) void ratio and (b) water content or degree of saturation versus total stress and matric suction (after Fredlund and Morgenstern (1976)).](image)

The choice of stress variables and incorporation of contact-level forces in a macroscopic effective stress equation remain to be fully evaluated (Mitchell and Soga (2005)).
A well-known macroscopic approach to describe an unsaturated (i.e. three phase) soil is that of Bishop, which is presented as a modified form of Terzaghi’s definition (Bishop (1959)):

\[ \sigma' = \sigma - u_a + \chi (u_a - u_w) \]  

(2.5)

where \( u_a \) — pore air pressure 
\( u_w \) — pore water pressure 
\( \chi \) — ‘effective stress’ parameter 
\( u_a - u_w \) — matric suction

In the above equation the concept of effective stress is extended to unsaturated soil. Effective stress is composed of the net total stress \( \sigma - u_a \) and some contribution of matric suction. As \( u_w \) is negative, the effective stress increases, which corresponds to earlier finding that negative pore pressures increase strength and decrease compressibility (Mitchell and Soga (2005)).

The parameter \( \chi \) varies between zero and unity and depends on degree of saturation, soil type and hysteresis effects due to wetting, drying and stress change (Bishop (1959)). Bishop’s work provided an important step towards understanding the relationship between shear strength behaviour and the soil water characteristic curve (dependent on S) (Barbour (1998)).

The introduction of the empirical \( \chi \) parameter has generated much debate on the description of deformation behaviour. From their experimental work, Jennings and Burland (1962) concluded that no unique relationship between volume change (void ratio) and effective stress was found for most soils, especially below a critical degree of saturation (as high as 85-90 % for clays).

More recently, better results have been obtained for the use of \( \chi \) when applied for fine-grained soft soils.

An alternative approach is that of the use of independent stress state variables (Fredlund and Morgenstern (1977), Fredlund (1985), Fredlund and Rahardjo (1993)). The roles of normal stress and matric suction in the mechanical behaviour or unsaturated soil are treated independently. The behaviour is described using these independent stress state variables in combination with conjugate material properties. Fredlund (1985) defined equation 2.6 with which the changing void ratio \( \Delta \varepsilon \) of an unsaturated soil could be determined.

\[ \Delta \sigma = a_t \Delta (\sigma - u_a) + a_m \Delta (u_a - u_w) \]  

(2.6)

\( a_t \) and \( a_m \) are coefficients of compressibility of respectively the changes in total stress and changes in capillary pressure.

The concept of two stress variables (and their modifications) has led to the development of elasto-plastic-based constitutive models (such as the famous Barcelona Basic Model) to describe stress-strain behaviour of partially saturated soils (e.g. Alonso et al. (1990)).

An example of such a model is the approach presented by Sheng et al. (2008). The model employs two independent variables: (the) net stress (vector) and suction, with their conjugate strains, soil skeleton strain and volumetric water content, respectively. The model aims to describe volume change, yield stress and shear strength behaviour as functions of suction. These functions are all based on equation (2.7) for volume change (i.e. volumetric strain) due to stress changes and suction under isotropic conditions.

\[ d \varepsilon_v = - \frac{d \nu}{\nu} = \lambda_{vp} \frac{d \bar{p}}{\bar{p} + s} + \lambda_{vs} \frac{d s}{\bar{p} + s} \]  

(2.7)

where \( d \varepsilon_v \) — rate of volumetric strain 
\( \nu \) — specific volume \((1 + e)\) 
\( \bar{p} \) — mean net stress \((p - u_a)\) 
\( s \) — matric suction \((u_a - u_w)\) 
\( \lambda_{vp} \) — slope of normal compression line 
\( \lambda_{vs} \) — slope for unsaturated case, gradually decreases to zero
The choice of stress variables, such as proposed in the aforementioned approaches is still under debate (Mitchell and Soga (2005)).

A third approach is generally referred to as the modified stress variables approach. Several authors over the years have tried to incorporate directional dependency, intergranular surface tension (Matyas and Radhakrishna (1968)), volume strain due to matric suction and degree of saturation in the formulation of stress variables.

2.3.5. Suction stress

Some authors argue that a complete expression for effective stress should incorporate both macroscopic forces and microscopic forces. Lu and Likos (2006) propose to combine all third category interparticle forces, as defined in section 2.3.2, into a macroscopic stress referred to as 'suction stress' in order to come to a description of the stress state.

Lu and Likos base their approach on the formulation of an equilibrium of forces on a fine-grained particle system. Figure 2.5 represents a general shape of soil particles in an unsaturated system, in which forces from all three categories act on wavy cross section A.

\[
\sigma_c = \sigma_t - u_a + (\sigma_{c0} + \Delta \sigma_{pc}) + \sigma_{cap} + (u_a - u_w)(1 - \frac{A_a}{A})
\]

where

- \( \sigma_t \): Stresses due to skeletal forces
- \( \sigma_{c0} + \Delta \sigma_{pc} \): Stress due to physicochemical forces, divided into reference stress in the saturated situation and the change in physicochemical stress during desaturation
- \( \sigma_{cap} \): Capillary stress

The last term on the right-hand-side can be seen as Bishop's parameter \( \chi \), yielding equation 2.9. Bishop indirectly captured in his equation 2.5 the role of local stresses on the soil behaviour but did not explicitly account for (a change in) physicochemical or capillary mechanisms.

\[
\sigma_c = \sigma_t - u_a + (\sigma_{c0} + \Delta \sigma_{pc}) + \sigma_{cap} + \chi(u_a - u_w)
\]

The concept of suction stress \( \sigma_s' \) is introduced as the resultant of all the type three interparticle stresses that act during the desaturation process. Equation 2.10 then becomes:

\[
\sigma_c = \sigma_t - u_a + \sigma_{c0} + \sigma_s'
\]

The final equation reminds one of Terzaghi's formulation of the principle of effective stress, in which the changing state of interparticle stress during desaturation is included.
2.3.6. Concluding comments

In this section, some considerations for the formulation of stress as a driver for soil behaviour are discussed. Knowledge of stresses and the factors that influence them is essential in understanding and quantification of constitutive relations. The stress state of a saturated material can be adequately captured with the effective stress principle. This formulation needs modification as the soil desaturates and pore water suction and physicochemical forces (especially in a clayey soil) gain influence. The assessment of the unsaturated stress state in a macroscopic equation is complicated by difficulty in choice of stress variables and in the exact positioning and treatment of contact-level forces.

2.4. Volume change behaviour

As mentioned in the introduction of the previous section, volume change is the soil behaviour of main concern in this research. A main driver for volume change is a change in the stress state. In the previous section, various approaches to describe the stress state as a function of effective or intergranular stress have been discussed. It was established that changing conditions, such as desaturation of a material, affect the stress state. In this section, a review is made of how such changing conditions can impact the volume of soil.

Volume change in a soil is influenced by compositional and environmental factors. Changes in stress was already identified as a factor. Particle size and shape strongly influence void ratio at any pressure and the effects of physicochemical and mechanical forces on volume change (Meade (1964)). Physical interaction of particles during deformation may contribute to (the resistance to) volume change. Relevant environmental factors are temperature and the chemical or organic environment. The stress history - under- or overconsolidation - may also effect volume change behaviour. (Mitchell and Soga (2005))

As a soil volume changes, particles are rearranged due to shear, sliding and crushing of particles or disruption of aggregates. The arrangement of particles and particle groups and the forces holding them in place are therefore of influence. Volume change can be identified as compression, shrinkage, swelling or collapse due to a effective stress reduction. In the next sections, the relevant volume change types are elaborated.

2.4.1. Compression

Compression in a soil is the result of transmitted normal forces between particles. A deformation of the soil takes place in which the volume changes but the shape of the soil mass remains (more or less) intact. The extent of compressibility of a certain soil is captured in the compressibility index, \( C_c \). Pure compression is a situation in which deformation in each direction is equal (e.g. in the case of isotropic loading). It can be said that due to compression void ratio deduces, and as the soil compacts, the stiffness increases. As opposed to normal forces, pure (external) shear forces cause a change in the relative arrangement of particles at a constant volume.

In laboratories, compression is often reduced to a one dimensional compression test, in which volumetric strains in horizontal direction are prohibited by a rigid ring. Vertical loading is applied on a porous top plate, allowing drainage of the pores in the sample. Volumetric strain in vertical direction can be approximated by the logarithmic compression formula 2.11, where dimensionless parameter \( C_{10} \) is a compression constant and \( \sigma_0 \) is the initial stress level. In table 2.1 average values for the compression constant are given (Verruijt (2012)).

\[
\epsilon_v = -\frac{1}{C_{10}} \log\left(\frac{\sigma}{\sigma_0}\right)
\]

<table>
<thead>
<tr>
<th>Type of soil</th>
<th>( C_{10} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>sand</td>
<td>20 - 200</td>
</tr>
<tr>
<td>silt</td>
<td>10-50</td>
</tr>
<tr>
<td>clay</td>
<td>4 - 40</td>
</tr>
<tr>
<td>peat</td>
<td>1 - 10</td>
</tr>
</tbody>
</table>

Table 2.1: Average values for compression constant \( C_{10} \) (Verruijt (2012))
Consolidation

Equation 2.11 suggests that soil deformation is mainly determined by stress. In reality, volume change behaviour of especially fine grained soil is also dependent. The rate of volume decrease is controlled by the time required for water to flow out of the pressurized soil. This process is referred to as consolidation.

The change in void ratio, which in a saturated case is a result of either compression of the pore water or drainage of the pore water, can be rewritten as a function of change in pore water pressure over time. If hydraulic conductivity is assumed constant over z (i.e. assuming vertical flow only) and the load (σ) is kept constant after application, the following diffusion equation describes a basic one dimensional consolidation process (Mitchell and Soga (2005), Verruijt (2012)). Further on in this section, the validity of a constant value of hydraulic conductivity is discussed.

\[ \frac{\delta u_w}{\delta t} = c_v \frac{\delta^2 u_w}{\delta z^2} \]  
(2.12)

\[ c_v = \frac{k}{\gamma_w (m_v + n \beta)} \]  
(2.13)

where

- \( u_w \): excess pore water pressure
- \( t \): time
- \( c_v \): coefficient of consolidation
- \( z \): distance to a drainage surface
- \( k \): hydraulic conductivity
- \( \gamma_w \): unit weight of water
- \( m_v \): coefficient of compressibility \( m_v = \epsilon / \sigma' \)
- \( n \): porosity
- \( \beta \): compressibility of water

From equation 2.13 can be concluded that a larger stress \( \sigma' \) gives a smaller \( m_v \) and thereby a larger coefficient of consolidation. This gives a larger change in pore water pressure over time (i.e. a larger consolidation effect).

The consolidation process takes infinitely long to complete. In engineering practice, it suffices to assume the consolidation process completed when 99% of the deformation has occurred. This moment is referenced to as \( t_{99} \) and is a function of the coefficient of consolidation and maximum distance to a drainage surface.

\[ t_{99} = c_v \frac{t}{k^2} \]  
(2.14)

The coefficient of consolidation of a material can be determined with a consolidation test c.q. 1D compression test. To reach a degree of consolidation \( U = 50\% \), it takes a certain amount of time \( t_{50} \). The value of \( c_v \) can then be calculated using equation 2.15. (Verruijt (2012))

\[ c_v = 0.197 \frac{h^2}{t_{50}} \]  
(2.15)

The theory discussed is a simplification of the actual soil behaviour, that may deviate from the theory due to changing conditions, specific soil characteristics and assumptions in modelling.

Hydraulic conductivity

Hydraulic conductivity is a measure of liquid flow through a soil and is dependent on the viscosity of the liquid \( \mu \) and the permeability of the material. An expression is given (Verruijt (2012)):

\[ k = \frac{\kappa \gamma_w}{\mu} \]  
(2.16)

The formula of Kozeny-Carman (equation 2.17) gives a reasonable relation for intrinsic permeability \( \kappa \).

\[ \kappa = c d^2 \frac{n^3}{(1-n)^2} \]  
(2.17)
where \( c \) coefficient depending on tortuosity of soil pore system
\( d \) measure or grain size
\( n \) porosity

From equations 2.16 and 2.17 the dependency of \( k \) on pore size, shape and distribution becomes clear. These may change over time. In diffusion equation 2.13 the hydraulic conductivity is assumed constant over \( z \), simplifying the math. This is allowed when a general approximation of 1D consolidation in saturated conditions is performed. As a result of consolidation, the pore size and distribution eventually change, affecting the hydraulic conductivity. For a more accurate assessment of consolidation behaviour, this assumption is no longer valid.

Consolidation in unsaturated soil
In a de-saturating soil, hydraulic conductivity \( k \) also becomes dependent on the amount and connectivity of water in the soil pores.

The hydraulic conductivity can be a function of degree of saturation, water content and matric suction. A general expression for hydraulic conductivity in unsaturated soil can be written as the saturated conductivity \( k_s \) in combination with relative permeability \( k_r \) (i.e. equation 2.18).

\[
k = k_r \frac{\rho g}{\mu} = k_r k_s
\]

(2.18)

where \( \kappa \) intrinsic permeability \( L^2 \)
\( \rho \) density of permeating fluid \( ML^{-3} \)
\( \mu \) viscosity of permeating fluid \( MT^{-1}L^{-1} \)

Dimensionless parameter relative permeability \( k_r \) ranges from 0 (zero permeability) to 1 (permeating fluid at full saturation). Brooks and Corey (1964) define a formulation for \( k_r \) for the wetting phase as follows:

\[
k_r = (S_e / S_r)^\lambda = (S_e)^\lambda
\]

(2.19)

where \( S_e \) effective saturation \( S_e = (S - S_r) / (1 - S_r) \)
\( S_r \) residual saturation
\( \lambda \) material constant that characterizes pore-size distribution

Fredlund et al. (1994) stress the importance of suction in an unsaturated system. They propose to determine the residual water content \( \theta_r \) (as discussed in section 2.3.3) from experimental data and then to fit the SWCC data to a mathematical formulation. Determining the SWCC in a laboratory is easier then measuring the hydraulic conductivity directly. \( k_r \) as a function of water content can then be computed using equation 2.20.

\[
k_r(\theta) = \frac{\theta - \theta_r}{\psi^2(\theta)} \int_{\theta_r}^{\theta} \frac{\theta - x}{\psi^2(x)} dx
\]

(2.20)

where \( \theta \) volumetric water content
\( \psi \) suction

In either formula, the relative permeability decreases as the water content or the degree of saturation decreases. This means that the hydraulic conductivity for unsaturated soil decreases with the further dessication of the material. According to equation 2.13 also the coefficient of consolidation decreases. The build-up of pore pressure due to consolidation decreases (as defined in equation 2.12) and one could argue that the consolidation process takes longer based on equation 2.15.
2.4.2. Shrinkage

In section 2.3.3 the development of pore water tension by capillary menisci during desaturation of a soil is discussed. As a fine grained soil dries, the tension causes a rearrangement of particles that is referred to as shrinkage. The extend of shrinkage depends on the size of the capillary stresses and the soil fabric.

Cracking during drying

Drying occurs from a (horizontal) surface, where the evaporation takes place. The (matric) suction, that develops as a result of air entry in the soil voids, produces two counteracting effects. Firstly, at any point in the soil, a more or less isotropic contraction takes place (given that the water phase is still continuous). Cracks form parallel to the drying surface. Secondly, the soil gains strength due to the negative pore water pressures and therefore the resistance to further cracking increases (Morris et al. (1992)).

Shrinkage curve

Shrinkage behaviour during drying can be captured in a Soil Shrinkage Characteristic Curve (SSCC), in which the volumetric change (i.e. decreasing void ratio) and the dessication process (i.e. decreasing water content) are plotted (e.g. figure 2.6). This curve is assumed to be unique for a material.

![Shrinkage Characteristic Curve (SSCC)](image)

Figure 2.6: Drying curve (SSCC) of an initially slurried soil sample (after Fredlund et al., 2002a).

Within the shrinkage process four stages can be distinguished; 1) structural shrinkage, 2) normal shrinkage 3) residual shrinkage and 4) zero shrinkage (Haines (1923), Stirk (1954), Cornelis et al. (2006)). During the first stage large pores and channels are emptied in such a way that air can enter. No considerable change in bulk volume takes place. The first stage occurs in structured well-aggregated soil and is therefore not relevant for slurried clay. In the second stage, the decrease of water volume in the material is equal to the reduction in bulk soil volume. The intra-aggregate pores remain saturated. At the end of this stage the general air entry point is reached. The material is almost at its plastic limit (Yao et al. (2014)). In the subsequent stage the reduction in water content exceeds the reduction in bulk volume. Air enters the pores of the material. In the final and fourth stage, the soil particles are compacted to their limit and during the residual drying time no change in volume (or void ratio) occurs. Bruand and Prost showed that in the final phase, due to reorganisation of clay particles, microscopic cracks are formed. The micro-cracks do not influence the bulk volume in this stage of extreme drying. However, these may affect the swelling process upon re-wetting (Bruand and Prost (1987)). The water content at the intersection of the saturation line and the minimal void ratio $e_{min}$, as indicated in figure 2.6, is referred to as the shrinkage limit of a material and is treated as a material property comparable to the Atterberg limits.

Influence of particle arrangement

According to equation 2.4, capillary stress increases with a decreasing average pore size. An arrangement of particles and particle groups where relative movement is easy increases the shrinkage potential. In figure 2.7
the shrinkage behaviour of flocculated and non-flocculated mature fine tailings is represented. The final void ratio of the flocculated material should, following the logic above, be larger than in a non-flocculated case. Also structure anisotropy influences shrinkage. For a soil with platy particles organized parallel to the horizontal, shrinkage in the vertical direction during drying is larger than in horizontal direction (Mitchell and Soga (2005)).

A shrinkage curve equation
Fredlund et al. (2002a) proposed an equation with which a variety of measured shrinkage curves can be fitted. Soil structure features and initial degree of saturation can be accounted for in the formulation.

\[
e(v) = a_{sh} \left( \frac{w^{sh}}{b_{sh}^{sh}} + 1 \right)^{1/c_{sh}}
\]

(2.21)

where
- \(a_{sh}\) minimal void ratio
- \(b_{sh}\) slope of the line of tangency
- \(c_{sh}\) curvature of the shrinkage curve
- \(a_{sh}/b_{sh}\) specific soil constant \(G_s\)
- \(S\) degree of saturation
- \(G_s\) specific gravity of the soil

The parameters in equation 2.21 all have physical meaning. \(a_{sh}\) represents the void ratio at the shrinkage limit. \(b_{sh}\) can be fixed once the minimal void ratio is established. Parameter \(c_{sh}\) determines the curvature and is derived from previous data-sets. The most critical parameter is found to be \(a_{sh}\) (Fredlund et al. (2002a)).

Uniqueness of the shrinkage curve
In literature the shrinkage curve is referred to as a characteristic curve, indicating a unique material relationship of water content and void ratio. Altering material properties, e.g. by adding a flocculant, changes the curve. One might raise the question whether changing an environmental factor (e.g. by applying overburden pressure) in fact has no impact on the shrinkage curve.

In the numerical work by Vardon and Van Tol (2014), the drying (and rewetting) behaviour of deposited slurry (tailings) is modelled. To approximate shrinkage behaviour, the authors utilize the equation as formulated by Fredlund et al. (2002a). An alteration is proposed to equation 2.21 to capture a possible relationship between overburden pressure and minimal void ratio. Vardon and Van Tol (2014) argue that for larger overburden...
pressures, the minimum void ratio \((a_{sh})\) reduces. The parameter is modified following a non-linear displacement relationship (following equation 2.11). For the degree of saturation \(S\) to remain unaltered, parameter \(b_{sh}\) must then be modified. The alterations are given in equations 2.22 and 2.23.

Whether this relationship actually exists or not, and if so to what extent, remains to be evaluated.

\[
a_{sh}^{\text{new}} = a_{sh}^{\text{old}} - a_{sh}^{\text{old}} \cdot \frac{1}{C_{10}} \log\left(\frac{\sigma'}{\sigma'_0}\right) \tag{2.22}
\]

\[
\frac{a_{sh}^{\text{old}}}{b_{sh}^{\text{old}}} = \frac{a_{sh}^{\text{new}}}{b_{sh}^{\text{new}}} \tag{2.23}
\]

where

- \(C_{10}\) compressibility parameter
- \(\sigma'\) effective stress
- \(\sigma'_0\) reference effective stress

Motivation use shrinkage equation

In the beginning of this section the use of constitutive relationships for volume change is discussed. Yet, for the actual approximation of shrinkage, a simplified and less robust equation (i.e. equation 2.21) is used. The main advantage for the use of this equation lies within this relative simplicity. The behaviour is described with one curve and parameters based on natural soil behaviour. The stages as defined for shrinkage are well captured. Since the shrinkage curve is only a small element in the sludge ripening model, the accuracy is deemed satisfactory.

In this thesis, no new numerical simulation for shrinkage is made. The experimental data is used to verify elements in the current model. Therefore, it is for now not considered necessary to formulate a different constitutive relationship specified to unsaturated shrinkage.

2.4.3. Swelling

As the effective stress in a fine-grained soil is decreased, by unloading or by addition of water, a swelling in the soil structure may occur that cannot solely be accounted for by an increasing water content. Both physicochemical interactions between particles and the soil fabric play a role. The swelling potential of a material is represented in the swelling index \(C_s\). The swelling index is higher for a remolded material than for an undisturbed sample (Mitchell and Soga (2005)).

2.5. Evaporation in soil

Previous sections often reference to the process of evaporation of water from the soil. Here, the phenomenon is briefly discussed.

Drying occurs as water particles are displaced from the soil pores. The exfiltration of water from the soil surface into the atmosphere as vapour is the process of evaporation. The evaporative flux is complex, for this process depends both on soil properties and climate conditions.

Open water surface evaporation is dependent on climate conditions such as relative humidity, wind speed and temperature. The maximum rate of evaporation can be referred to as potential evaporation (PE) in mm/day. A fully saturated soil surface might be compared to such an open water surface. Whenever the surface becomes unsaturated, the supply of water becomes limited and the actual rate of evaporation (AE) will decline relative to the potential evaporation. In the final stage of the drying process, only a residual value for rate of evaporation remains. At this point, the water phase in the soil has become discontinuous. The flow of water to the surface ceases and transport out of the soil occurs through the process of vapour diffusion (Wilson et al. (1994)).

2.6. Considerations for this research

In applications for the engineering practice, the existence of impact of soil volume change due to consolidation is expected. In literature however, shrinkage is often assumed a characteristic material relationship. The motivation for this thesis is to optimize the prediction of settlement behaviour of deposited fine-grained soils. Therefore, the impact of overburden pressure on unsaturated shrinkage behaviour must be assessed.
After the deposition of a slurried soil, two main processes take place in the material: shrinkage through drying (i.e. evaporation) and consolidation under a certain overburden pressure. A range of intergranular forces that exist or develop in a (desaturating) soil, drives either of these. When both processes occur simultaneously, a coupled situation develops. The influence of consolidation, which is dependent on the hydraulic conductivity, may decrease as the soil desaturates to a point where the water phase is no longer continuous. Also, the extend and rate of shrinkage in a soil sample due to evaporation, may be influenced by the applied compression.

In this thesis, the relationship of the two processes is investigated experimentally. If the shrinkage curve is characteristic, or unique, to a specific soil, the application of overburden pressure should not affect its path. However, if a change in the shrinkage behaviour is witnessed, the extend and conditions of the impact by overburden pressure must be evaluated. The experimental results can be used to verify the relationship as is proposed in the Sludge Ripening model (Vardon and Van Tol (2014)). If this can be done, the mathematical formulation can be used to quantify the impact of overburden pressure.
3. Methodology

3.1. Introduction
In this thesis, the possibility of pressure dependent unsaturated shrinkage behaviour in fine-grained soil is investigated. Unsaturated shrinkage is defined as the decrease in void ratio in a soil as the water content decreases and can be represented in a curve, the shrinkage curve. This chapter describes the methods used and steps taken to determine experimentally such a curve whilst applying overburden pressure. The experimental results shall then be used to verify the proposed relationship of overburden pressure and shrinkage in the numerical model developed by Vardon and Van Tol (2014).

3.2. Experimental work
In this section, a detailed description is given of the performed experimental work.

3.2.1. Choice of method
In literature on shrinkage various methods to determine the shrinkage curve are described. In most methods the soil dries under (more or less) atmospheric pressure and are therefore in their current form not suitable for the objective of this research. Adaptations can be made in order to improve the fit with the research goals. The following tests were considered for the experimental work:

Adjusted balloon test  The balloon test is developed by Tariq and Durnford (1993). The soil sample is dried by guiding air in and again out of a balloon filled with specimen. The test is normally performed under atmospheric pressure. The balloon is every now and then submerged in water to measure the volume(changes). From this test data points for a experimental shrinkage test are derived.
As an adjustment, the balloon could be kept in a enclosed space with an elevated (air- or water)pressure. Some additional measures must be taken, such as fixating the sample and facilitating several volume measurements and weighings.

Adjusted wax test  In a large container, a saturated soil can be left to dry under its own weight. After the soil has become unsaturated, soil clods will appear. At set times, a clod can be taken, weighed, dipped in molten wax and submerged in water (to obtain the volume using Archimedes' principle). For a new measurement, the wax can be removed and the clod can be reused (Pellissier (1991)) after loading or a new clod can be taken. The wax test can be performed with an additional (porous) load placed on the sample to represent the overburden pressure in such a way that evaporation is still possible.

Dry oedometer test  Soil samples are placed in an oedometer ring, covered with a porous cap and subjected to a load. The normal oedometer procedure is performed under wet conditions to avoid desaturation of the sample. However in this case, evaporation from the soil is allowed.
The vertical settlement is measured continuously. The test must be stopped at certain time intervals to determine the weight loss due to water driven out by consolidation and evaporation. Eventual horizontal shrinkage can be measured using CT scanning.
**Hyprop test** A hyprop test is primarily used to obtain the soil water characteristic curve (as introduced in section 2.3.3). The equipment continuously measures suction developing in the sample and weight loss. The test set up can be used to derive a shrinkage curve if the changes in volume are somehow monitored. The required pressure can be applied via a topload on a porous plate.

In appendix B an elaborate substantiation is given on the choice of method for the research. Here it suffices to say that the dry oedometer method is the most suitable to perform several tests with various loads. The test set up is complemented with a hyprop test, which will provide information on the suction development in the sample and can be used as a control test.

### 3.2.2. Determination of basic soil properties

The material used for the tests is a common river clay referred to as Vingerling clay. For the testing purposes, it is necessary to simulate the slurried tailing consistency. The material should be fully saturated and brought to a high water content, preferably near the liquid limit.

Before the actual tests start, it is valuable to gather information on some basic properties of the test soil. Such properties are the grain size distribution, soil particle density and the Atterberg limits. This information may be of help during the result interpretation.

The following laboratory tests are performed:

- Hydrometer test
- Pycnometer test
- Atterberg limit (i.e. liquid limit, plastic limit) tests

For a more detailed description of the listed tests, one is referred to the Soil mechanics laboratory manual (Verwaal and Mulder (2006)). In Appendix C the results of the hydrometer test can be found. In table 3.1, the other basic physical properties are given. From the plasticity index value the soil is qualified as a high plastic clay.

<table>
<thead>
<tr>
<th>Property</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soil particle density</td>
<td>$\rho_s$ 2,6232 g/cm$^3$</td>
</tr>
<tr>
<td>Initial water content</td>
<td>$w_i$ 31.4 %</td>
</tr>
<tr>
<td>Liquid limit</td>
<td>$LL$ 58.0 %</td>
</tr>
<tr>
<td>Plastic limit</td>
<td>$PL$ 23.0 %</td>
</tr>
<tr>
<td>Plasticity index</td>
<td>$LL - PL$ 35.0 %</td>
</tr>
</tbody>
</table>

Table 3.1: Basic physical properties Vingerling Clay

### 3.2.3. Dry Oedometer test

Dry oedometer tests are performed in order to determine shrinkage curves for the material and to investigate the possible relationship of minimal void ratio and overburden pressure.

In a standard oedometer test a soil sample, confined by a stiff ring, is loaded vertically through a porous plate at the top. The sample ring is placed in a cell filled with water. Due to the load, the sample consolidates as pore water drains from the soil. Due to the water, the sample remain saturated during the entire test. The consolidation process is continuously measured as vertical sample deformation over time.

For this thesis, evaporation from the oedometer samples is allowed and over time the samples dry. Additional measures are taken to monitor the volume change of the samples. In this section, the test procedure is described in detail.

**Test set up** Four test frames are set up in a climate room of $\pm 20$ ° Celsius. Three frames are subjected to different loads which correspond to a certain overburden pressure. The fourth frame functioned as a control test. This sample is loaded only with the weight of the frame cap and transducer. All displacement transducer were connected to a computer, which monitors the settlement of the samples continuously. In figure 3.4 the test setup can be found.
The frames used for the test are selected on their sturdiness and stability. All the weights of the beams and weight hangers are taken and added to the total sample load (listed in table 3.3). During the test run, the samples must be taken out, weighted and (from a certain point) scanned in a CT scan. Therefore, the consolidation cells as well as the samples should be accessible and the loads taken off and replaced without problems. By placing separate metal blocks on the weight hanger, the required loading could be achieved and easily adjusted.

The oedometer test rings must be suitable for CT scanning. Therefore, a set of PVC rings and bottom plates has been made. The plates were necessary to contain the slurried sample. It must be noted that by use of the bottom plates, the evaporation of the sample is limited. The set up of ring, bottom plate and top cap is illustrated by figure 3.2.

In tables 3.2 and 3.3 additional information on the sample dimensions and loading can be found. For the calculation of sample volume, a sample height of 3,05 cm is used. This is done to include a small margin for the gap between the sample ring and bottom plate as observed after the sample preparation was completed.

<table>
<thead>
<tr>
<th>Test set up</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>20,9 °C</td>
</tr>
<tr>
<td>Relative humidity</td>
<td>53,0 %</td>
</tr>
<tr>
<td>Initial water content</td>
<td>58,0 %</td>
</tr>
<tr>
<td>Volumetric weight ($\gamma_s$)</td>
<td>16,00 $kN/m^3$</td>
</tr>
<tr>
<td>Ring height</td>
<td>3,0 cm</td>
</tr>
<tr>
<td>Ring diameter</td>
<td>6,5 cm</td>
</tr>
<tr>
<td>Area</td>
<td>33,2 $cm^2$</td>
</tr>
<tr>
<td>Sample volume</td>
<td>101,2 $cm^3$</td>
</tr>
</tbody>
</table>

Table 3.2: Test conditions, sample properties and dimensions

<table>
<thead>
<tr>
<th>Frame</th>
<th>Overburden [m]</th>
<th>[kPa]</th>
<th>[kg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>0,12</td>
<td>1,96</td>
<td>0,65</td>
</tr>
<tr>
<td>3</td>
<td>0,47</td>
<td>7,46</td>
<td>2,47</td>
</tr>
<tr>
<td>1</td>
<td>0,93</td>
<td>14,95</td>
<td>4,96</td>
</tr>
<tr>
<td>2</td>
<td>1,41</td>
<td>22,61</td>
<td>7,50</td>
</tr>
</tbody>
</table>

Table 3.3: Loading in kg on various oedometer frames is based on potential soil overburden height.

**Sample preparation**  The original gravimetric water content of the Vingerling clay was 31,4 %. In order to simulate the behaviour of the slurried mature fine tailings in the tests, the material water content should be as high as feasible. Also it should still be possible to apply a load unto the sample. Therefore demineralized water is added to the clay until the liquid limit of 58 % of the material is reached. The necessary amount of water is back-calculated from the initial water content. The water is added manually and the clay is remolded until an even paste has formed. The material then is stored in an airtight container and left overnight to let the water permeate through the soil mass. A small sample was taken to dry at 105 ° C for 24 hours. With this sample, the assumed water content of 58 % could be confirmed.

After 24 hours, the test samples could be prepared. Since the consistency of the material is to liquid to be contained only by a ring, a pvc plate is fastened unto the ring with tape to keep the sample from leaking. All pvc elements are weighted. Next, the container is carefully filled with the soil. The application is done with spatulas to keep a smooth surface and to limit the formation of air bubbles in the sample. After the ring is filled up, the total sample is weighted and a moist filterpaper is placed on top to limit immediate evaporation in the sample. Finally, the sample is placed in the consolidation cell in the oedometer frame. This process is repeated three times.

**Course of tests**  The test is started when the first loading is applied on the sample. Due to the high water content, the bearing capacity of the material proofed sufficient to sustain loading over 7,5 kPa on the first day.
3. Methodology

(a) Four oedometer frames are set up in the climate room. Each frame is subjected to a different load. Frame 1 and 2 are loaded with separate blocks. (b) Consolidation cell with sample, loaded by a cap and weight hanger. The measurement transducer is connected to the computer.

Figure 3.1: Oedometer test set up in climate room.

![Point Load on porous plate/cap](image1)
Stress transmitted to sample by the plate/cap
Evaporation possible through tubes
Vertical volumetric strain allowed
Outbound horizontal strain limited by sample ring
Limited evaporation possible due to bottom plate

Figure 3.2: Representation of the sample ring loaded by porous plate or cap.

On the second day, the additional load according to table 3.3 was added. Various methods are used to monitor the behaviour in the samples. All the data are gathered and analysed in Microsoft excel. All activities of the main test days are logged in appendix D, table D.1.

Measurements - Settlement The vertical settlement of the sample is recorded by the measurement transducer connected to the oedometer frame. The transducer generates data with an theoretical accuracy of 0.001 mm. During the entire test, the measurement transducer has not been set to zero or paused. At time intervals, the settlement data is exported for analysis and monitoring of the test progress. At the time of a weighing, a manual notation of the settlement was made. The recorded data can be verified by comparing it with the CT scan data.

Measurements - Weighing The change in water content in the soil samples is monitored by frequent weighing. For each weighing the procedure in table 3.4 is followed. The measurements are taken with an accuracy of 0.01 gram.
3.2. Experimental work

1. Manual notation of the the displacement (as measured by the transducer)
2. If relevant - removal of additional weights on the weigh hanger
3. Fixation of cap (and weight hanger) above the consolidation cell
4. Removal of the consolidation cell
5. Weighing of sample (together with ring and bottom plate) and notation
6. Replacing sample ring in cell and cell in oedometer frame
7. Reposition sample ring to exact location cap
8. Reposition sensor
9. Loosening of cap and placement on sample
10. If relevant - Replacement of additional weights on the weight hanger

Table 3.4: Weighing procedure

Measurements - CT scan At a certain point in time, the sample will also show horizontal shrinkage, which is more difficult to monitor. To this end, at time intervals CT scans are made of all the samples. From these scans, the volume of the sample can be determined quite accurately through visual analyses.

After the weighing process, before replacing the samples in the frames, they were taken to the CT scanner. The CT scans were made by licenced employees of Delft University of Technology. The accuracy of the scanner is 0,3 mm.

Table 3.5: Scan procedure

After the scan is made, the sample is replaced in the oedometer frame as described in table 3.4.

Measurements - Microscan To be able to investigate and compare the soil fabric on a even smaller scale, two microscans are made of respectively the sample with highest load and the sample with the smallest load (the control sample). The accuracy of the scanner is set to 10 µm.

The scans are made on the final test day, after a CT scan was made. The scans are made by a licenced employee of Delft University of Technology.

End of test The oedometer tests have run for a total of 85 days. The progress is closely monitored by frequent weighings and CT scans. From the initial water content, derived from the small test sample, the water and soil mass could be back-calculated. The rate with which the water left the sample, is also documented. The tests are stopped at an estimated gravimetric water content of 4-5 %. From the back-calculated results the tests were considered in a far enough state to draw conclusions. Also the rate of evaporation had decreased to ± 0,1 gram per day, indicating a very slow further decrease of the water content to a final dry state of the soil, which conflicted with the available time for the experimental work.

After the end of the test the samples are taken out of the frames and dried for 24 hours at 105 ° C to find the actual dry soil mass and check initial water content which are essential for further calculations.

3.2.4. Hyprop test

The hyprop test is performed with the same objective as the oedometer test: to find a shrinkage curve. An additional benefit of the hypop test is that a Soil Water Characteristic Curve (SWCC) may be found, which gives additional information on processes in the soil during desaturation.

Test set up A hyprop test is mainly designed to measure continuously the change in mass as a sample desaturates as well as the developing suction in the soil. From this data a SWCC can be constructed and other soil properties can be back-calculated. The set up consists of a hyprop measurement cell with two tensiometers (for top and bottom of the sample). On the hyprop cell a sample ring filled with material can be placed. The combination of cell and sample are then placed on a default hyprop scale, which connects to a computer.
From the start of the test onward, regularly a measurement is made and processed in the hyprop software. For more details on the general test set up, one is referred to the hyprop user manual (UMS GmbH (2015)).

For this thesis, a hyprop cell and scale are set up in a climate room op ± 25 °C and connected to a computer. Via this set up, the mass change and suction development in the test sample can be registered at regular time intervals. More information on the test set up can be found in appendix E. Before the cell can be used for testing, various procedures must be followed to saturate the cell and the suction measurement equipment (c.q. ceramic tips). This procedure was done 24 hours in advance of the test start. For an accurate description of the necessary preparations, one is referred to the hyprop user manual (UMS GmbH (2015)). After the saturation process, the hyprop cell can be connected to the computer and the scale tared to the cell weight.

As the objective of the test is to find a relationship between unsaturated volume change and overburden pressure, a type of loading must be applied onto the hyprop cell. Due to the limitation of the default hyprop scale, only a relatively small weight can be applied. Another consideration for the test set up is that evaporation from the soil should not be hindered by the application of the load. As a solution a separate mass is placed on a porous plate on top of the sample. To limit hindrance of evaporation, contact area was reduced by placing the mass on small metal screws. The total pressure on the hyprop cell is ± 2 kPa, which is comparable to the loading on oedometer ring 4.

The test ring must be suitable for CT scanning and therefore a PVC ring is used. In table 3.6 an overview of the sample dimensions are given. In table 3.7 information on the loading of the cell is given.

### Table 3.6: Conditions, sample properties and dimensions

<table>
<thead>
<tr>
<th>Test set up</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>24.5 °C</td>
</tr>
<tr>
<td>Relative humidity</td>
<td>43.7 %</td>
</tr>
<tr>
<td>Initial water content</td>
<td>55.0 %</td>
</tr>
<tr>
<td>Volumetric weight</td>
<td>16 kN/m³</td>
</tr>
<tr>
<td>Sample (ring) height</td>
<td>5.0 cm</td>
</tr>
<tr>
<td>Sample (ring) diameter</td>
<td>8.1 cm</td>
</tr>
<tr>
<td>Sample Area</td>
<td>51.5 cm²</td>
</tr>
</tbody>
</table>

### Table 3.7: Loading in kG

<table>
<thead>
<tr>
<th>Hyprop cell</th>
<th>Overburden [m]</th>
<th>kPa</th>
<th>[kG]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.11</td>
<td>1.83</td>
<td>0.94</td>
</tr>
</tbody>
</table>

**Sample preparation** For the sample preparation, material and method similar to those described in section 3.2.3 are used. The gravimetric water content of the material is increased to 55 %, slightly lower than the liquid limit. Again, the material is left to saturate for 24 hours. After this period, the hyprop ring is filled with the soil. At the time, from CT scans, it had become clear that quite some air bubbles had formed in the oedometer samples. Therefore more effort is done to avoid this for the hyprop sample. The ring is filled half way and placed in a vacuum pump. Afterwards, the sample ring is filled up completely. Again, the sample is subjected to a vacuum.

Now, the sample is weighed and moist filterpapers are placed both on the top and bottom. Next, small holes are made in the sample to accommodate the suction tips. The sample is placed on the hyprop cell according to the hyprop user manual UMS GmbH (2015) and secured by two clips. The test set up is completed.

**Course of tests** The cell with the sample are connected to the computer and placed on the scale. The test is started when the computer measurements start. The PVC ring used to hold the sample is less stiff then the default metal ring and a deformation takes place as the clips, to secure the ring, are fastened. The area of the ring has now become an ellipse and the shape of the porous plate, to be placed on top of the sample, has to be adjusted. Due to this delay, the mass was placed on the sample on day two of the test run.
A log of the activities related to the hyprop test can be found in appendix E. During the run of the test, seven CT scans of the sample connected to the cell are made. The scans are made by a licensed employee of Delft University of Technology. Unfortunately, the first four of the scans are incomplete due to incorrect scanner settings. The available data can still be used.

**Measurements - Computerdata** In the hyprop computer program, the mass changes in the sample and development of suction (tension) over time are processed instantly. The accuracy of the scale is 0.01 gram. The suction accuracy is 0.01 hPa. Also the temperature in °C is recorded. The first ± 2.5 hours of the test, measurements are taken per minute. After this point, every 10 minutes a measurement is done. The data can be exported to excel at any time.

**Measurements - CT scan** For every scan, the test was stopped and the cell disconnected from the computer. After the scan, the cell is again attached and the test is continued. The scanning procedure is similar to that of the oedometer tests.

**End of test** The hyprop test has run for 35 days. At this point, the air entry points of the two tensio shafts have been reached (i.e. air penetrates the ceramic) and no more suction development can be registered. The gravimetric water content has decreased to 5%. Enough data is available to draw conclusions and the test is stopped.

The test setup is dismantled as described in the hyprop manualUMS GmbH (2015) and stored away in the hyprop box. The sample material is collected and dried 24 hours at 105 °C to determine the actual initial water content and dry soil mass.

### 3.3. Data processing and interpretation

In the previous sections, the actual experimental tests were described to generate the necessary data for this thesis. In this section, the methods used to process and interpret this data are elaborated. For analysis of the oedometer test and the hyprop test, similar processing methods are used.

#### 3.3.1. Basis of the analyses

The objective of this thesis is to find a shrinkage curve for the material. This curve represents the relationship of void ratio \( e \) and water content as a material dries. Therefore, the saturation line (degree of saturation \( S = 100\% \)) is often used as a reference line, since void ratio is water content if no air enters the system. The following equations form the basis of this analysis:

\[
w = \frac{M_{\text{water}}}{M_{\text{soil particle}}}
\]

or

\[
\theta = \frac{V_{\text{water}}}{V_{\text{soil particle}}}
\]

and

\[
ev = \frac{V_{\text{voids}}}{V_{\text{soil particle}}} = \frac{V_{\text{water}} + V_{\text{air}}}{V_{\text{soil particle}}}
\]

The mass and volume of soil particles and water can be directly derived from the test measurements. The dry soil mass \( M_s \), the changes in water mass \( dM_w \) and the densities of water \( \rho_w \) and the soil particles \( \rho_s \) are known. Therefore, it is of little influence whether gravimetric water content \( w \) or volumetric water content \( \theta \) is used. In this thesis, the preference goes out to the use of volumetric water content, for \( \theta \) is directly proportional to void ratio.

Determining the volume of the air that one assumes will enter the sample at a certain point during drying is more complicated. In this thesis the mass of air is assumed negligible. From equation 3.4 follows that from the total volume of the sample the volume of air can be derived.

\[
V_{\text{air}} = V_{\text{total}} - V_{\text{soil particle}} - V_{\text{water}}
\]
To determine the total volume of the sample during testing several methods are available. The choice for a method strongly depends on the definition of sample volume that is used. For this thesis three volume definitions are employed. For the minimal total volume, only the soil matrix is taken into account. The maximum volume change is defined solely by macroscopic settlement. In between these boundaries, a range of volumes is found that depends on the interpretation of the author. For each of these sample volumes, a void ratio and water content can be determined, yielding three different types of shrinkage curve.

**a)** The first definition for total sample volume, $V_{\text{soil matrix}}$, takes into account only the actual soil matrix. Cracks and air pockets are excluded from the total volume. The extend of the exclusion depends on the accuracy of the data and choices made by the interpreter. $V_{\text{soil matrix}}$ can be determined from the CT scan data.

**b)** The second definition, $V_{\text{settlement}}$, is more general as only vertical volumetric strains are taken into account. The settlement of the sample over time is taken and multiplied by a constant area $A_{\text{ring}}$, as shown in equation 3.5. Any horizontal shrinkage and crack formation is considered part of the sample. This approach is based on soil settlement due to consolidation on a macroscale.

$$V_{\text{settlement}} = (H - \Delta h_{\text{settl}}) \times A_{\text{ring}} \tag{3.5}$$

**c)** The third volume definition of total volume, $V_{h\&v-\text{shrink}}$, includes both vertical and horizontal shrinkage of the sample. Whether large cracks are also excluded from the total volume depends on the desired accuracy and available data. In equation 3.6 the approach for $V_{h\&v-\text{shrink}}$ is given:

$$V_{h\&v-\text{shrink}} = (H - \Delta h_{\text{settl}}) \times A_{\text{sample}} \tag{3.6}$$

The vertical volumetric stains are equal to the ones used to find $V_{\text{settlement}}$ and its values are multiplied by an area $A_{\text{sample}}$. To determine $A_{\text{sample}}$, several methods are available. Per sample per measurement a constant area can be chosen, or a gradient over the sample to match irregularities and/or large cracks. An elaboration on the definition for $A_{\text{sample}}$ can be found in the subsequent section.

In figure 3.3, the three definitions for total sample volume are illustrated for a conceptual vertical sample cross section.

![Figure 3.3: The total volume of a sample is dependent on which definition is used. In the figure, for a schematic sample, the volume is determined according to three different definitions. Within $V_{h\&v-\text{shrink}}$ various options, for example for the choice of $A_{\text{sample}}$ are available.](image)

**3.3.2. $V_{\text{soil matrix}}$**

The CT scan data is used to find the volume of the soil matrix. The data is imported into Avizo 8.01, a strong visual analysis program, where the data is translated into voxels representing a 3D object.
Ideally, the entire object is scanned and can be imported into Avizo 8.01. Different material densities are represented as a grayscale in the image. A selection can be made on which densities or materials to take into account in the analysis. This is done by setting a threshold\(^1\). For example, the accuracy in exclusion of air pockets is set by this threshold. Next, the volume of the object voxels within the threshold can be calculated. Unfortunately, the grayscale representing the PVC ring, which was used to allow CT scanning in the first place, is similar to that of the sample material, making it impossible to completely leave out ring voxels in the calculation of the sample volume. A solution would be to compute the volume of sample and ring together and later subtract the constant ring volume.

After inspection of the CT scan data, it is discovered that not every data set contains the entire object but that often a small piece of the pvc ring or even of the sample itself is missing.

To deal with this setback, a volume edit operation is performed in which the ring data and other objects are cut out. A constant cylinder volume is saved into a template, in which a scan data set is imported. Only the position and orientation of the cylinder is altered per data set to create a reasonably standardized procedure and result with a set maximum. In table 3.8 the method is given that is employed to generate the soil matrix volume:

0. Create template file with standard cylinder volume edit
1. Import CT scan data into template
   a. [Open data]
2. Choose manner of representation data
   a. [Ortho slice x,y,z directions]
   b. [Volume rendering]
3. Adjust cylinder volume position to fit data (figure 3.4a)
   a. [compute - volume edit]
   b. [cut - outside]
4. Apply standardized threshold for sample data (figure 3.4b)
   a. [Image segmentation - binarization - interactive threshold ]
   b. [Left threshold: 555,86]
   c. [Right threshold: 2249,68]
5. Calculate volume sample based on threshold
   a. [Measure & analyse - individual measures - label]
6. The separate volumes of the sample are represented in a table
   a. [Window - table ]
   b. Export Volume column to excel
7. If necessary the separate volumes can be summarized in excel and processed.

Table 3.8: Method Avizo 8.01 3D soil matrix volume computation. In brackets, the actual program steps are referenced.

---

1 For a 16 bit data set, grayscale values range between 0 and 65535. A threshold creates a surface around all voxels with grayscale values within the set range.

---

Figure 3.4: Stages in the volume computation process as described in table 3.8
3.3.3. $V_{\text{settlement}}$
For the oedometer test, the settlement is directly measured and processed by the measurement transducer and the connected computer. From this settlement and the constant ring diameter, $V_{\text{settlement}}$ can be computed. However, for the hyprop test, the settlement must first be determined before any volume computations can be done. This is done using the CT scan data. The data analysis to find the vertical settlement is performed for both tests. For the oedometer test, this additional data set can be used as verification of the transducer results.

The soil mass volume after settlement can be measured directly in visual analysis program Avizo. The Avizo measurement tool automatically locks to a certain grayscale in the object and can calculate the dimensions of a shape, for instance a line on a orthogonal section of the sample. However, corrections for non-orthogonal scans or displaced samples are not accurate. Since this causes problems with many scan data sets, this is not considered a suitable approach.

Instead of importing the data as 3D object in Avizo, the scan data can be exported (e.g. to JPEG) as slices corresponding to the voxel size (0.3 mm) in orthogonal directions. The vertical cross sections are corrected in adobe illustrator\(^2\). The sample measurement is now taken and normalized for a known value such as the height of the pvc ring. Due to limited accuracy of the scanner and a variety in the grayscale, several options for the sample boundaries can be considered. After an option is chosen, it is important to be consequent in all analyses.

Per sample, for several cross sections such a analysis can be done to assess the accuracy of the result. The results are processed in microsoft excel and $V_{\text{settlement}}$ is computed with equation 3.5.

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.</td>
<td>Create template with dimensions ring and plate in adobe illustrator</td>
</tr>
<tr>
<td>1.</td>
<td>Import scan image in adobe illustrator file</td>
</tr>
<tr>
<td>2.</td>
<td>Measure height sample</td>
</tr>
<tr>
<td>a.</td>
<td>Approximate sample shape with rectangle</td>
</tr>
<tr>
<td>b.</td>
<td>If necessary, correct for rotated image or displaced sample</td>
</tr>
<tr>
<td>c.</td>
<td>Outline top of rectangle with sample border in scan image</td>
</tr>
<tr>
<td>d.</td>
<td>Outline bottom of rectangle with sample border</td>
</tr>
<tr>
<td>3.</td>
<td>Correct data for known ring height</td>
</tr>
<tr>
<td>4.</td>
<td>Process data in excel</td>
</tr>
</tbody>
</table>

Table 3.9: Procedure to determine $h_{\text{settlement}}$

Figure 3.5: Due to limited accuracy scanner and variety in the grayscale, several options for the sample boundaries can be considered. The white arrow indicates the definition of $h_{\text{settlement}}$ in this thesis.

3.3.4. $V_{\text{h&v-shrinkage}}$
In figure 3.3 several options are illustrated of what volume should be taken into account for $V_{\text{h&v}}$. This is strongly depended on the chosen definition of to what extend voids and cracks are part of the sample. The implication for the definition of the sample area are discussed here. Figure 3.6 further illustrates the four considered options.

\(^2\)The analysis can be performed using other programs. Illustrator is convenient due to representation of vectors and ease of use.
3.3. Data processing and interpretation

Volume option I  
For option I, the largest circular sample area is taken into account. An advantage of this definition is that the entire soil sample is captured. A drawback could be that large cracks (and therefore a very large void ratio) are taken into account due to a small percentage of soil. The behaviour of this small element may not be representative to the overall soil shrinkage behaviour (e.g. the roughness of the ring may be of influence). The results will then yield a relatively high minimal void ratio. In table 3.10 the steps taken for this analysis are described.

1. Import CT scan data
   a. [Open data]
2. Choose manner of representation data
   a. [Ortho slice x,y,z directions]
   b. [Volume rendering]
3. Find largest sample area (visually) by 'scrolling' through data
4. Measure the radius
   a. [Measurement tool - circle]
5. Process data

Table 3.10: Procedure for option I

Volume option II  
Volume option II is based on the radius of the sample core, disregarding soil that has stuck to the ring or is otherwise separated. One can argue that this volume conveys the true shrinkage process. It then seem fair to use the diameter of this volume as a basis of $V_{hke}$. An additional argument for this approach is that the determination of the area is straightforward for the sample 'core' is quite compact and cylinder shaped.

A disadvantage is that the air volume and thus the void ratio might be slightly underestimated. The volume of the soil particles is based on the total dry soil mass and the water volume on the current water content of the entire sample. The overestimation of these two leads to an underestimation of void ratio. To avoid this, a correction must be made for the soil mass not included in the 'core' volume.

In table 3.11 the steps taken for this analysis are described.
1. Import CT scan data
   a. [Open data]

2. Choose manner of representation data
   a. [Ortho slice x,y,z directions]
   b. [Volume rendering]

3. Find sample ‘core’ area, check by ‘scrolling’ through data

4. Measure the radius
   a. [Measurement tool - circle]

5. Process data in excel

Table 3.11: Procedure for option II

For the correction of the data, an additional analysis is done:

1. Determine dry mass of the sample core from total dried test sample
2. Compute \( V_{\text{core-soil particle}} \)
3. Back-calculate \( V_{\text{core-water}} \)
4. Determine \( V_{\text{voids}} \)

Table 3.12: Correction procedure

**Volume option III** In Avizo, the scan data can be analysed in the xy direction. After the volume edit operation and setting of a threshold (as described in section 3.3.2), the area of each slice (c.q. image per voxel) can be computed. Summarizing each of these areas gives us the soil matrix volume. However, if the maximum computed area is taken and extrapolated for the entire sample, an approximation is made of the soil volume including cracks.

The advantage of this method is that the maximum area of the actual sample is computed, instead of relying on a manual measurement. The disadvantage is that the method is slightly arbitrary and does not correspond as much to the physical sample as the other options.

0. Create template file with standard cylinder volume edit
1. Import CT scan data into template
   a. [Open data]

2. Choose manner of representation data
   a. [Ortho slice x,y,z directions]
   b. [Volume rendering]

3. Adjust cylinder volume position to fit data
   a. [compute - volume edit]
   b. [cut - outside]

4. Apply maximum range threshold for sample data
   a. [Image segmentation - binarization - interactive threshold]
   b. [Left threshold: 20]
   c. [Right threshold: 2300]

5. Compute area sample
   a. [Measure & analyse - global measures - area]
   b. [Object in xy direction]
   c. [Window - table]

6. The maximum computed area is governing for further analysis
7. Process data in excel

Table 3.13: Procedure for option III

In the options one to three, an area is defined, from which a volume can be computed. To do this, area is multiplied by a value of \( h_{\text{settlement}} \). For this, the procedure to find \( V_{\text{settlement}} \) is used.
3.3. Data processing and interpretation

Volume option IV  Ideally, the exact volume of the sample trace is calculated. Large cracks are excluded from the volume, but small cracks and air pockets are considered part of the soil volume. The final sample volume (c.q. the last measurement) can be calculated using Archimedes’ principle. During the test however, this cannot be done without seriously disruption of the process and samples. With Avizo, or a different data analysis program, such a volume may be computed. For this thesis, an attempt is done in Avizo, unfortunately without success. Two main problems were encountered: First, even with the lowest threshold, air pockets are excluded from the computed volume. Second, when a trace is made, large and small cracks as well as air pockets are considered surface area and included in the calculations. This approach is therefore dropped for this thesis.

The first three options are tested and the results compared.

3.3.5. Creating the shrinkage curve

Based on $V_{\text{soil matrix}}$, $V_{\text{settlement}}$ and $V_{\text{h&v-shrink}}$ the corresponding void ratio can be computed, which are referred to as $e_{\text{soil matrix}}$, $e_{\text{settlement}}$ and $e_{\text{h&v-shrink}}$. The subscripts (and abbreviations) referring to the three definitions are frequently used in the subsequent chapters. The shrinkage curves are obtained by plotting the various void ratios against the volumetric water content. A saturation line, based on a degree of saturation $S$ of 100 %, is also included as a reference line.

3.3.6. Microscan analysis

Besides the CT scan technology, also a micro scanner can be used to assess the volume of the sample. Due to the duration and heat production of a scan session, the method is not suitable for use during the dewatering phase for the release of the sample and evaporation would be compromise the data. However, a test can be executed on the final test day. The microscan sample volume can be compared to that obtained with the CT scan to give insight in the void ratio on a small scale. The accuracy of the microscan data is 10 $\mu$m.

For this analysis, a square volume is cut out of the microscan and CT scan data set. Now the same steps are performed as for the analysis of $V_{\text{soil matrix}}$. The threshold that is applied is 10000 to 15000. In figure 3.7 representations of the set threshold and the cut out volume are given. The micro scan void ratio can be back-calculated using the ratio between the sample $V_{\text{soil matrix}}$ and the square. It must be noted that for the back-calculation a homogeneous sample is assumed.

![Set threshold for the micro scan analysis](image1.png)

(a) Set threshold for the micro scan analysis

![Volume render for microscan analysis](image2.png)

(b) Volume render for microscan analysis

Figure 3.7: For sample 2 kPa an analysis of the sample volume is performed at an accuracy of 10 $\mu$m.

3.3.7. Sensitivity analysis

In this section, the sensitivity of the computed data is discussed. Several factors that possibly influence the accuracy of the total soil volume are reviewed. The accuracy of the scan data is known to be 0,3 mm $^3$. This is $^5$The voxels, created from the scan data, have a dimension of 0,3 mm x 0,3 mm x 0,3 mm.
always accounted for in a sensitivity analysis.
To be able to determine the accuracy of the results, a difference must be made between measurement errors
(random errors) and limitations of the method (systematic errors) (Carlson (2002)). For the first category, a
range of values can be produced, of which the impact on the final results is analysed. Inaccuracy of the second
category is inherent to the chosen definitions of total sample volume and not further elaborated. Lastly, the
accuracy of a datapoint can also be compromised if the data set is not complete. Either the datapoint can be
omitted or corrected (in which case an error of the previous two categories may occur).
In table 3.14 a summary of the analysis is given per separate parameter.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Content</th>
<th>Factors</th>
<th>Cause of inaccuracy</th>
<th>Type or error during interpretation</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_{ring}$</td>
<td>area ring</td>
<td>measurement</td>
<td>measurement error</td>
<td>Random</td>
</tr>
<tr>
<td>$A_{sample}$ option I,II</td>
<td>sample</td>
<td>scan image</td>
<td>assumed circular area</td>
<td>Systematic</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(non) completeness</td>
<td>Incomplete dataset</td>
</tr>
<tr>
<td></td>
<td>option III</td>
<td>visual analysis</td>
<td>scan data</td>
<td>Random</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>subjective assessment of sample diameter</td>
<td>Incomplete dataset</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(non) completeness</td>
<td>Random</td>
</tr>
<tr>
<td></td>
<td>option III</td>
<td>visual analysis</td>
<td>threshold</td>
<td>Systematic</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>subjective positioning volume edit</td>
<td>Incomplete dataset</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>subjective definition of void vs. soil matrix</td>
<td>Random</td>
</tr>
<tr>
<td>$h_{settlement}$</td>
<td>variable</td>
<td>sample</td>
<td>assumed horizontal top/bottom</td>
<td>Systematic</td>
</tr>
<tr>
<td></td>
<td></td>
<td>scan image</td>
<td>(non) completeness</td>
<td>Incomplete dataset</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>accuracy /grayscale</td>
<td>Random</td>
</tr>
<tr>
<td></td>
<td></td>
<td>visual analysis</td>
<td>subjective assessment sample top/bottom</td>
<td>Random</td>
</tr>
<tr>
<td>$V_{soilmatrix}$</td>
<td>variable</td>
<td>scan data</td>
<td>(non) completeness</td>
<td>Random</td>
</tr>
<tr>
<td></td>
<td></td>
<td>visual analysis</td>
<td>accuracy /grayscale</td>
<td>Random</td>
</tr>
<tr>
<td></td>
<td></td>
<td>threshold</td>
<td>subjective positioning volume edit</td>
<td>Random</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>subjective definition of void vs. soil matrix</td>
<td>Systematic</td>
</tr>
</tbody>
</table>

Table 3.14: Sensitivity analysis

$A_{ring}$
The area of the sample ring is back-calculated from the ring diameter. The area is assumed to be a perfect
circle. The measurement is performed with a caliper and has an accuracy of 0.1 mm.

$A_{sample}$
The use of a circular area is inherent to the methods (option I,II) in the previous section and is therefore not
taken into account in the sensitivity analysis of the variable. Due to the predefined shape of the area, the
completeness of the scan image is not relevant as long as a sample diameter can be determined.
The sensitivity for option I and II is mainly caused by the subjective definition of the radius of the sample in
Avizo.

For option III, the completeness in the xy direction of the data becomes relevant. If a dataset is incomplete,
the maximum area cannot be computed and must be compensated for or is incorrect. In such a case, mention
must be made in the representation of the results.

For every avizo analysis, the manual placement of the volume edit is of (some) influence on the outcome. The
impact is further discussed for the parameter $V_{soilmatrix}$.

For the threshold, a maximum range is chosen to include small cracks and small air pockets. A higher upper
boundary threshold is not relevant in this case. Therefore, the threshold is considered to be inherent to the
method and not accounted for in the sensitivity analysis.

$h_{settlement}$
Whether $h_{sett}$ is found with the oedometer measurement transducer or with visual analysis, the sample top
is assumed to be horizontal. A deviating formation of the actual sample are therefore not accounted for in the
sensitivity analysis.

If the sample scan is incomplete in the vertical direction, $h_{settlement}$ cannot be be determined and the data
Verification of the numerical results

In chapter 2, the Sludge Ripening Model by Vardon and Van Tol (2014) is briefly introduced. The tool, developed at Delft University of Technology, is used to model the behaviour of tailings during layered deposition. The soil water retention relationship, shrinkage and re-wetting behaviour and the influence of hydraulic conductivity can be simulated. For this thesis, mainly the modelled shrinkage behaviour is relevant.

3.4.1. Relevant characteristics of the model

In section 2.4.2 the main equations with which the shrinkage behaviour is modelled are given. The shrinkage curve properties are \( a_{sh}, b_{sh} \) and \( c_{sh} \) as defined by Fredlund et al. (2002a). If overburden pressure is taken into account, \( a_{sh}, b_{sh} \) are replaced by compression coefficient dependent \( a_{sh}^{new} \) and \( b_{sh}^{new} \). Alterations can be made in the material properties interface (i.e. figure 3.8).

![Figure 3.8: Model interface for shrinkage curve properties](image)
3. Methodology

3.4.2. Verification of the relationship

The experimental data obtained for this thesis, can be used to verify the proposed relationship between minimal void ratio and overburden pressure.

In the chapter 2 the shrinkage equation and the motivation for an alteration are discussed. The relevant equations for the verification of the relationship are repeated here.

\[
\epsilon_v = -\frac{1}{C_{10}} \log\left(\frac{\sigma}{\sigma_0}\right)
\]  

(3.7)

where

\[
\epsilon_v = \frac{\Delta a_{sh}}{a_{sh}^{old}} = \frac{a_{sh}^{new} - a_{sh}^{old}}{a_{sh}^{old}}
\]

(3.8)

giving

\[
a_{sh}^{new} = a_{sh}^{old} - a_{sh}^{old} \frac{1}{C_{10}} \log\left(\frac{\sigma'}{\sigma_0}\right)
\]

(3.9)

In figure 3.9 the concept of the relationship is presented. During Atmospheric Fines Drying, layers of slurried material are deposited and left to dry. If overburden pressure is applied on top of a drying layer, a decrease in minimal void ratio is expected. If the relationship can be described with equation 3.9, from the experimental data set for minimal void ratio and applied stresses a single value for \(C_{10}\) must be found.

![Figure 3.9: The influence of overburden pressure as assumed in the Sludge Ripening Model](image)

If the data set does not give a perfect linear relationship, an approximation can be made using a linear regression model. The quality of the fit can be indicated using the coefficient of determination \((R^2)\), which ranges from 0 to 1. A \(R^2\) of 1 indicates a perfect fit, and a value of 0 indicates no fit at all. Due to the small data set in this thesis, the reliability of this method is limited and must be seen as an indication rather than confirmation.

3.5. Validation of the results

The motivation for the thesis objective springs from the demand for accurate prediction of volume change behaviour of deposited slurried soils in the engineering practice. The question whether overburden pressure impacts the unsaturated shrinkage behaviour was raised. In the scientific community, the impact is assumed to be non-existent. Observations from experimental tests on shrinking material may help clarify the matter, thereby increasing knowledge for either discipline.

The value of the thesis can be assessed by the applicability and relevance of the results for the scientific community and the engineering practice.
3.6. Concluding remarks

The main research question is investigated by means of experimental tests. A sequence of oedometer tests under dry conditions is executed as well as a hyprop test. The test samples are subjected to a range of constant loads in order to compare the various soil responses. In order to monitor the volume change behaviour, the samples are frequently weighed and scanned during the test. The obtained data is analysed using visual analysis methods. Based on the test and scan data the water content and void ratio of the samples over time are computed.

Three soil volume definitions are introduced that describe volume change behaviour on an increasingly detailed scale. The definitions are relevant for different disciplines and useful in the interpretation of the findings. In line with the definitions, three types of shrinkage curve are constructed per sample (based on the experimental data). Per curve conclusions can be drawn on the impact of the applied overburden pressure on the shrinkage behaviour.

In the numerical model developed by Vardon and Van Tol (2014) a certain impact of overburden pressure is assumed and formulated as a relationship between minimal void ratio and applied stress based on a compression constant $C_{10}$. If impact is observed for the experimental shrinkage curves, the relationship can be confirmed qualitatively. The test data can then be used to verify the numerical approach of predicting the impact.

In the subsequent chapter, the results obtained with the procedures described in this chapter are addressed.
4.1. Introduction
In the previous chapter, the research methodology is presented. Three steps are defined: experimental work, interpretation of the data and verification and validation of the model. These steps are also relevant for this chapter. First, the raw experimental results are presented, giving information on the state of the samples and the research process. Subsequently, the interpretation of the results yields the various shrinkage curves that are the objective of the thesis. In the final step, the results can be compared to the numerical results produced by the Sludge ripening model.

4.2. Experimental results
In this section, the direct results from the laboratory tests are presented and discussed. Both test types, the oedometer test and the hyprop test, are standard tests, modified in order to meet the objective of the thesis. The modifications have influence on the procedure, as is described in the previous chapter, but also on the test output. In this section, three main test output categories are defined: consolidation, dewatering and matric suction development. For each of the categories, the results are presented and analysed.

4.2.1. Consolidation
In section 2.4 the concept of soil volume change was elaborated and various influencing factors introduced. One important factor in volume change that was discussed is consolidation due to one dimensional compression. The main objective of this research is to assess the impact of overburden pressure on the unsaturated shrinkage behaviour of fine-grained soils. Since overburden pressure can be interpreted as one dimensional compression, the assessment of this particular behavioral aspect in the experimental results is relevant.

The samples were subjected to various loads. For each sample, a different settlement over time was registered. In figure 4.1 the settlement as registered by the oedometer transducers is given for the samples subjected to 2 kPa and 22,5 kPa. The datasets are corrected for initial displacement of the sensor during the start of the test, inaccuracies and extreme fluctuations during the test. The time axis is set to a logarithmic scale, as is common for consolidation representations. The figure displays several gaps, which are the intervals at which the samples were taken out for a weighing and/or CT scan. It is visible that at the start of most intervals, the transducers register a small reduction in settlement.

The curve for the sample subjected to 2 kPa is relatively smooth. The sample loading was kept constant during the entire test (with the exception of the weighing and scan moment(s)). $t_{50}$ is reached after approximately 10 test days. The final sample settlement is measured at -7,84 mm.

The curve for 22,5 kPa shows a disruption as the additional load is placed on the sample on test day 2. The consolidation rate increases in the subsequent day. $t_{50}$ is reached after approximately four test days. The total settlement is according to the dataset of -9,85 mm.

1The fluctuations that are corrected for arguably do not correspond with reality, as is addressed later in this thesis.
4. Results

Figure 4.1: Settlement of oedometer test samples subjected to 2 kPa and 22.5 kPa.
4.2. Experimental results

In this section, only the settlement paths of oedometer samples subjected to 2 and 22.5 kPa are shown. It is thought that these samples represent the extremes within the test and are representative for transducer measured settlement. The settlement of the hyprop was not measured directly and was obtained from CT scan analysis.

4.2.2. Dewatering

During the oedometer test and the hyprop test, evaporation is allowed as the sample consolidates. The decrease of (water)mass is closely monitored. In figure 4.2 the registered mass change due to consolidation and evaporation is represented for the oedometer samples. The behaviour of the samples can be characterised by the two extremes (i.e. samples loaded by 2 kPa and 22.5 kPa). In the beginning of the test, the sample curves differ only slightly as a result of a small range in loading. After the application of additional loading, the samples loaded with 15 and 22.5 kPa show a rapid decrease in pore water mass. The 2 kPa sample water mass decreases more slowly and fairly constant. After the fourth test day however, the intermediate samples diverge from the 22.5 sample path and creep towards the 2 kPa sample curve. One week into the test, a clear gap becomes visible between the 22.5 kPa sample and the other samples. Only at the very end of the test run, the curves converge again as the samples approach their residual water content.

All sample curves show to a certain extent three different stages. The interpretation of each stage is deferred to the subsequent chapter.

In figure 4.3 the rate with which the water is driven out of the sample is shown. This is expressly referred to as dewatering rate (as opposed to evaporation rate) for the two processes of consolidation and evaporation are at work. The figure is plotted on a logarithmic scale to stress the changes in the dewatering rate at the beginning of the test. For every sample, large spikes are visible at the beginning of the test. After a while, the dewatering rate becomes more steady, although some fluctuations remain. Also in this figure, the gap that was observed in figure 4.2 is present, be it slightly less pronounced.

On test days 8 to 10, a larger fluctuation is visible for all samples. The dewatering rate drops and increases again. After this point, the dewatering rates for the samples 2 kPa, 7.5 kPa and 15 kPa are similar and remain more or less constant around -1 gram/day. The curve for sample 22.5 kPa decreases over time toward zero as the water mass becomes constant (i.e. nearing the residual water content). In the final test days, all rates approach zero.

In figures 4.4 and 4.5 the dewatering curves of the hyprop test sample are represented. The curves are similar.
4. Results

Figure 4.3: Dewatering rate of the oedometer test samples. A spike is seen due to the loading of the samples on test day 2.

The dewatering rate is compared to the ones seen for oedometer sample loaded with 2 kPa. The main difference is the time in which the test is performed; the hyprop test took much less time. Another difference from the oedometer 2 kPa sample is that the hyprop loading was applied on the second day. Therefore any consolidation effect (not taking into account self-weight consolidation) is present from test day two onward. Also, the hyprop test was stopped before the residual water content was reached.

In figure 4.4 for the first test day a decrease of water content is seen due only to evaporation. As the loading is placed on day two, a small change in curvature appears. In figure 4.5 a peak on the first test day is visible. The application of the load on day two does not cause an increase in dewatering rate (as was observed for the oedometer samples); a sudden decrease is visible. The data shows quite a few fluctuations but the trend of the rate going to zero is detectable.

Figure 4.4: Pore water mass change of the hyprop sample, subjected to 2 kPa
4.2. Experimental results

4.2.3. Suction

In chapter 2 the phenomenon of matric suction (i.e. the development of pore water tension) as a driver for volume change has been discussed. The hyprop test is designed to measure the suction by means of tensiometers. Measurements are made in the top and bottom of the sample.

From the continuous weighing of the hyprop sample, the volumetric water content can be derived. The relationship between suction and water content is captured in the soil water characteristic curve (figure 4.6). The tensiometer data is corrected for the initial hydrostatic pressure. The dashed line in the figure indicates the water content that corresponds to the moment in time when the load is placed on the sample. A temporary decrease in suction is observed. Afterwards, the suction increases further. In the figure, only the representative part of the data is used. In reality, the development of suction continues as the water content of the soil drops further.

Figure 4.5: Dewatering rate of the hyprop test sample, subjected to 2 kPa

Figure 4.6: Soil Water Characteristics Curve (SWCC) for the hyprop sample loaded to 2 kPa.
According to theory, the suction develops until the air entry pressure is met, at which point pore water is displaced by air and the volumetric water content drops. If this is the case, the air entry pressure for the unloaded sample is around 0.8 kPa. The suction increases almost up to 4 kPa as the load is placed, at which point the suction drops to 3 kPa. After a small fluctuation, the suction increases exponentially (i.e. linear on log scale).

In figure F.1 the development of suction over time as measured by the tensiometers is given. Only a small decrease in tension is observed as the loading is placed on top of the sample. The curve shows the soil behaviour up to test day ten, after which the limits of the apparatus are met and the measurements are no longer (completely) representative.

4.2.4. Scan data

The unsaturated shrinkage behaviour of the oedometer and hyprop samples is monitored by frequent CT scanning. From these scans, the volume of the samples can be derived following the procedures described in section 3.3. The results of the interpreted data are presented in the next section. In this section, the results that illustrate the shrinkage process are presented.

Oedometers

To illustrate the shrinkage process as the samples dry, three moments in time are presented per sample. For these moments, a horizontal and a vertical cross section are shown. Which moments are taken, is defined in table 4.1. The horizontal cross section is taken at 1.5 mm from the bottom of the sample. The vertical cross section is taken at the largest diameter of the sample.

The samples are not completely homogeneous. In the scan images, the range in the grayscale refers to several material densities. Also, at the time of the first scan, the samples contain air pockets and small fissures. It is most likely that they are products of the sample preparation process.

The initial registered volume change behaviour in the sample is vertical settlement. At a certain point, also horizontal shrinkage becomes visible. Generally speaking the samples ‘contract’ as a unity and a continuous crack or gap forms between the sample and the ring. In the images it can be seen that this is not everywhere the case. Soil material sticks to the ring and/or the cracks follow an alternative path.

From the image sequences of the samples over time, it becomes plausible that the origin of diverging cracks is influenced by a air pocket or fissure. The configuration of shrinkage is a fairly random process.

<table>
<thead>
<tr>
<th>Date</th>
<th>Test day</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>28-07-2015</td>
<td>8</td>
<td>first sample scans, no horizontal shrinkage observed (yet)</td>
</tr>
<tr>
<td>18-08-2015</td>
<td>28</td>
<td>horizontal shrinkage is observed in all samples for the first time</td>
</tr>
<tr>
<td>13-10-2015</td>
<td>85</td>
<td>final test day, residual water content reached</td>
</tr>
</tbody>
</table>

Table 4.1

Figure 4.7: Oedometer sample subjected to 2 kPa (horizontal cross section)
4.2. Experimental results

Sample 2 kPa  In the horizontal cross section, the presence of air pockets in an early state is visible. As horizontal shrinkage starts (figure 4.7b), most of the sample separates from the sample ring. At the bottom of the image, it can be seen that the crack takes an alternative path through a large air pocket. In the final stage of the shrinkage, the entire sample has separated from the ring (figure 4.7c). The alternative crack path remains intact, yielding a non-homogeneous sample.

In the vertical cross section, the development of the horizontal shrinkage is visible. It may be noted that the sample curls up at the rim over time. Cracks inside the sample start at the bottom of the sample but do continue over the whole sample height. The top of the sample remains therefore homogeneous. Over time the sample takes on a trapezoidal shape as the sample diverges slightly at the bottom.

Sample 7.5 kPa  The behaviour witnessed in sample 7.5 kPa is similar to that of sample 2 kPa. The large air pockets and fissures visible in figure 4.9a seem to provide shortcuts for cracks forming during horizontal shrinkage. The sample in figure 4.9c ends up looking rather frayed.

In the vertical cross sections, a slight curl is visible near the sample rim. The diameter of the sample increases towards the bottom (figure 4.10c) due to presence of cracks in the sample.
Sample 15 kPa  This sample deviates from the others for this sample shows the most 'standard' shrinkage. With the exception of the large air pocket visible at the top edge of image sequence of figure 4.11, the sample is fairly homogeneous. No large cracks form in the sample; the sample separates from the ring as one piece. This is confirmed in the vertical cross section (figure 4.12). The sample diameter is more of less constant over the sample height. The top of the sample shows only a very slight curl.

Sample 22,5 kPa  In figure 4.13a many air pockets and fissures are visible. In the subsequent images, it can be seen that often these abnormalities develop into larger pockets and cracks. The sample in figure 4.13c
shows a layered pattern. In the vertical cross section, the crack development over time is clear. Also here, the cracks seem to originate from the bottom of the sample and do not continue over the entire sample height, leaving the top scatheless. The sample takes on a trapezoidal shape over time.

**Hyprop**

For the hyprop sample a similar analysis can be made on the shrinkage process. Due to scan errors, the first four data sets are incomplete (as can be seen in figure 4.15a). The shrinkage pattern in the soil originates from early cracks or fissures in the sample. Unlike the oedometer samples, the hyprop sample is not massive due to the tensiometers. In figure 4.16 the top tensiometer is visible. The soil shrinks around the tensiometer in horizontal *and* vertical direction. From test day 14\(^2\) onward the sample balances on top of the sensor (figures 4.16b and 4.16c). The dates of the presented scan images are explained in table 4.2.

<table>
<thead>
<tr>
<th>Date</th>
<th>Test day</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>27-08-2015</td>
<td>3</td>
<td>first sample scan, no horizontal shrinkage observed (yet)</td>
</tr>
<tr>
<td>14-09-2015</td>
<td>20</td>
<td>horizontal and vertical shrinkage on all sides</td>
</tr>
<tr>
<td>28-09-2015</td>
<td>34</td>
<td>final test day</td>
</tr>
</tbody>
</table>

Table 4.2

Figure 4.15: Hyprop test CT scan data

Figure 4.16: Vertical cross sections of the hyprop cell and sample.

\(^2\)At this date, the phenomenon was observed first. It may have occurred before this time.
4.3. Processed results

The results as presented in the previous section give information on the soil behaviour during drying. In order to obtain the shrinkage curve, the results must be processed and interpreted.

The shrinkage curve gives the relationship between void ratio and (volumetric) water content. The water content can be directly computed from the water mass in the sample and the dry soil volume. The void ratio depends strongly on what is considered 'void'. In section 3.3 three definitions of total soil volume were discussed as well as the computational procedure to derive their values. In figure 4.17 the important variables to obtain the total sample volume and void ratio are repeated.

Figure 4.17: The variables in the boxes can be derived from the data or are known. The subsequent steps can be taken to find the total sample volume and void ratio.

4.3.1. Variables derived from the scan data

From the scan data the variables \( h_{settlement}, r_{sample}, A_{sampleIII} \) and \( V_{soilmatrix} \) are derived as described in section 3.3.

\( h_{settlement} \)

In figure 4.18, \( h_{settlement} \) over time is given for the oedometer samples. The data is compared to the transducer data (i.e. the dotted lines). It is visible that the transducer data and the scan data fit quite well for the first 10 to 20 test days. Afterward, larger fluctuations show. Mainly the transducer data for samples 2 kPa and 22.5 kPa show deviant behaviour. After a certain point in time an increase in sample height is registered. For the samples 7.5 kPa and 15 kPa no such thing is observed. For generation of the shrinkage curves, \( h_{settlement} \) (from the scan data) is used.

The curves for samples subjected to 2 kPa and 22.5 kPa show the extremes of settlement behaviour. All samples experience a rapid decrease in height in the first test phase. The 22.5 kPa sample shows the steepest curve and reaches a more or less constant sample height \( h_{settlement} \) after 40 test days. The rate of settlement for the other samples approaches zero between 60 and 70 test days.

Image 4.18 justifies the corrections performed for figure 4.1. The final sample height is listed in table 4.3.

<table>
<thead>
<tr>
<th>Sample</th>
<th>( h_{set} ) [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 kPa</td>
<td>21.81</td>
</tr>
<tr>
<td>7.5 kPa</td>
<td>21.08</td>
</tr>
<tr>
<td>15 kPa</td>
<td>20.75</td>
</tr>
<tr>
<td>22.5 kPa</td>
<td>20.24</td>
</tr>
</tbody>
</table>

Table 4.3: Height oedometer samples after final settlement

\( r_{sample} \)

Due to the irregular shape of the oedometer samples, several options are available for \( r_{sample} \). In the methodology they are referred to as option I and II. In figure 4.19 the radii for the top and the bottom of the samples
4.3. Processed results

Figure 4.18: $h_{\text{settlement}}$ for the oedometer samples as derived from the scan data is compared to the (corrected) transducer data. The curves for samples 7.5 kPa and 15 kPa fit reasonably well. The curves for samples 2 kPa and 22.5 kPa show increasingly different behaviour.

are plotted over time. The green curves represent the radii at the bottom of the samples (option I). Here, the largest possible radius is taken. The shrinkage pattern becomes relevant. In section 4.2.4 is was shown that this pattern can be slightly arbitrary. The broad range of radii in figure 4.19 corresponds to this observation. The orange curves represent the sample radii at the top of the sample (option II)\(^3\). The sample radii at the top are smaller, which was also seen in section 4.2.4 in the vertical cross sections. All orange radii decrease linear until a more or less constant sample radius is reached. The green curves show a more gradual and less consistent shrinkage behaviour. It may be noted that both curves for sample 15 kPa are closer together than the other sample curves. This corresponds with the observation in section 4.2.4, where it was noted that sample 15 kPa shows more homogeneous horizontal shrinkage.

The horizontal shrinkage as presented in the orange curves is more consistent (e.g. less fluctuations) and is less influenced by the sample composition than that in the green curves. Therefore, for the subsequent analysis steps, the (orange) radii referred to as $r_{hv-II}$ are used.

$A_{\text{sampleIII}}$

$A_{\text{sampleIII}}$ are determined with the use of Avizo. The curves for area $A_{\text{sampleIII}}$ are similar to the area curves based on $r_{\text{sampleI}}$. The data set shows some unexpected fluctuations. In figure E2 $A_{\text{sampleIII}}$ is shown and compared to the other sample areas. After comparing the area curves, it was decided that the added value of using $A_{\text{sampleIII}}$ was limited. Further analyses are performed with a sample area based on $r_{\text{sampleI}}$ (figure 4.19).

$V_{\text{soil matrix}}$

From the CT scan data the soil matrix volume can be computed directly. The results are presented in next section, where total sample volume is discussed.

\(^3\)The radius was measured for a horizontal cross section 1.2 mm from the top of the sample.
4.3.2. $V_{\text{total}}$

In figure 4.20 the computed curves for the three total sample volumes are given. The extremes of the range are the curves for sample subjected to 2 kPa and 22.5 kPa. The first scan was made on test day eight, which is the start point of the scan data curves. The curves for $V_{\text{settlement}}$ are complemented by the transducer data for the first test days. The volume for sample 22.5 kPa decreases rapidly and becomes more or less steady after 40 test days for each of the volume definitions. The 2 kPa sample volume decreases more gradually and reached a more or less constant level after 65 test days. This observation corresponds to what was seen in the figures for $h_{\text{settlement}}$ (figure 4.18) and sample radius (figure 4.19).

The final total sample volumes for each definition are given in table 4.4.
4.4. The shrinkage curve

A common representation of shrinkage behaviour is the shrinkage curve, in which the changing void ratio and water content are plotted. In this section, the impact of overburden pressure is investigated by comparing the various experimental curves. Also the sensitivity of the experimental shrinkage curve results is tested and a comparison with curves from literature is made.

4.4.1. Experimental shrinkage curves

From the total volume and the dry soil and water volume, the void ratio and water content can be computed. The minimal void ratio for the three different volume definitions are given in table 4.5, as well as the final volumetric water content for this test.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$V_{\text{soil matrix}}$ [cm$^3$]</th>
<th>$V_{\text{settl}}$ [cm$^3$]</th>
<th>$V_{\theta}$ [cm$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 kPa</td>
<td>51.23</td>
<td>72.15</td>
<td>57.31</td>
</tr>
<tr>
<td>7.5 kPa</td>
<td>51.46</td>
<td>69.67</td>
<td>55.73</td>
</tr>
<tr>
<td>15 kPa</td>
<td>52.03</td>
<td>68.86</td>
<td>55.57</td>
</tr>
<tr>
<td>22.5 kPa</td>
<td>51.76</td>
<td>67.15</td>
<td>55.40</td>
</tr>
</tbody>
</table>

Table 4.5: Minimal void ratio for oedometer samples.

In figure 4.21 the shrinkage curve is plotted for samples 2 kPa and 22.5 kPa. The samples represent the extremes for the test. As a reference, the saturation line is included in the figure. The dots in the curve refer to a data point, the connecting lines are interpolations. Three sets of curves show, as a result of the volume definitions. At the start of the test, all curves follow the saturation line. The settlement curve for the 2 kPa sample deviates at a high water content of 111%, relative to the sample 22.5 kPa settlement curve, which starts to deviate at more of less 80% water content. The void ratio decreases gradual relative to the water content until a more or less constant minimal void ratio is reached. The minimal void ratio for the 2 kPa sample is 83%. For the 22.5 kPa sample the minimal void ratio is 68%.

The curves taking into account both horizontal and vertical shrinkage, more or less follow the saturation line for a longer stretch (relative to the settlement curves). The curve for 2 kPa deviates from the S line somewhere between at around 85% water content. The minimal void ratio is registered at 47%. For the 22.5 kPa sample, the curve deviates from the S line at around 60% water content. The minimal void ratio is 40%.

In contrast to the previous two sets of curves, the soil matrix curves show a similar behaviour for the samples subjected to the whole loading range. Both curves leave the saturation line at a water content of 33%. The minimal void ratio for the 2 kPa and 22.5 kPa samples are respectively 29% and 30%.

Some fluctuations are visible in the curves. In reality, the void ratio can never be smaller than the volumetric water content. When the curves dive below the saturation line, there must be some measurement error.

In table 4.6 the shrinkage limits for the oedometer samples are given.

In figure 4.3 the shrinkage curves for all samples is included. The figure supports what has been observed in the previous paragraph. Three different curve sets are identified, corresponding to the three different volume definitions. For the curves related to settlement and both horizontal and vertical shrinkage, a range for the samples subjected to different loads is observed. For the curves referring to the soil matrix, no such range is seen.
Figure 4.21: Shrinkage curve oedometer samples
4.4. The shrinkage curve

<table>
<thead>
<tr>
<th>Sample</th>
<th>$SL_{soil\ matrix}$</th>
<th>$SL_{sett}$</th>
<th>$SL_{h&amp;v}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 kPa</td>
<td>0.11</td>
<td>0.32</td>
<td>0.17</td>
</tr>
<tr>
<td>7.5 kPa</td>
<td>0.11</td>
<td>0.29</td>
<td>0.18</td>
</tr>
<tr>
<td>15 kPa</td>
<td>0.11</td>
<td>0.27</td>
<td>0.15</td>
</tr>
<tr>
<td>22.5 kPa</td>
<td>0.11</td>
<td>0.26</td>
<td>0.15</td>
</tr>
</tbody>
</table>

Table 4.6: Shrinkage limit oedometer samples in gravimetric water content.

In figure 4.22 the shrinkage curve is given for the hyprop sample. The test is continued until a volumetric water content was reached of 14 %. Similar to the oedometer sample shrinkage curves, three different curves are found. The hyprop sample was subjected to 2 kPa. When compared to the oedometer 2 kPa sample, the hyprop curves show some differences. The settlement curve and horizontal & vertical shrinkage curve are not on the saturation line at the start of the test. This may be explained by the volume of the tensiometers, that was not taken into account. Also the final void ratio for the two curves is higher than what was found for the oedometer sample. At the end of the test, the residual water content was not yet reached. Therefore the final void ratio and shrinkage limits are derived from extrapolation (tables 4.7 and 4.8). The soil matrix data is not complete due to an scanning error. The first four data points are corrected as indicated in figure 4.22. The minimal void ratio for the soil matrix is equal to the oedometer sample minimal void ratio.

![Shrinkage curve hyprop sample](image)

Figure 4.22: Shrinkage curve hyprop sample

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\varepsilon_{soil\ matrix}$</th>
<th>$\varepsilon_{sett}$</th>
<th>$\varepsilon_{h&amp;v}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>hyprop 2 kPa</td>
<td>0.30</td>
<td>0.93</td>
<td>0.58</td>
</tr>
</tbody>
</table>

Table 4.7: Extrapolated values for void ratio hyprop sample

<table>
<thead>
<tr>
<th>Sample</th>
<th>$SL_{soil\ matrix}$</th>
<th>$SL_{sett}$</th>
<th>$SL_{h&amp;v}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>hyprop 2 kPa</td>
<td>0.11</td>
<td>0.34</td>
<td>0.22</td>
</tr>
</tbody>
</table>

Table 4.8: Shrinkage limit hyprop sample in gravimetric water content
4.4.2. Microscan analysis

The microscan analysis as described in chapter 3 is performed for samples 2 kPa and 22.5 kPa. The data is compared to the CT scan data. The results are given in table 4.9. For an accuracy of 10 µm a small air volume is found, indicating that only a very small percentage of the voids smaller that the scan accuracy is filled with air.

Because only one microscan is performed, per sample only one data point is available. From the data is can be concluded that the sample is still saturated when perceived on micro-scale. Because only one microscan is performed, it is unknown whether this level of water content can be seen as the shrinkage level or not.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Microscan</th>
<th>CT scan</th>
<th>CT total</th>
<th>%</th>
<th>V_w</th>
<th>V_s</th>
<th>V_a-micro</th>
<th>V_a-CT</th>
<th>ε_micro</th>
<th>ε_CT</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 kPa</td>
<td>0,45</td>
<td>0,52</td>
<td>51,23</td>
<td>1,02</td>
<td>0,04</td>
<td>0,41</td>
<td>-1,09E-03</td>
<td>7,11</td>
<td>0,10</td>
<td>0,29</td>
</tr>
<tr>
<td>22,5 kPa</td>
<td>0,45</td>
<td>0,52</td>
<td>51,76</td>
<td>1,01</td>
<td>0,04</td>
<td>0,40</td>
<td>7,62E-04</td>
<td>7,64</td>
<td>0,11</td>
<td>0,30</td>
</tr>
</tbody>
</table>

Table 4.9: Microscan volume analysis

In figure 4.23 a representation of the microscan data is given from the middle of sample 2 kPa. In the right top corner, a fragment of a CT scan is inserted. Both fragments represent the same sample height. After comparing the two accuracies, the large difference between void ratio's ε_micro and ε_CT is better understood.

4.4.3. Sensitivity analysis

In chapter 3 the motivation for the sensitivity analysis is addressed. For r_sampl, the default accuracy was already known as well as for the scan data. For the variables h_settle, r_sampl and V_sm an additional investigation was done.

These variables are derived by means of visual analysis, which entails a certain degree of subjectivity. For each of the variables, the analysis was repeated, for h_settle, trice, for r_sample and V_sm twice. Based on the range of values, the accuracy was determined. For h_settle, a range was found between -0.51 and +0.55 mm. The default scan accuracy is 0.3 mm. Since the top and bottom are measured, the range increases to 0.6 mm. For the sensitivity analysis the range -0.6 mm < h_settle > 0.6 mm is taken into account.

A similar analysis is done for r_sample. Here too, the default accuracy of 0.3 mm was larger than the range found for the additional investigation. The range -0.3 mm < r_sample > 0.3 mm is taken into account. For V_sm the largest range for a sample was 230 mm³. For the sensitivity analysis, this value is taken into account. In table 4.10 the assumed accuracies are listed.

In figure 4.24 the results of the sensitivity analysis for the extremes in the test are given. For each separate variable, a shrinkage curve is plotted for the accuracy range maximum and minimum. h_settle has the largest range and this translates to a large range for the shrinkage curves. Yet even for the most extreme scenario, the difference in minimal void ratio between samples 2 kPa and 22.5 is substantial.
4.4. The shrinkage curve

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$r_{ring}$</td>
<td>0.01 mm</td>
</tr>
<tr>
<td>$h_{settle}$</td>
<td>0.6 mm</td>
</tr>
<tr>
<td>$r_{sample}$</td>
<td>0.3 mm</td>
</tr>
<tr>
<td>$V_{sm}$</td>
<td>230 mm$^3$</td>
</tr>
</tbody>
</table>

Table 4.10: Range for relevant parameters / variables in determining the shrinkage curve.

In figure 4.24 the sensitivity analysis for oedometer samples subjected to 2 and 22.5 kPa.

4.4.4. Verification of the experimental shrinkage curve

In figure 4.25 the experimental results for samples 2 kPa and 22.5 kPa are compared to a typical shrinkage curve as presented by Cornelis et al. (2006). In chapter 2 the stages as indicated in the figure are elaborated. The experimental curves for $e_{settle}$ and $e_{soilmatrix}$ display a behaviour similar to the curve from literature. The curves follow the saturation line up to an air entry point, after which the curve deviates. In the final stage, a shrinkage limit is reached and void ratio no longer decreases.

The $e_{h\&v}$ curve for sample 22.5 kPa shows a slightly different behaviour in the final shrinkage stage. The characteristic horizontal end of the curve, indicating the shrinkage limit, is not seen. Instead, the void ratio drops slightly, even though the water content approaches the residual water content.
4. Results

Figure 4.25: Shrinkage curve compared to literature. left: experimental shrinkage curve results, corrected for values in shaded area. right: theoretical shrinkage curve after Cornelis et al. (2006).

Figure 4.26: Shrinkage curve compared to previous findings. left: experimental shrinkage curve results. right: experimental shrinkage curve for MFT after Yao et al. (2014).
In figure 4.26 the shrinkage results are compared to previous experimental work performed on Mature Fine Tailings (MFT) by Yao et al. (2014). The MFT shrinkage test was performed with a balloon test under atmospheric pressure, yielding results similar to the shrinkage test for the volume definition $V_{hkv-shrinkage}$ in this thesis. The minimal void ratio by Yao et al. (2014) is slightly lower than expected based on the thesis test results for reference sample 2 kPa.

### 4.5. Verification of numerical results

In the shrinkage curve based on the oedometer samples, for some volume definitions, impact due to overburden pressure is observed as a decrease of minimal void ratio with respect to the reference curve. In an attempt to quantify the impact, the method proposed in the Sludge Ripening Model by Vardon and Van Tol (2014) is investigated. If in fact the impact can be predicted by the proposed formulation, this yields a valuable reference point for future layered deposition modelling.

The oedometer experimental results are used to verify the proposed relationship in the Sludge Ripening Model by Vardon and Van Tol (2014) as described in chapter 3. The experimental minimal void ratio's for samples subjected to 7.5, 15 and 22.5 kPa are taken as $a_{new}^{old}$ and the minimal void ratio for the 2 kPa sample as $a_{sh}^{old}$ (i.e. the reference sample) in equation 3.9. In equations 3.7 and 3.8 it is suggested that the coefficient of the terms $(\Delta a/a_0)$ and $log(a'/a_0)$ is given by $(1/C_{10})$. If, from the plotted experimental data, such a linear relationship is found, the coefficient (and $C_{10}$) can be derived.

For volume definitions $e_{settlement}$ and $e_{hkv-shrinkage}$, an individual compression constant $C_{10}$ is expected. For $e_{soilmatrix}$ no change in minimal void ratio is detected for a range of applied overburden pressures. Therefore, all values for $(\Delta a/a_0)$ are equal for the stress range, yielding an infinite coefficient $C_{10}^4$ (figure 4.27).

No perfect linear relationship was found for the $e_{settlement}$ and $e_{hkv-shrinkage}$ data sets. A linear regression model was made, for which coefficient of determination $R^2$ can be given. Based on the linear approximations, values for $C_{10}$ are computed and given in table 4.11. Both values for $R^2$ approach 1, indicating a good fit. The data set is small, therefore the reliability of this value is limited.

The plotted experimental results for $e_{settlement}$ and $e_{hkv-shrinkage}$ and their approximations are presented in figures 4.28 and 4.29, respectively.

In figure 4.27 the suspicion is confirmed that the relationship between minimal void ratio and overburden pressure in the numerical model is not applicable for the volume definition including only the soil matrix.

<table>
<thead>
<tr>
<th>$e$</th>
<th>$C_{10}$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$e_{sm}$</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$e_{sett}$</td>
<td>10.19</td>
<td>0.993</td>
</tr>
<tr>
<td>$e_{hkv}$</td>
<td>7.93</td>
<td>0.953</td>
</tr>
</tbody>
</table>

Table 4.11: $C_{10}$ values based on the minimal void ratio derived from experimental oedometer tests

$^4C_{10}$ is the coefficient of $x$ over $y$. 

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This material is based upon work supported by the National Science Foundation under Grant No. 1547066.
Figure 4.27: For $e_{\text{soil matrix}}$ no relationship between overburden pressure and void ratio is observed.

In figure 4.28 a good linear relationship is detected. The linear approximation is made to find a single value for $C_{10}$. Based on the figure, it seems possible to predict a minimal void ratio for $e_{\text{settlement}}$ based on $C_{10}$ and a known overburden pressure.

Figure 4.28: Determining $C_{10}$ for $e_{\text{settlement}}$

For the $e_{\text{hkr}}$ data set also an acceptable linear approximation can be made with a coefficient of determination $R^2$ approaching 1. The deviation of the data points is larger than for the $e_{\text{settlement}}$ data set, yielding larger prediction errors (or a root-mean-square error).
4.6. Validation of the results

The main research question was initiated by players in the engineering practice, thereby questioning the assumption of “no impact” made often in the scientific community. The value of the thesis results can be assessed by the applicability and relevance in the scientific community and the engineering practice. Based on the obtained shrinkage curves as presented in the previous section, it becomes clear that before a final conclusions can be drawn on the existence of impact, the relevance of each volume definition must be assessed for the different disciplines. Discussion on the relevance of volume definitions per discipline and the validity of the thesis is deferred to the subsequent chapter.

4.7. Concluding remarks

In order to answer the main research question, fine-grained soil behaviour is investigated experimentally as the material dries while subjected to overburden pressure. This is done by means of a shrinkage curve, where change in void ratio is plotted against change in water content. All test results are interpreted in order to compute this curve. In this chapter, all test results are presented and described in chronological order to give insight in followed procedure. This should not diminish the importance of the main figure, the shrinkage curve.

The shrinkage results are subjected to additional analyses. A comparison is made with micro scan, to investigate the relativity of the definitions for the soil matrix. Also, the sensitivity of the shrinkage curve for the accuracy of the parameters and variables is examined. The experimental results were verified by comparing the curve to literature and previous findings.

In the results some impact was observed for two of the three volume definitions (as given in chapter 3). This was taken as a confirmation that in the numerical model for layered slurry deposition some relationship can be assumed. The experimental data is used to verify the proposed mathematical formulation of the impact. In the subsequent chapter, the results are subjected to an elaborate discussion, in which the meaning and interrelationships of the findings are further examined. Preliminary conclusions and limitations of the research are addressed. Based on the discussion, the value of the thesis is determined.
Discussion

5.1. Introduction
In the previous chapter, the results derived from the experimental work and data interpretation, as instructed in the methodology, were presented and discussed briefly. In this chapter, the discussion is continued in an attempt to account for and interpret further the results and their main drivers. This chapter should provide the grounds to draw conclusions and to give an answer to the main research question in this thesis.

5.2. Discussion of the methodology
In this section, the methodology is discussed. The execution of the tests and data interpretation has provided insight on advantages of and objections to the choices made in advance. The experimental test phase and the interpretation phase are discussed separately.

5.2.1. Experimental work
Dry oedometer tests
The oedometer test frames are designed to apply vertical compression to a confined sample. In saturated conditions, the test is seen as an element test. The impact of overburden pressure can be simulated for the unsaturated case. Due to the discrete unsaturated shrinkage process, the samples can no longer be seen as a single element.
Based on the oedometer test results alone, shrinkage can only be determined for the volume definition $V_{\text{settlement}}$, given that the transducer data is reliable. For other definitions of volume and decrease of water mass, additional measurements, such as scanning and weighing, are done to gather the results. These measurements require that the test is stopped, the samples are unloaded and reloaded as the test continues. These intervals influence the test procedure and the results (e.g. after unloading and reloading a volume increase is detected). The more measurement intervals, the more accurate the registration of the shrinkage. However, the more disturbance is caused in the oedometer test results. The oedometer test is therefore not completely suitable as a shrinkage test but was at the time considered the best option available.

In the test set up, sample preparation and during the experiments, some elements might be adjusted to yield better results.
The duration of the test is determined by the consolidation and evaporation rate. The former is influenced by the applied loading. The latter is influenced by the allowed evaporation from the sample and the test conditions (e.g. temperature, relative humidity). The test sample was closed off with a bottom plate to contain the near liquid sample. Only evaporation from the top was allowed through the loading cap. During the experiment, small tests were performed with other options. The use of a filter paper fastened to the ring instead of a bottom plate proofed effective to contain the sample and could be used in future testing.
The oedometer frames were set up in a climate room to ensure the safety of the set up and create constant test conditions. The conditions resulted in a relatively low evaporation rate (e.g. compared to the hyprop test). A separate transducer was connected to the oedometer frames and the weight hangers were supported by a single pin. This proofed too unstable a set up for a situation where samples had to be retrieved often.
5. Discussion

The risk of measurement errors, due to displaced sensors and deformed equipment, as well as accidents was significant.

From the results the relative importance of the preparation of homogeneous samples becomes clear. The horizontal shrinkage behaviour of the sample is strongly influenced by existing air pockets and fissures. More is said on the subject in the section 5.3.1. Although care was taken to limit the amount of air pockets in the samples, the results show that this still occurred. Options for improvement are to apply even smaller quantities of soil at a time and use more appropriate tools than a spatula for the application. Also the initial soil water content can be increased. The latter will influence the loading moment in the test procedure. Due to the liquid state of the clay, the strength was too little to apply the full overburden pressure. This is inherent to the desire to investigate slurry behaviour. In the interpretation of the results, this delay and the subsequent deviant behaviour must be taken into account.

Hyprop test

The hyprop test is useful to quantify the suction development in a soil sample. However, for the objective of this test, the equipment is not appropriate. The default scale is not suitable for a larger applied overburden pressure than 2 kPa, limiting the test possibilities. The main objection to the test is the presence of the tensiometers in the soil, that influences the shrinkage behaviour and the transferring of the load.

The preparation and execution of the test can also influence the results. To increase the homogeneity of the soil, after preparation the sample was placed in a vacuum pump. From CT scans it becomes clear that a large crack was formed in the sample. This crack defined the shrinkage behaviour during the subsequent test days. It originated as air bubbles were removed from the sample. Better results are achieved with the vacuum pump for more homogeneous or more liquid samples.

The relative flexibility of the PVC ring caused a delay in the loading of the sample. The ring deformed as the sample was secured to the hyprop cell. Due to this deformation, the porous plate no longer fit and had to be resized, causing a one day delay in the loading of the sample. In this time, the test was not stopped and evaporation had started to desaturate the sample. The measured results would have been more valuable if the start of the test and the moment of loading had coincided. In retrospect, it might have been better to terminate the test and start over after the adjustment of the porous plate.

The first four (out of seven) CT scans mistakenly did not capture the entire sample. Therefore, variables based on these data sets all had to be corrected for the missing sample volume.

5.2.2. Data processing and interpretation

The assumption that three definitions for sample volume can capture soil behaviour is reviewed. The definitions of sample volume are meant to describe in increasing detail the shrinkage behaviour of soil. The definition of vertical settlement volume applies to the engineering practice, where soil volume change is treated as consolidation on a macroscopic level. \( V_{\text{hker-shrinkage}} \) relates to the sample volume that would be obtained if Archimedes’ principle was applied. It refers to what is generally conceived as soil shrinkage. The soil volume decreases more or less isotropically, the larger cracks are excluded from the soil sample and the smaller cracks are considered part of the sample. Standard shrinkage tests such as the balloon test (Tariq and Durnford (1993)) implicitly makes use of this definition.

The third definition zooms in on the true soil fabric characteristics. Results for this definition give information on the inter-aggregate pore drainage due to shrinkage. The shrinkage behaviour is unaffected by larger soil-structural factors (such as the formation of air pockets and cracks due to sample preparation).

An experiment is performed in order to see whether analysis on a smaller scale is useful. On the final test day the samples 2 kPa and 22.5 kPa are scanned with an accuracy of 10 \( \mu \)m. The samples have reached their residual water content and can still be considered saturated. At the end of the test, no unsaturated shrinkage is detected on this scale. Although this is an interesting finding, it is felt that discussing soil behaviour structurally on this scale is not meaningful in the context of this thesis.

The use of three definitions of soil volume ensures a complete enough description of soil behaviour during unsaturated shrinkage for this thesis.

A visual analysis of the CT scan data is used to find each of the defined sample volumes. The choice for this type of analysis had advantages and disadvantages. The main advantages are ease of use and control of output. With the latter is meant that the method is similar to how one would measure the sample with a caliper. The results are easily ‘sanity checked’. A disadvantage is that the execution of the analysis is time
5.3. Discussion of the results

In this section, a more detailed analysis of the thesis results is made. To increase the ease of reading, the division of the subjects is similar to that of chapter 4, starting with the experimental results. If necessary, cross links between results will be made and expressly mentioned.

5.3.1. Experimental results

Consolidation and evaporation

In the curves, presented in figure 4.1, the vertical settlement of the samples 2 kPa and 22,5 kPa is given. The settlement reduction visible for most measurement intervals are related to the relieve of the sample from its load. The sample is compressed and during unloading, air may enter the drained pores, causing a temporary rise of the sample volume.

A main issue of discussion, when regarding the vertical settlement of the samples, is the influence of consolidation versus evaporation. In the curves some similarities and differences from a typical consolidation curve for a fine-grained soil may be found. The curvature for samples 22,5 and 15 kPa is affected by the delayed placement of the additional load on $t_{load}$. The initial curve, visible for samples 2 kPa and 7,5 is similar to the shape of the default curve. At this time, the sample is still saturated and consolidation is driving the sample volume change (i.e. settlement). After the upper curve, the sample curves are decreasing linear (on the log scale), also similar to the default consolidation curve. However, the characteristc S-curve for primary and secondary consolidation is not quite visible. As time progresses, water is removed from of the samples by evaporation as well as compression. Capillary pressure develops and matric suction reaches the point at which pore water in the samples is displaced by air and the sample desaturates further. Over time the unsaturated hydraulic conductivity $k$, depending among other things on the soil porosity and degree of saturation, decreases according to the equations in section 2.4.1. The contribution of consolidation decreases and suction drives the final volume change, until the residual water content is reached. This explains the increasing difference in behaviour between saturated and unsaturated consolidation.

It must be noted that the past paragraph is based on the concept of a homogeneous sample. From the scan data results it can be concluded that this is not necessarily the case as the sample dries and cracks, influencing $k$ and $S$. This does not mean the description becomes untrue in general, but it is no longer a description of a specific sample.

Differences in material, stress history, loading and initial water content are all relevant for assessing the precise influence of consolidation and evaporation. Additional data on the saturated consolidation of the soil under similar loads would facilitate quantitative analyses.

Dewatering

The influence of consolidation and evaporation can seen in figures 4.2 and 4.3. The change in volume is caused by the loss of water in the soil. The curves in figure 4.2 are similar to the settlement curves. After the initial effect of the loading, the sample subjected to 2 kPa shows a gradual decrease in water mass. The other samples show a more pronounced response to the (larger) load. The differences between samples 7,5 , 15 and 22,5 kPa on the first day are caused by the small difference in initial loading. More water is displaced after additional loading, corresponding to the extend of the load. This is confirmed by the peaks showing for samples 22,5 kPa and 15 kPa on the second and third test day in figure 4.3. On test day three displaced (surface) water is visible on the samples as they are weighted. After day four, the largest surplus of water has been displaced (based on figure 4.3). As the samples dry, they show similar behavioural phases, albeit for different time periods. In figure 5.1 the phases that are referred to are indicated with orange lines for the samples 2 kPa and 22,5 kPa. Initially, during consolidation water is driven out of the soil. Then, the influence of evaporation and suction increases as the sample volume decreases, and water is displaced at a linear pace. Finally, the water phase in the soil is no longer continuous and only evaporation drives further loss of water mass. The greater the influence of consolidation (e.g. the larger the load) the smaller the water content as the second phase commences. This phase is therefore shortened as the water phase becomes discontinuous and evaporation is governing. At last a residual water content, depending on test conditions, is reached for all samples.
In the description of figure 4.2 the development of a gap was discussed. The behaviour of the extremes is explained with the phases in figure 5.1. For sample 7.5 the behaviour, after the dissipation of excess pore pressure, becomes similar to that of sample 2 kPa. In the figure for change of mass, the initial effect of consolidation determines the difference between two samples until the end of the test. For sample 15 kPa, the initial behaviour is similar to that of sample 22.5 kPa due to consolidation. After the impact wears off, the dewatering rate drops low (test day 8). At test day 10, the load cap of sample 15 kPa was changed for one more similar to the others. This new cap allowed more evaporation. At test day 10, the dewatering rate becomes equal to that of sample 2 and 7.5 kPa. It seems that the first cap caused a ‘delay’ in the dewatering that influences the sample behaviour during the rest of the test. In retrospect, changing the conditions during the test is not ideal, for one has to speculate on the impact before and after.

At the dashed line in figure 5.1 the second of two larger fluctuation in dewatering is visible for all samples. These fluctuations correspond the first two scan moments, where the samples are unloaded and exposed to a different climate for approximately 30 minutes. The impact of unloading and reloading (visible as the decrease and steep increase in the dewatering rate) confirms that at this time, the effect of the consolidation is not yet over. At later scan moments (e.g. table D.1), no longer such fluctuations are observed for all samples. In the figure, for sample 15 kPa, a steep decrease in dewatering rate and little mass change is observed.
For the hyprop sample, also the water mass change (figure 4.4) and dewatering rate (figure 4.5) are analysed. Although the additional loading is equal to the 2 kPa oedometer sample, no quantitative comparison can be made due to differing test conditions and timing. As described in chapter 3, the temperature and humidity in the hyprop climate room were more favorable to evaporation. Also, due to the deformation of the test equipment, the loading was placed on test day two (instead of day one). Evaporation was allowed from the first test day. In figure 4.5 the impact is visible. High evaporation rates are deduced during the first day, as full surface evaporation is allowed. The gap in the figure represents the placement of the load. From this moment on, a significantly lower dewatering rate gradually approaches zero over time. This figure gives information on the limitation of evaporation due to the placement of the porous plate and loading. Due to the initial evaporation (decrease in water content) and desaturation (temporary increase of effective stress) of the sample, the impact of the consolidation starting on the second day has become small.

In figure 5.2, a qualitative comparison was made for the two samples. In the figure, the dashed line indicates the moment in time that the load and porous plate were placed on the hyprop sample. The oedometer curves is scaled to start on the second test day. The behaviour deviates most in the beginning of both tests. The surplus of the oedometer sample water is driven out by consolidation as observed by the peak in the curve in the dewatering rate. For the hyprop, the impact is limited. Over time, the behaviour of the samples becomes more similar, as evaporation and suction development become governing.

Soil Water Characteristic relationship
In figure 5.3 the Soil Water Characteristic Curve (SWCC) for the hyprop sample is compared to literature. The curves for clay are similar in the sense that suction builds up to a certain point, after which the water content drops. The suction develops linear (on the log scale). The hyprop curve is extrapolated based on soil water characteristic behaviour after Fredlund et al. (2002b). The hyprop test was stopped before a residual water
content $\theta_r$ was reached, therefore in the figure a value, slightly lower than the one found for the oedometer samples, is assumed.

At the time the load is applied, suction has already been registered and the water content has decreased. The small reduction in suction (i.e. increase in pore pressure due to the loading) in the figure corresponds with the limited consolidation effect on the sample that was seen in figure 5.3.

![Diagram of SWCC clay (literature) and SWCC sand (literature)](image)

**Figure 5.3:** Soil Water Characteristic Curve. (a) SWCC after Fredlund et al. (2002b). (b) SWCC for hyprop sample.

In chapter 2 mention was made on the limitations of the SWCC with respect to the stress state of the soil and volume definition. If the curve is given for degree of saturation $S$ instead of water content, the aspect of volume change of a sample is taken into account in the term for porosity\(^1\). For the various definitions of volume the Soil Water Characteristic Curve can in theory be compared for impact of overburden pressure.

For the hyprop test not enough suction data is available to create a meaningful SWCC for degree of saturation. Also, since only one test is performed, no comparison can be made between samples with a range of loads. Finally, the results are not reliable with respect to the impact of overburden pressure due to the influence of the tensiometer (as was discussed in section 5.2).

For the oedometer samples the suction was not measured. Based on other results and literature some comments can be made on process in the samples.

According to consolidation theory, applied overburden pressure generates pore water pressure, that are slowly dissipated by the (limited) compression of water and the outflow of water. For the contribution of consolidation to volume change, the soil (matrix) may be considered saturated. At the same time, evaporation is allowed at the surface and capillary pressures build up. Suction develop and the soil desaturates. If the stress state is described with equation 2.5 for intergranular stress, difficulties arise for the factor $\chi$ and the state and definition of degree of saturation of the samples.

This ambiguity translates to the different shrinkage curves that were found in figure 4.21. A relationship of applied pressure and volume change was observed for volume definitions $V_{settlement}$ and $V_{\text{hyprop shrinkage}}$. For $V_{\text{soil matrix}}$, no difference in shrinkage curves was seen for the range of samples.

The fact that in the soil matrix no impact is seen, indicates that the processes of consolidation and evaporation balance each other out in reference to volume change. Both 'external' compression and 'internal' tension lead to the same minimal void ratio in the soil matrix. The contribution of either process depends on the applied load. If a sample is subjected to a higher load, the volume changes more due to consolidation and less due to suction driven shrinkage relative to a sample with a smaller load. Additional experimental tests may show whether in fact less tension is measured over time in the former sample.

The main influence of the shift in contribution of either process due to load lies in the configuration of the soil sample. This is discussed in more detail in the following section.

\(^{1}(S = \theta/n)$
5.3. Discussion of the results

Scan data
In the previous sections, the samples are considered and discussed as elements that show homogeneous behaviour. This is done to simplify the discussion and to make the link towards literature. In this section, more attention will be paid to the particularities of the samples.

From the scan data presented in the previous chapter, it became clear that the general shrinkage behaviour of the samples is similar but that the specific configuration for each sample is arbitrary. With general behaviour is meant that the sample volume decreases according to expectation: consolidation and more or less isotropic shrinkage from all surfaces of the sample is observed. The configuration of cracks depends on the initial soil structure or preparation of the sample.

In figure 5.4 the various observed behaviour types for shrinkage are illustrated based on a schematized sample. All shrinkage occurs from a surface where evaporation may occur. For the test samples, all shrinkage types are observed.

<table>
<thead>
<tr>
<th>Original sample</th>
<th>Shrinking sample</th>
<th>Description of shrinkage behaviour</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1" alt="Original sample" /></td>
<td><img src="image2" alt="Shrinking sample" /></td>
<td>'Homogeneous' contraction of sample (from surface) &amp; migration of air pockets</td>
</tr>
<tr>
<td><img src="image3" alt="Original sample" /></td>
<td><img src="image4" alt="Shrinking sample" /></td>
<td>Contraction of sample (from surface) &amp; 'short cut' development</td>
</tr>
<tr>
<td><img src="image5" alt="Original sample" /></td>
<td><img src="image6" alt="Shrinking sample" /></td>
<td>Development of cracks from fissures &amp; expansion of air pockets (from 'internal surface')</td>
</tr>
</tbody>
</table>

Figure 5.4: Observed shrinkage behaviour types for a schematized sample

**Oedometer samples** In figure 5.5 the shrinkage pattern for the final test day of sample 22.5 kPa is compared with earlier horizontal cross section as presented in chapter 4. The origin of many later cracks can be traced back to air pockets and fissures in the sample on the first scan day. These air pockets are most likely products of the sample preparation.

An argument for this assumption is the presence of circular fissures that develop into cracks, which may be a result of the layered application of the material in the sample ring. The other samples do not show similar internal cracks, indicating that it is not a standard shrinkage configuration. In figure 5.6, the origin and development of cracks are indicated with red arrows.

Another argument originates from theory. During consolidation, the sample is compressed and no new air pockets will develop. When the sample starts to desaturate, isotropic tension develops, from the surface inwards. The air pockets in this case are also a surface. As suction develops further, the effective soil stress
increases, preventing new cracks in the sample. Instead, as the sample shrinks, the existing cracks and air pockets expand. If large air pockets or fissures are present near the edge of the sample, the main sample volume can contract from this potential crack surface instead of the actual sample surface. The resistance to crack formation here is relative low. Also, the formation of the crack surface path is aided by the friction between the plastic ring and the soil. This is the case for sample 22.5 kPa. In figure 5.7 the development of cracks in sample 15 kPa is presented. The initial sample is more homogeneous than the 22.5 kPa sample, resulting in more homogeneous shrinkage behaviour.

In figure 5.8 the difference in shrinkage between samples 22.5 and 15 kPa in vertical direction is clear. The homogeneous 15 kPa sample shrinks evenly over the entire sample height (i.e. a constant diameter). The 22.5 kPa sample dilates at the bottom. Again it is visible that small fissures can develop into large cracks. It may be noted that all samples have an intact sample top. This can be attributed to the filter paper that was placed on top of the sample at the beginning of the test. The filter paper stuck to the sample and provides enough support for the top not to crack.

Figure 5.5: Crack development over time in sample 22.5 kPa. f.l.t.r. 28-07-15, 18-08-15, 13-10-15

Figure 5.6: Crack development and shrinkage behaviour in sample 22.5 kPa. The cracks visible on the final test day are placed on an image of the sample at the beginning of the test. The red arrows indicate the origins of cracks and air pockets.
5.3. Discussion of the results

Figure 5.7: Crack development and shrinkage behaviour in sample 15 kPa. The cracks visible on the final test day are placed on an image of the sample at the beginning of the test. The red arrows indicate the origins of cracks and air pockets.

Figure 5.8: Crack development and shrinkage behaviour for sample 22.5 kPa (top) and 15 kPa (bottom).

**Hyprop sample**  In the hyprop sample images a similar crack development is visible. A crack is present at the beginning of the test and develops over time. Initially, the soil separated by the crack, attaches to the sample ring, indicating that the ring exerts some attraction on the soil. An explanation may be that the PVC ring surface is less smooth than the default metal ring. As the soil dries further, the separated soil crumbles, as is visible in figure 4.16c.

For the same figure, it was noted that the sample hangs from the tensiometer. At this point, the added load is supported only partially by the sample (figure 5.9). The results are therefore not comparable to the oedometer samples and less relevant for the thesis objective, where the impact of overburden pressure is investigated.
This does explain possible differences in the shrinkage behaviour between the experimental test results. For example, in the hyprop shrinkage curve, slightly higher minimal void ratio's were found for the sample compared to the 2 kPa oedometer sample. This may be explained by the fact that the sample was not loaded to the same extent.

5.3.2. Result interpretation

$h_{settle}$ versus transducer data

In figure 4.18 the oedometer results from the visual analysis for $h_{settle}$ and the transducer results are compared. It was noted that the curves fit well in the first 10 or so test days and then start to deviate. Also it was seen that for samples 2 kPa and 22.5 kPa the transducer values indicate a swelling of the sample, where from the scan data no such thing was observed. The transducer values for the samples 7.5 kPa and 15 kPa however give fairly similar results as the visual analysis.

Several explanations are given. A theory would be that the samples 22.5 and 2 kPa actually show an increase in height as a transducer would measure it, for example because the top rims curl up. This curl was seen for sample 2kPa, however not for sample 22.5 kPa (figure 4.14). Also, a curl was observed for sample 7.5 kPa, where no rise in $h_{settle}$ was recorded. Another counterargument is that for the visual analysis, the sample height was assessed from the very top of the sample, thereby taking into account possible surface curling.

Another theory is that horizontal shrinkage influences the sample volume in a way that does not show on the scan data. A backing for this possibility is that the transducer data start to deviate around the time that horizontal shrinkage is first registered for samples 22.5 kPa and 2 kPa. However, it does not seem likely that the unloading of sample 22.5 kPa causes an instant decrease in sample height.

A final theory is that the mechanical equipment over time alters position during the repeated process of unloading, weighing and reloading the sample. The transducer is fastened onto the frame with a bolt. On the same frame the weight hanger is placed supported only by a pin. The transducer sensor gains degrees of freedom as the bolt is loosened over time and the frame is unloaded and reloaded. The movement that is allowed for the weight hanger (due to the connection at one point) contributes to the process. Finally, also the surface on which the sensor rests may not be completely level, causing measurements errors at the replacement of the sample (and sensor). The impact of these alterations may differ per oedometer frame, explaining the differences in transducer data accuracy when compared to the visual analysis data. This seems to be the most probable theory.

Based on the above and figure 4.18, it is justified to continue the research with the $h_{settle}$ data set (as opposed to the transducer data set). The quality of the initial transducer measurements is good enough to complement the visual analysis data for the days prior to the first scan moment.
Sample area
In chapter 4 several sample radii were compared to find a representative sample area for the computation of $V_{vertical \& horizontal}$. It was argued that the sample top was most representative for the results were least affected by the sample preparation process. However, in section 5.3.1 it was observed that this area is mostly homogeneous because of the presence of a filter paper. This raises the question whether this area is in fact representative.

An counterargument is that due to the cohesion supplied by the filter paper, the sample behaviour at the top is not representative for 'natural' behaviour, where cracks are expected to form top down. In a natural soil, the soil structure is also a major influence for crack formation. The area at the bottom of the samples would in this sense be more appropriate.

An advantage however for the use of the area at the top is that the sample conditions are most similar. The filter papers all are the same size, therefore area differences in the samples are due to the conditions that are investigated in this thesis, not a result of chance.

Since in this thesis the sample are compared for their general behaviour and not individual crack formation, the use of top area was deemed most suitable.

Ratio sample height to area
In the previous sections it became clear that for the range of samples, the impact of consolidation versus suction driven shrinkage shifts, yielding different results. When simplifying this to consolidation in vertical direction versus horizontal shrinkage, it may be observed that the sample with the highest load, 22.5 kPa, has exhibited the largest decrease in sample height. Meanwhile, the 22.5 kPa sample has the relatively the largest sample area. For the sample with the minimum load, 2 kPa, the opposite is true. The sample height $h_{settlement}$ is the largest, but the sample area is smallest. The other samples also display behaviour corresponding to their loading.

The total volume calculated from $h_{settlement}$ and $A_{sample}$ also has the samples 22.5 kPa and 2 kPa for extremes. For the range that has been investigated in this thesis applies that the larger the load, the greater the total volume reduction.

5.3.3. The shrinkage curve
The main objective for this thesis is to assess the impact of overburden pressure on the unsaturated shrinkage behaviour of fine-grained soil.

In chapter 2 was mentioned that the shrinkage curve is often considered characteristic for a material. In figure 4.21 was seen that the uniqueness of the shrinkage curve depends on which definition of soil volume is taken into account.

In the previous paragraphs, the contribution of consolidation and suction driven shrinkage is discussed as well as the contradictory properties of the processes with respect to saturation. In figure 5.10 the shrinkage curve for samples 22.5 and 2 kPa is given. Depending on the definition, volume change of a sample is saturated or unsaturated at the same time (measured in volumetric water content). In small diagrams the soil behaviour at the various moments of soil 'becoming unsaturated' (i.e. curve deviates from saturation line) are represented.

Important to realize is that the soil sample does not behave as a single homogeneous element that is either saturated or unsaturated, either consolidates or develops suction due to evaporation. At most one could state that one of the processes predominates the behaviour.

The contribution of either process is determined by the applied overburden pressure. On a soil matrix level, the actual volume change is not impacted. For the other two definitions of volume, it is clear that overburden pressure influences the configuration of the sample.

An example is given for the curves for sample 22.5 kPa. The sample is loaded vertical. Water is driven out of the soil by build up of pore pressure and evaporation. Vertical strain develops. No change in horizontal strain is allowed due to the ring. Due to the load, the main sample remains saturated. At a certain moment, due to the evaporation at the surface, suction develops to such an extent that the sample shrinks horizontal and more water is driven out. The horizontal strain caused by the suction stress is larger than the outbound horizontal strain due to the loading. The settlement curve deviates from the saturation line. The curve related to horizontal and vertical shrinkage, however, stays on the saturation line. The main soil body is still saturated and consolidates further while also enough suction develops that horizontal shrinkage occurs. This is contradictory if the sample was seen as a single element. Apparently in a sample, multiple processes are ongoing.
In chapter 4 mention was made of the impossibility for real soil behaviour to cross the saturation line because this would imply a negative air volume in the soil. Therefore, for the curves for \( e_{\text{settlement}} \) and \( e_{hvk} \), data points below this line are considered to contain a measurement error. This is further discussed in the section on sensitivity.

For \( \varepsilon_{\text{soil matrix}} \) also the possibility of a measurement error is taken into account. However, data points below the saturation line also suggest another type of error, related to the chosen methodology. A certain threshold is chosen and applied for all soil samples. It is an option that this threshold may have been to strict, yielding too small a total volume. Subtracting the water and soil particle volumes, then leaves a negative air volume. This is not further investigated for this thesis.

### 5.3.4. Sensitivity

In the sensitivity analysis only the sensitivity of the shrinkage curve for the separate variables is investigated. The results, represented in figure 4.24 suggest that the qualitative shrinkage behaviour is relatively unaffected for possible inaccuracies in the variables determination. For \( e_{\text{settlement}} \) curves of samples 2 kPa and 22.5 kPa, a clear difference in void ratio remains even in the most unfavourable scenario.

The \( e_{hvk} \) curves have less margin. Gathered from the results, an additional investigation to increase the accuracy might be useful. Then conclusions on the shrinkage behaviour might be drawn with more confidence. The sensitivity of the shrinkage curve for \( \varepsilon_{\text{soil matrix}} \) is limited for the assumed accuracy.

Combining the variables may give additional information on the sensitivity of the results. For now, an extreme range is taken into account. Combining the variables for these value ranges might lead to a too large sensitivity of the results, diminishing their value unjustified. Also the likelihood of all variables being off is considered low.

Another point of discussion for the sensitivity analysis is the assumption that the accuracy is constant during the test. For \( r_{\text{ring}}, h_{\text{settlement}} \) and \( r_{\text{sample}} \), the default accuracy is used. These remain constant over time, justifying the use of a constant range. For \( V_{\text{soil matrix}} \) this is not the case. The use of a constant range in volume for a decreasing volume means that the accuracy decreases.

The actual likelihood of a measurement error sooner decreases than increases as the sample shrinks. Therefore, in retrospect it might be better to use a percentage or error than a constant range.

Finally, the fact that a subjective factor is involved in the visual analysis might work in favor of the accuracy provided that a consistent procedure is followed. If for example \( h_{\text{settlement}} \) is measured conservatively for sample 2 kPa, it is probable that the same conservative view is employed for the analysis of sample 22.5 kPa. In that case, the sample curves will exhibit a similar erroneous range, leaving the observed qualitative behaviour unchanged.

### 5.3.5. Verification of the experimental shrinkage curve

In the figure 4.25 can be seen that the experimental shrinkage curves are similar to those given in literature. Similar stages of shrinkage are identified. The curve for \( e_{hvk-\text{shrinkage}} \) for sample 22.5 kPa deviates slightly during the final shrinkage phase. The \( e_{\text{settlement}} \) curve based on sample height does show the final phase, although a small drop in void ratio is seen at the end of the curve. This small decrease together with a small reduction in registered sample area, enlarges the effect on \( e_{hvk-\text{shrinkage}} \).

It is more likely that a measurement error has occurred than that the 22.5 sample truly behaves differently for this definition of volume. The comparison with the experimental curve by Yao et al. (2014) in figure 77, strengthens the idea that the volume definition of \( e_{hvk-\text{shrinkage}} \) approaches best the volume determination method based on Archimedes’ principle, as is used during the balloon test. The results show different values that can be explained by a number of things. First of all, a different material was used; Yao performed the test with mature fine tailings. This means a different soil particle density and other soil properties due to the material composition. Another explanation is that during the balloon test under atmospheric pressure, more homogeneous shrinkage occurs. A final explanation is that the Archimedes’ principle is slightly more accurate: after all, no generalisations of sample area and height are necessary.

### 5.4. Verification of the numerical results

No perfect linear relationship was found from which the compression constant \( C_{10} \) could be derived. Thus the existence of a logarithmic relationship between overburden pressure and minimal void ratio is not proven instantly. For the results of minimal \( e_{\text{settlement}} \) and minimal \( e_{hvk} \) a linear regression model was made. The
computed coefficients of determination $R^2$ for both linear approximations approach 1, indicating a good fit.

Up for discussion is the legitimacy to make a linear approximation for the oedometer data as a basis for $C_{10}$. Given the information derived from the sensitivity analysis, a small quantitative inaccuracy may exist for the final void ratio’s, but the qualitative behaviour is captured well by the experimental shrinkage curve. A linear approximation might be acceptable if the discrepancy in the curve is caused by some difference in minimal void ratio, but the general behaviour can be characterized as linear. If this characterization cannot be made, the linear approximation is not deemed valid. According to this reasoning, the linear approximation and derivation of $C_{10}$ is allowed for $\varepsilon_{\text{settlement}}$ and $\varepsilon_{\text{h&v}}$.

This can be supported with literature. Verruijt (2012) introduces equation 2.11 for a situation of one dimensional compression, yielding vertical strain. However, the equation is derived from volumetric strain in general. It is therefore acceptable that the relationship of overburden pressure and minimal void ratio based on $C_{10}$ is used for either definition of volume change.

Conclusions on the appropriateness of the use of equation 2.22 are based on a total of 8 data points. This is perhaps too narrow a data set to draw a definite conclusions. Perhaps it would be useful to expand the range of samples subjected to overburden pressure. Then with more accuracy something can be said on whether the observed relationship is in fact linear (and deviations are related to measurement errors) or not.

A final issue is the probability of the derived values for $C_{10}$. In table 2.1 a range for $C_{10}$ was given. For clays values between 4 and 40 were found in laboratory tests. The values found for this thesis are within this range. However, the values are rather low considering the relative stiff test material.

### 5.5. Validation of the results

The validity of the thesis can be examined on grounds of value for the scientific community and the engineering practice.

In chapter 2 it was mentioned that shrinkage is often assumed to be characteristic material relationship. In this thesis, it was shown that before this assumption can be confirmed or challenged, clarity on a definition of soil volume is essential. Which definition is relevant, differs per discipline.

Three definitions are introduced that in increasing detail describe soil volume: $V_{\text{settlement}}$, $V_{\text{h&v-shrinkage}}$ and $V_{\text{soil matrix}}$.

The first two definitions are considered most relevant for the engineering practice. The first is used to describe settlement of a soil on a large scale, e.g. in a 1D approximation of consolidation. The second definition is more exact and takes into account larger cracks. It has limited value to consider soil on a smaller scale, because relevant soil behaviour will be influenced by cracks and larger air pockets.

Results for these definitions indicate an impact of overburden pressure on the soil volume change during drying. The higher the load, the more the volume reduces in time.

An example of an application of these findings in the engineering practice is for the numerical simulation of Atmospheric Fines drying. Slurried soil layers are deposited and dried. If a drying soil layer is subjected to overburden pressure, the crack formation is suppressed proportionally to the load. However, it is probable that some horizontal shrinkage and cracking will occur. When a new slurry layer is placed on top of the dried and cracked layer, the cracks are filled up, reducing by default the volume of the toplayer before shrinkage has started.

The definition of volume after horizontal and vertical shrinkage is most suitable to capture the described behaviour. The extent of the impact an be predicted using a mathematical relationship as proposed by Vardon and Van Tol (2014). This is verified with experimental results in this thesis. The formula states that the impact of overburden pressure on the decrease of minimal void ratio can be computed with specific compression constant $C_{10}$ and the log of incremental stress.

The volume definition $V_{\text{soil matrix}}$ is mainly relevant for the scientific discipline for on this level, the assumed characteristic material relationship for shrinkage is confirmed. If the soil matrix is considered, the application of load influences the process of volume change and the contribution of consolidation versus suction driven shrinkage. The result, captured here as the minimal void ratio, is unaffected by the overburden pressure.
5.6. Concluding remarks
In this chapter, the employed methods and results are discussed. Several observations deserve extra mentioning.

The question of soil volume definition is leading in this thesis. Several options can be given, all relevant for different disciplines and all true. Three leading definitions are taken into account, referring to several scale steps of soil description. For each of these definitions, the main research question can be answered differently.

The interpretation of the results is based on a generalisation of the soil samples and their behaviour. Yet it becomes clear that the samples are not homogeneous elements in which only a single process can be identified. Observed discrete shrinkage in the samples substantiates this perception. In the interpretation of the shrinkage curves, this notion must be taken into account.

Based on the discussion, conclusions can be drawn and an answer can be formulated for the main research question. Also, recommendations for future research can be made.
Figure 5.10: The results for the shrinkage curve and diagrams for characteristic behaviour.
Conclusions and Recommendations

6.1. Introduction
In this chapter, the conclusions and recommendations for further research are presented. First, the main research conclusions are formulated and substantiated. Some limitations related to the conclusions are addressed as well. Subsequently, additional conclusions based on methodology and results are discussed. In the second part of the chapter, recommendations are made for future research.

6.2. Main conclusions
In this section, an answer is formulated to the main research question. The main conclusions are substantiated and some limitations are addressed.

6.2.1. Main research conclusions
Main research question
"Does applied overburden pressure impact unsaturated shrinkage behaviour of a fine-grained soil with a high initial water content? If so, what is the impact and how can the impact be quantified?"

Answering the main research question
Whether overburden pressure impacts unsaturated shrinkage behaviour of fine-grained soil, depends on the employed definition of total soil(sample) volume.
For the volume definition \( V_{\text{soil matrix}} \), for which only the soil matrix is taken into account, no impact is detected. This observation indicates that shrinkage behaviour is characteristic for the material on soil matrix level.
For the volume definitions based on vertical volume change, \( V_{\text{settlement}} \), and on the combination of vertical and horizontal volume change, \( V_{h&v-shrinkage} \), impact of overburden pressure on the shrinkage behaviour is observed.
For both definitions, the minimal void ratio obtained after drying of the material, decreases proportionally with the application of overburden pressure.
The impact can be quantified with a mathematical formulation based on a compression constant \( C_{10} \) that links change in minimal void ratio to incremental overburden pressure.

Statement on hypothesis
Whether the hypothesis is confirmed or challenged depends on the employed definition of total sample volume.
For the volume definition \( V_{\text{soil matrix}} \), the hypothesis is challenged.
For the volume definitions based on vertical volume change, \( V_{\text{settlement}} \), and on the combination of vertical and horizontal volume change, \( V_{h&v-shrinkage} \), the hypothesis is confirmed.

6.2.2. Substantiation of the main conclusions
In this thesis the impact of overburden pressure on unsaturated shrinkage behaviour of fine-grained soil is investigated. Both observed behaviour and impact are different for each identified definition of soil volume
in this thesis.
The process where total sample volume decreases and the soil particle volume remains constant, is referred to as shrinkage (due to evaporation) or consolidation (due to vertical compression). If during this process the water volume decreases and the volume of air in the sample increases, unsaturated shrinkage is occurring. Said unsaturated shrinkage can be quantified by measuring the sample void volume, which again depends on the total sample volume.

Three definitions are formulated for total sample volume, which describe the state of a soil sample on several levels of accuracy. The first definition, \( V_{\text{soil matrix}} \), takes into account only the soil and water particles and small air volumes that are part of the soil matrix. Cracks and air pockets are excluded. The second definition gives a soil volume that is only influenced by vertical volume reduction, \( V_{\text{settlement}} \). A third definition of soil volume, \( V_{h\&v-shrinkage} \), takes into account any vertical volume reduction as well as horizontal shrinkage, excluding larger cracks from the soil total. Shrinkage of a drying soil can be captured in a shrinkage curve, giving the relationship between pore water volume and pore volume. For each of the total sample volume definitions, a different pore volume is found, yielding three separate shrinkage curve types.

The impact of overburden pressure was assessed as observed change due to incremental pressure in the shrinkage curve types, derived from experimental results. For the first curve type based on \( V_{\text{soil matrix}} \), no impact was detected. The experimental data suggest that when a soil sample is considered on soil matrix level, the shrinkage curve represents a material relationship. The shrinkage limit is not affected by the application of overburden pressure, nor is the minimal void ratio. The application of pressure does impact the process of volume change. The applied load corresponds to the contribution of consolidation versus suction driven shrinkage.

Conclusions related to the definition of total soil volume are valuable for scientific purposes. The assumed characteristic material relationship given by the shrinkage curve is confirmed for the soil matrix.

For the curves based on \( V_{\text{settlement}} \) and \( V_{h\&v-shrinkage} \) respectively, the range of applied loads yields a range of shrinkage curves. For both types of curve, the minimal void ratio decreases with an increase of applied overburden pressure, although to different extents. It can be concluded that the application of load suppresses the formation of cracks in favor of vertical settlement.

The measured volume change for \( V_{\text{settlement}} \) is caused by consolidation and evaporation driven vertical shrinkage. The contribution of consolidation is enlarged by the application of a larger load. This translates directly to the impact measured in the shrinkage curve. For \( V_{h\&v-shrinkage} \), the impact of overburden pressure on the shrinkage curve is lessened because also evaporation driven horizontal shrinkage is taken into account. The horizontal shrinkage slightly counteracts the contribution of consolidation on total sample volume over time.

This conclusion is substantiated by the dewatering behaviour of the samples. If more water is driven out due to consolidation, less water is left to evaporate before the residual water content is reached. The contribution of shrinkage to volume change is decreased. The opposite is true if the contribution of consolidation is smaller due to a small load. Then more unsaturated shrinkage is observed.

The definitions for \( V_{\text{settlement}} \) and \( V_{h\&v-shrinkage} \) with respect to volume change are useful for the engineering practice where macroscopic soil behaviour is relevant. In the case of a 1D consolidation approximation, the vertical volume change is leading. For a more accurate model of the deposition of a slurried soil layer on a dry layer (including crack formation), the horizontal volume change and resulting back-fill of cracks becomes relevant as well.

The impact of overburden pressure on the shrinkage curve for \( V_{\text{settlement}} \) and \( V_{h\&v-shrinkage} \) can be quantified using a mathematical relationship. The impact is formulated as a decrease in minimal void ratio caused by increased applied pressure and a compression constant \( C_{10} \), that is unique for each type of shrinkage curve. The new found minimal void ratio can be used as a parameter in the shrinkage equation by Fredlund et al. (2002a) to find the approximated behaviour. The equation is part of the sludge ripening model (Vardon and Van Tol (2014)), that is made to predict the behaviour of fine-grained soil during layered deposition.

**6.2.3. Value of the thesis**
The value of the thesis is found in the application of the main conclusions for the scientific community and engineering practice.
The conclusion that soil shrinkage considered at soil matrix level gives a characteristic material relationship is a valuable reference point for future research.

For the engineering practice, prediction of slurried fine-grained soil behaviour during deposition is valuable for many situations (e.g. land reclamation and storage of mining tailings). Volume definitions of $V_{\text{settlement}}$ and $V_{\text{shrinkage}}$ are relevant here, for on larger scale soils contain cracks and air pockets which influence behaviour. For these soil volume definitions impact of overburden pressure on shrinkage behaviour was observed and must be accounted for in numerical models.

An example of an application of the thesis results for the engineering practice is found for the sludge ripening model by Vardon and Van Tol (2014). The proposed relationship for overburden pressure and minimal void ratio is confirmed both as a concept and quantitatively based on the experimental results. To model the process of drying, rewetting, back-fill of cracks and again drying, inherent to layered deposition of slurry, the volume definition for horizontal and vertical shrinkage is most relevant.

6.3. Limitations to the main conclusions

Several limitations related to the main conclusions are addressed. They are to be kept in mind during further processing of the research results and conclusions. Also, the limitations are used for the formulation of recommendations in this thesis.

- The definitions of total sample volume are inherent to the conclusions of the thesis. The three definitions can be seen as increasingly exact descriptions of the soil sample that are relevant for different disciplines ranging from engineering practice to academia. The use of a framework for volume implies an approximation of the sample dimensions. Indeed it was concluded, based on the test results, that actual shrinkage is not a homogeneous process. Approximations lead to an increased range for parameter accuracy and increased sensitivity of the results. One must account for this sensitivity in the interpretation of results based on the framework.

- Conclusions related to the impact of overburden pressure on shrinkage are drawn based on experimental test results. The observed behaviour due to compression and desaturation is generalized for the entire samples. Due to the discrete shrinkage process of soil, this is not necessarily true for the actual situation. The applied stress is not homogeneously transferred, possibly influencing the results and derived conclusions. Nor can one state of saturation be identified. The soil matrix may remain saturated while at the surface water evaporates and cracks develop.

- In this research, the effect of two simultaneous processes is investigated, that of consolidation and evaporation induced shrinkage. The two are coupled and their effect is not easily separated during analysis of the results. In the experimental phase of the thesis, the processes were not investigated separately. Therefore, no quantitative conclusions can be drawn on the contribution of either process for this particular material under these conditions.

- The most exact definition for soil volume is based on the soil matrix. What exactly constitutes soil matrix is a subjective matter. In general it can be said that all cracks and large air pockets are excluded from the soil matrix volume and continuous fabric is considered. The issue of inclusion or exclusion of smaller air volume in the fabric becomes one of desired scale and accuracy of research equipment. This is an important note for further use of the definition.

- For this thesis no impact of overburden pressure on the unsaturated shrinkage behaviour of soil was observed for the volume definition related to the soil matrix. A main limitation here is the applied pressure. In theory, if the applied pressure is large enough, grain crushing may occur and the minimal void ratio may be reduced in reference to what was found in this thesis.

- The experimental data was used to verify the use of a compression constant to predict a reduction in minimal void ratio due to incremental pressure in the sludge ripening model. Subsequently, the conclusion is drawn that the impact of overburden pressure on unsaturated shrinkage behaviour can be quantified using a mathematical expression based on the same constant. This conclusion is based on a small data set of three test samples and a reference test. This is merely a limitation to the conclusion.
6.4. Additional conclusions

Based on the experimental process and derived results, several additional conclusions can be drawn. In this section, they are addressed.

6.4.1. Conclusions related to the method

Experimental tests

- Two tests with different original purposes, the oedometer test and hyprop test, were modified to meet the thesis requirements. The tests were stopped frequently to conduct additional measurements to gather the necessary data to assess shrinkage behaviour, disturbing the original test procedure. The frequency of weigh/scan breaks determines the amount of data points in the shrinkage curves. Yet, the more breaks, to larger the (risk of) disturbance in the test becomes. Due to this inconsistency, it is concluded that both tests are not entirely appropriate for the thesis objective.
  - The dry oedometer test procedure is effective for subjecting a sample to a vertical load. Due to the sample cell and oedometer frame design, the contact surface between load cap and sample is fairly good. The connected transducer measures vertical settlement without disturbance well.
  - The main disadvantage of the use of the hyprop is the characteristic presence of the tensiometers in the soil. Over time the tensiometers take on some of the subjected pressure as the soil dries and shrinks. At this point the sample data are no longer suitable to answer the main research.

6.4.2. Conclusions related to the results

Consolidation and evaporation driven shrinkage

- Unsaturated volume change of a soil subjected to overburden pressure is driven by the coupled processes of consolidation and evaporation driven shrinkage. The larger the applied pressure, the greater the contribution of consolidation to the volume change. The contribution of evaporation driven shrinkage then decreases relative to a sample with a lesser load. And vice versa.

- Evaporation is allowed from the beginning of the test, causing the sample to slowly desaturate. The desaturation influences the soil properties such as hydraulic conductivity. The rate of consolidation then decreases over time. At the same time suction develops and the sample shrinks in vertical and horizontal direction.

- The process of volume change continues until the residual water content of the soil is reached. Although the processes of consolidation and evaporation occur mostly simultaneous, different regimes in the dewatering process can be seen. Initially consolidation drives water outflow. Then a period of coupled processes can be seen until a linear evaporation rate is established. As the residual water content is reached, the dewatering rate slowly approaches zero. The length in time and water loss of each regime per sample depends on the applied overburden pressure.

Configuration of shrinkage

- The test samples show discrete shrinkage behaviour. Small air pockets and fissures that originate during the sample preparation develop into larger air pockets and cracks. The location of the original elements is an important factor for the final configuration of the sample. Irregularities close to the edge of the sample lead to the severance of smaller volumes as the main sample contracts along a crack surface. A homogeneous original sample gives homogeneous shrinkage behaviour as it contracts more or less isotropically from the surface inwards.
6.5. Recommendations

In this thesis the impact of overburden pressure on unsaturated shrinkage behaviour of fine-grained soil is investigated experimentally. It was found that the existence of such impact depends on the employed definition of soil sample volume. In this section, several recommendations are made in order to improve procedures followed and deepen knowledge on the subject.

6.5.1. Recommendations related to the method

Optimize test preparation and conditions

- In order to accurately compare test results of the samples, care must be taken to prepare homogeneous samples. Methods to achieve a highly homogeneous sample are:
  - Increase initial water content of sample material.
  - Apply material with small quantities and proper equipment.
  - Place samples in vacuum pump to remove air bubbles.
  - Perform check by back-calculating air volume in sample from weight and soil particle density.

- The evaporation rate is controlled by climate conditions and the available evaporation surface. Either may be altered to achieve the desired evaporation rate before the start of the test.

- The risk of measurement errors must be reduced by limiting the displacement of test elements during and after a weighing/scanning interval. The position of the transducer and sensor must be kept constant.

Investigate alternative methods

- It was concluded that the oedometer test and hyprop test are sub-optimal methods to meet the test objective. Therefore it is recommended to further investigate the use of other tests or perhaps - if need be - to develop a new test that may be used to continuously measure sample volume and mass changes.

- The scan results are analysed using several visual analysis methods. It is worthwhile to investigate the use of different analytical tools that are less subjective, time consuming and prone to measurement errors.

6.5.2. Recommendations related to results

Perform a saturated oedometer test

- In this thesis, no conclusions can be drawn on the exact contributions of consolidation and evaporation in the process of volume change of the soil samples. It is recommended to perform a saturated oedometer test on similar material at least for the extremes of the test range. From these results, more can be said on the additional effects of unsaturated shrinkage.

Extend the test load range

- It is recommended to perform a test sequence with a broader range of applied loads. With this test sequence the conclusions in this thesis are verified for higher loads. In particular, one can investigate the load range for which no impact of overburden pressure is observed for the shrinkage curve based on $V_{soil\, matrix}$.
  Also, with an extended data set of curves for different loads, the validity of the use of the mathematical relationship based on $C_{10}$ can be confirmed with more certainty.

Investigate suction development for a higher load

- The development of suction for a larger range of loads can be investigated. More can be said on the dissipation of pore pressures and possible delaying effects on the development of suction and subsequent shrinkage. A hyprop may be used but one needs to be aware of the possible influence of the tensiometer on the baring capacity and consolidation of the sample.
Use $V_{h\&v\text{-}shrinkage}$ for slurry deposition modelling

- An advice is given on which volume definition is most relevant for modeling layered deposition of slurred soil as is intended in the Sludge Ripening model. Due to the back-fill of cracks as a new layer is placed, the volume of the new layer decreases by default before any water has dissipated. Even if only vertical settlement is modelled, this default decrease in layer height due to actual horizontal shrinkage (crack formation) must be taken into account. This is best captured in the volume definition for horizontal and vertical shrinkage.


BGC Engineering inc. Oil sands tailings technology review, 2010.


Soil particle mineralogy

A.1. Soil mineralogy

In this section, the phase of the soil particles is viewed more closely. Soil particles are made out of minerals. For most soils, these minerals form crystals with an orderly internal atomic structure. The three dimensional crystals are bound by surfaces on which forces are at work. For small particle sizes (< 2 µm) the surface forces (e.g. attraction and adsorption of molecules, cohesion with the surface of another mass) gain in influence on the soil behaviour in comparison to larger soil particles. In a fine grained soil with a platy particle shape, such as clay, the surface area can be very large compared to the mass, increasing the susceptibility to the surface forces.

A.1.1. Gravel, sand and nonplastic silt particles

Characteristics of non cohesive soils are mainly defined by particle size, shape, surface texture and particle size distribution. Hardness and resistance to weathering are dependent on the mineral composition. The most abundant mineral is quartz. The gravel, sand and silt fraction of a soil usually consists of bulky particles in the size range of 75mm to 2 µm.

A.1.2. clay minerals

Clay minerals are mostly composed of two or three sheets of molecular structural units. Different clay mineral groups are characterized by the stacking arrangements of these sheets and of how the layers are held together. Some types of clay mineral absorb water and/ or cations between the unit layers, affecting the properties of the material (e.g. volume increase). The sheets of the clay minerals extend in two directions, yielding very small (< 2 µm) platy particles.

A.2. Fabric

The term fabric refers to the arrangement of particles, particle groups and pores. The term structure can be used to refer the soil fabric in relation to composition and interparticle forces. Sand and gravel particles are large and bulky enough to behave as independent units and together behave as a particle arrangement. Clay particles are much smaller and are often encountered in particle groups. An association of (face-to-face) particles is called an aggregate. Edge-to-edge or Edge-to-face particles or aggregates are referred to as being flocculated. An organisation of particle units with a physical boundary is an assemblage. Soil fabric can be discussed using three levels of scale. Microfabric consists of aggregates and the small pores in between them. Minifabric entails the microfabric aggregations and interassemblage pores. Macrofabric consists of the previous and also includes cracks, fissures and root holes.
(a) Molecular formations (tetrahedrons and octahedrals) as sheet structures.

(b) The clay minerals are composed of sheets. The basic units form a 1:1 or 2:1 mineral. Water molecules or other cations can also be part of the structure, changing the properties of the mineral.

Figure A.1: Clay minerology

(a) A schematic representation of particle association in clay suspension: 1) dispersed particles 2) aggregated particles 3) flocculated particles

(b) A schematic representation of particles assemblages and interassemblage pores in the minifabric

Figure A.2: Clay fabric and fabric elements
Choice of experimental method

Criteria for the choice of test are formulated as such:

- (Proved) effectiveness of the test
- Laboriousness versus result
- Flexibility in loading scenario’s
- Accuracy of results
- Reproducibility of test results
- Impact of specimen size and cracking behaviour

These criteria are used to select the most suitable test method for the research objective. The assessment of the criteria per test is represented in figure B.1.

<table>
<thead>
<tr>
<th>Criteria per laboratory test</th>
<th>Oedometer test</th>
<th>Adjusted Balloon test</th>
<th>Adjusted Wax test</th>
<th>Hyprop test</th>
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</thead>
<tbody>
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<td>1</td>
<td>2</td>
<td>2</td>
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<tr>
<td>Laboriousness versus result</td>
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<tr>
<td>Reproducibility of test results</td>
<td>3</td>
<td>2</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>Impact of specimen size and cracking behaviour</td>
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<td>2</td>
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<tr>
<td><strong>Total</strong></td>
<td><strong>15</strong></td>
<td><strong>10</strong></td>
<td><strong>12</strong></td>
<td><strong>13</strong></td>
</tr>
</tbody>
</table>

Figure B.1: Each test option is evaluated on the set criteria.

Based on above criteria, the balloon test is considered unsuitable. The advantage of the adaptability of the confining pressure does not weigh up to the uncertainty of result quality and complexity of the test. The wax test is fairly simple but laborious. The results are not easily reproducible due to the natural formation of clods. The oedometer tests are standard and therefore provide relatively reproducible results. The equipment is designed for loading of a sample which simplifies the procedure. The continuous measurement of vertical settlement increases the accuracy of the test. The hyprop test is similar to the oedometer test, with the advantage of continuous measurements of the change in soil mass. Also the test measures the developing suction in the sample, which is though to be the main driver for (atmospheric) shrinkage. This may be valuable information during the result interpretation. A downside is that the loading on the default scale is limited.

From the criteria analysis can be concluded that the oedometer tests and the hyprop test are the most suitable for the research objective.
Soil particle distribution

In the figure, the results from the sieving test and hydrometer test are combined in a graph to give the soil particle distribution of the Vingerling clay.

Figure C.1: Grain size distribution Vingerling clay
D.1. Oedometer test set up
In figure D.1 the test set up and all relevant denotations are represented.

D.2. Oedometer test log
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Table D.2: Oedometer test log
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</tr>
<tr>
<td>17-09-15</td>
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<td>4</td>
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<td>3</td>
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<td>15</td>
<td>1</td>
<td>Weighing, CT scan</td>
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<td>Weighing, CT scan</td>
<td></td>
</tr>
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<td>08:00:00</td>
<td>2</td>
<td>4</td>
<td>Weighing, CT scan</td>
<td></td>
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<td>3</td>
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<td>Weighing, CT scan</td>
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<td>Weighing, CT scan</td>
<td></td>
</tr>
<tr>
<td>28-09-15</td>
<td>10:45:00</td>
<td>2</td>
<td>4</td>
<td>Weighing, CT scan</td>
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<td>Weighing, CT scan</td>
<td></td>
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<td>2</td>
<td>Weighing, CT scan</td>
<td></td>
</tr>
<tr>
<td>05-10-15</td>
<td>08:00:00</td>
<td>2</td>
<td>4</td>
<td>Weighing, CT scan</td>
<td>Displaced sensor</td>
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<td>7.5</td>
<td>3</td>
<td>Weighing, CT scan</td>
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<td>1</td>
<td>Weighing, CT scan</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>22.5</td>
<td>2</td>
<td>Weighing, CT scan</td>
<td></td>
</tr>
<tr>
<td>13-10-15</td>
<td>10:00:00</td>
<td>2</td>
<td>4</td>
<td>Weighing, CT scan, Microscan</td>
<td>Transducer battery down</td>
</tr>
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<td>7.5</td>
<td>3</td>
<td>Weighing, CT scan</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>15</td>
<td>1</td>
<td>Weighing, CT scan</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>22.5</td>
<td>2</td>
<td>Weighing, CT scan, Microscan</td>
<td></td>
</tr>
<tr>
<td>14:00:00</td>
<td></td>
<td>2</td>
<td>4</td>
<td>End of test</td>
<td></td>
</tr>
<tr>
<td></td>
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<td>7.5</td>
<td>3</td>
<td>End of test</td>
<td></td>
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<td></td>
<td>15</td>
<td>1</td>
<td>End of test</td>
<td></td>
</tr>
<tr>
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<td></td>
<td>22.5</td>
<td>2</td>
<td>End of test</td>
<td></td>
</tr>
</tbody>
</table>

Table D.3: Oedometer test log
E.1. Hyprop test set up

In figure E.1 the set up for the hyprop test is visible. The mass is placed on small screws to limit evaporation hindrance.
E.2. Hyprop test log

<table>
<thead>
<tr>
<th>date</th>
<th>time</th>
<th>kPa</th>
<th>activity</th>
<th>additional comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>25-08-15</td>
<td>16:18:00</td>
<td>0</td>
<td>Start of test</td>
<td>Necessary adjustment shape of porous plate</td>
</tr>
<tr>
<td>26-08-15</td>
<td>10:46:00</td>
<td>2</td>
<td>Weight placement +942.83 g</td>
<td></td>
</tr>
<tr>
<td>27-08-15</td>
<td>11:40:00</td>
<td>2</td>
<td>CT scan</td>
<td>No complete scan</td>
</tr>
<tr>
<td>07-09-15</td>
<td>10:55:00</td>
<td>2</td>
<td>CT scan</td>
<td>No complete scan</td>
</tr>
<tr>
<td>09-09-15</td>
<td>09:55:00</td>
<td>2</td>
<td>CT scan</td>
<td>No complete scan</td>
</tr>
<tr>
<td>14-09-15</td>
<td>10:55:00</td>
<td>2</td>
<td>CT scan</td>
<td>No complete scan</td>
</tr>
<tr>
<td>17-09-15</td>
<td>11:30:00</td>
<td>2</td>
<td>CT scan</td>
<td>Large crack formation causes instability of mass</td>
</tr>
<tr>
<td>21-09-15</td>
<td>08:00:00</td>
<td>2</td>
<td>CT scan</td>
<td>Partial settlement in sample</td>
</tr>
<tr>
<td>28-09-15</td>
<td>10:40:00</td>
<td>2</td>
<td>CT scan</td>
<td>End of test</td>
</tr>
</tbody>
</table>

Table E.1: Hyprop test log
Results

F.1. Experimental results

F.1.1. Suction development

Figure F.1 gives the suction development in time as measured by the hyprop. In the figure, various measurement stages are given which can be used to back-calculate the suction after the regular measurement range is exceeded.

![Suction development graph]

Figure F.1: Overall development of suction in time

F.2. Processes results

In figure F.2 for all samples, results for area computed by avizo are given. Due to inconsistency of the data and the relative little added value compared to the computed area based on the sample radii, the results are not taken into account in the computation of the shrinkage curves.
Figure E.2: Comparison of sample areas I, II and III. In the analysis for the shrinkage curve, sample area II is used.

F.3. Shrinkage curve
In figure E.3 the shrinkage curve for all samples are given. The figure confirms the conclusions that were drawn based on the curve for samples 2 kPa and 22.5 kPa.
Figure E3: Shrinkage curve for all oedometer samples