Report Bachelor Assignment
Influence of different pore fluids on micro mechanics and shear strength of non-active clay

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1 Preface

The reason to do research into geomechanics for my Bachelor Assignment was to find out whether this specialization of Civil Engineering would interest me enough to do my master in it. During the preparation phase of the Bachelor Assignment I already got really interested into geomechanics and thus I signed up for the Geo Engineering master at the TU Delft. While doing the Bachelor Assignment this interest kept growing and confirmed that I made the right choice by signing up for the Geo Engineering master.

However, during the assignment and also while writing this report there were some difficulties for me understanding all the concepts, since the concepts were very detailed and I had not worked with them before. I guess because of my interest for the specialization it was easier to learn and understand the new concepts.

I would like to thank Arianna Pagano for her daily supervision. She never seemed to get tired of all the questions that I had and took the time to explain everything to me. I am very thankful for that. And I would also like to thank Alessandro Tarantino for all his help and explanations about the difficult topics. The concept of the Mohr circle is clear to me now. I guess I do not understand all concepts you explained yet, but still I learned some major things about geomechanics during this assignment.

Then I also want to thank Vanessa Magnanimo and Gerrit Snellink for their supervision from the University of Twente. You gave some valuable input for the proposal report and the final report.

Contents

2 Summary

The objective of this Bachelor Assignment was to find out the influence of the different pore fluids on the micro mechanics and shear strength of non-active clay. This objective was divided into different research questions. One question focused on the influence of the pH-level of the pore fluids, another on the influence of the dielectric permittivity. Also the influence of the different OCR states was investigated. The last research question was to find out what micromechanisms would be responsible for the observed behaviour on macro level.

The first result that was observed is that for pH 4 water the ultimate strength is smaller than the critical strength, even though they should have been the same. For the pH 9 water both ultimate strength and critical strength had the same value. This difference in behaviour can either be explained by looking at the different ways of interpreting the Mohr Circle, but it can also be explained by looking into the difference in microstructure. Further research into the microstructure of the clay using the MIP method could help understanding these mechanisms, but this was out of the scope of this bachelor assignment.

The difference in ultimate and critical strength can also be explained by looking at the Taylor's equation. This equation holds for sand, but it is not sure whether it also holds for clay. In the report it is proposed that this equation should have an additional term that takes into account the elastic energy. For sand this term would become negligible, but for clay not. Taking into account the elastic energy would explain the difference in ultimate and critical strength, but also the fact that the peak strength does not occur at the maximum dilatancy and that pH 9 does not have a peak.

Another result is the difference in friction angle of the failure envelopes for the pore fluids with different values of dielectric permittivity. The research of Pedrotti and Tarantino (2017) already showed this difference upon 1D compression, but now it is shown that this difference also occurs upon shearing. Why this difference in shear strength can be observed between the pore fluids with different dielectric permittivity again can be explained by looking at the microstructure. The higher the repulsion between the particles, the bigger the angle between the particles in edge-to-face configuration. What happens at micro level that causes particles with a bigger angle to have a higher shear strength still has to be found out by doing further research on microscopic level.

A conclusion that was drawn upon these results is that for clayey soils elastic energy has to be taken into account while looking at the Taylor's equation. (Taylor, 1948). This would mean an additional term needs to be added to the equation. A detailed explanation can be found further in this report.

3 Introduction

This report will explain the research that was performed by Dianne Jacobs at the University of Strathclyde. The research looked into the behaviour of non-active clay on micro level under influence of four different pore fluids.

The reason to start the research was the fact that not much is known about the influence of the microstructure of the clay on its macro level strength. The two main problems with clay are as follows. Firstly, the size of the clay is much smaller than the size of granular materials like sand. Because of this small size not only mechanical forces act on the particles, but also electrochemical forces. This makes it more complex to do research into the soil behaviour, because one cannot be sure whether a change in strength is caused by the mechanical or electrochemical force. The small size of the clay particles also makes it hard to explain what happens at macro level by looking at the behaviour on micro level. There is no experimental equipment yet to test the micro level behaviour of clay. The second problem is that the shape of a clay particle is not spherical, but flake-like. This means that the particle is hard to model and thus rarely any simulation study has been done into the behaviour of clay.

After some thorough literature research into what was already known about the clay and what knowledge was still missing, one established the following hypothesis: There will be differences in the mechanical behaviour of clay with different pore fluids upon direct shearing. It is expected that the more repulsion occurs in the soil, the weaker its strength will be. This hypothesis is tested during the research and in this report it will be explained whether this hypothesis was true or false and why. The research aim for the research was: determine the influence of different kind of fluids with different pH and dielectric permittivity under different OCR conditions on the shear strength of non-active clay. Before starting the experiments it was expected that the pH level and dielectric permittivity influence the microstructure of the clay and that they influences the behaviour on macrolevel, not the differences in OCR states. A few boundaries were established for this research aim. Firstly, the research focused solely on non-active clay and not on active clay. In this way possible problems that could occur after the swelling could be neglected. Secondly, one only looked at the mechanical properties of the clay and in specific its shear strength. The third boundary was that the research was only done upon the clay itself. So this fundamental research only looked at the properties of clay itself and not how it interacts with other structures. And the last boundary was that the research was fully experimental.

In the next chapters the literature review, preliminary tests, method, results, discussion, conclusions and recommendations for the research will be explained. The appendix contains a detailed description of the experiments steps and contains all the output graphs. A brief evaluation of the planning will also be included in the appendix.

4 Literature Review

This chapter will show the literature review that was already done for the proposal report, but will expand the literature review by looking into the soil behaviour with more detail. The specific mechanisms that occur at the different pH levels will be explained.

4.1 General introduction into kaolin and research performed

First clay in general will be explained. Clay consists of flake-shaped microscopic and submicroscopic particles made of mica, clay minerals and other minerals. Clay particles are classified the smallest particles of all soils. According to several institutions in the United States including the Massachusetts Institute of Technology clay has a particle size of less than 0.002 mm.(Das and Sobhan, 2013) It has the characteristic to develop plasticity when it is mixed with water. Clay minerals consist of two elements: (1) quartz sheets and (2) metal oxides sheets. These sheets are stacked on each other and connected by different kind of bondings. Different combinations of sheets and bondings form different clay minerals. The combination of isomorphous substitution and a break in the continuity of the structures at the edges of the clay particles causes that the particles have a negative charge at the surface. Besides the negative charge of the particle surface, the particle edges can have a positive or negative charge. This is due to protonation or deprotonation of broken edges and is strongly dependent on the pH-level. Isomorphous substitution entails that one element is changed for another without changing the crystalline form of the molecule.(Das and Sobhan, 2013) The negative charge of dry clay is balanced by the presence of cations like Na+ and Ca2+. In wet clays anions flow around the clay particle and form a diffuse double layer. The water in this double layer is kept near the particle by the force of attraction. This orientation of water around clay gives clay plastic properties. When a soil as a whole consists for more than half of clay minerals, this has a big influence on the engineering properties of the whole soil. The clay then has the biggest share in the decision of the engineering properties.

Next the structure of clay will be explained. Clay can be categorized into the cohesive soils. The forces that act between clay particles are repulsive and attractive. The repulsive force is caused by the diffuse double layer that each particle has. Van der Waals forces that are independent of the characteristics of the water that the clay is in are responsible for the attractive forces. The repulsive and attractive forces are also due to the electrostatic interaction between charged particles and faces. When the space between two particles is small, the attraction forces have a greater influence than the repulsive forces. Also the positive edges can be attracted by the negative parts of the clay particle. The gravitational forces between the clay particles are negligible, since the particles are very small.(Das and Sobhan, 2013) The interaction between these individual particles can form a bigger structure. The two structures that clays occur in are the dispersed structure and flocculent structure. An overview can be found in Figure 1. When clay is put into water it first will show a Brownian motion. This is a random zigzag motion of the particles in suspension. The sediment that is the result of this motion has a dispersed structure, in which the particles are located parallel to each other.(1a) Next these individual clay particles can change into flocs that have edge-to-face contacts: also called flocculation. An edge-to-face contact is an electrostatic attraction of the positively charged edge and negatively charged face of a clay particle. When the flocs have a significant size, under the

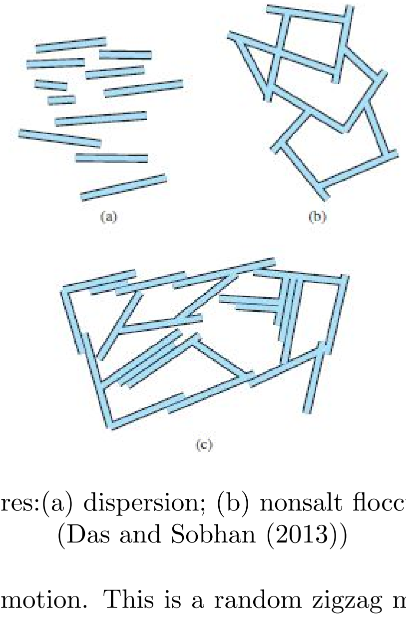


Figure 1: Sediment structures:(a) dispersion; (b) nonsalt flocculation; (c) salt flocculation (Das and Sobhan (2013))

force of gravity they will form a flocculent structure. Clays with a flocculent structure are lightweighted and have a high void ratio e. And when one adds salt to the clay, the ions of the salt depress the diffuse double layer of the clay particle. This will result in the reduction of the repulsive forces. A higher pH-level will be gained. Clays that occur in sea are highly flocculent (1c) and clays in freshwater have an intermediate structure between disperse and flocculence.(1b)

The following section will discuss the three most important types of clay minerals shortly. The first is kaolinite that consists of silica and gibbsite sheets and each layer has a thickness of 7.2 Å. These layers are held together by hydrogen bondings. The hydrogen atoms in the gibbsite sheet are shared with the oxygen atoms in the silica sheet. The form of kaolinite is flake-shaped, with a thickness between 100 to 1000 Å. The surface area is 15 m2/g.(Das and Sobhan, 2013) The other two important clay minerals are illite and montmorillonite. Illite is also called clay mica and consists of sillica and gibbsite sheets that are bonded by potassium ions. A negative charge is needed to balance the potassium ions. This negative charge comes from the substitution of aluminium for silicon in the silica sheets. This is called isomorphous substitution and was explained in the previous paragraph. The other clay mineral is montmorillonite, which has a simular structure as illite. Yet in montmorillonite there is a isomorphous substitution of iron and magnesium replacing the aluminium in the gibbsite sheets. Because potassium ions are not present, a large amount of water is attracted between the layers. And overview of the layers in the three different clay minerals can be found in Figure 2. Other common clay minerals are chlorite, halloysite, vermiculite and attapulgite. In the Bachelor Assignment the focus will be on the kaolinite mineral. In the kaolinite mineral the quartz sheet consists of silica tetrahedron and the metal oxides sheets are made of alumina octahedron. The silica tetrahedran forms a silica sheet and the alumina octahedron forms a octahedral sheet. There is an option in which the aluminium atoms are replaced by magnesium: in this case the octahedral sheet will become a brucite sheet: again an isomorphous substitution. The possible bondings between the clay particles are also already mentioned. The research by Wang and Siu (2006) found that a pH level below the isoelectrical point of edge surfaces of kaolin clay causes the Coulombian attractions between the face and edges to dominate, thus resulting in a mainly edge-to-face configuration. In pH levels above this point the double layer repulsion is dominating, thus a deflocculated and dispersed fabric emerges. This double layer repulsion can dominate, because all positive charges on the particle edges are de-activated. So the face-to-face configuration is mainly present at these pH levels. They mentioned that commerical kaolinite has an IEP of 5.8. This is almost the same value as the pH value above which the positive charges are de-activated: 5.0. In the next paragraph the principle of activity will be explained.

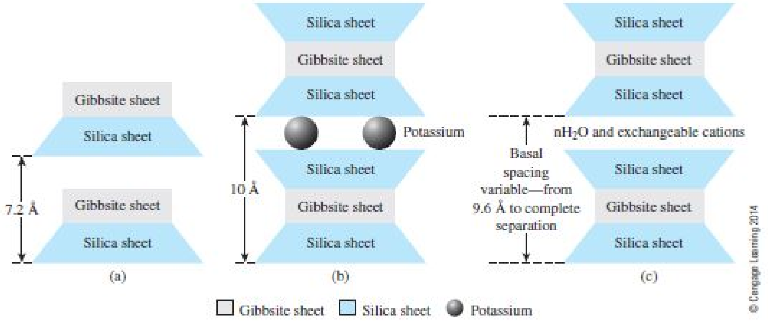


Figure 2: Overview of different layers of clay minerals (Das and Sobhan (2013))

A characteristic of clay is its activity. This is used to identify the swelling potential of a clay.(Das and Sobhan, 2013) Table 4.2 of the Das and Sobhan (2013) book showed that kaolinite has the lowest activity of all clay minerals, with an activity in the range of 0.3 till 0.5. The unit of activity is the index for identifying the swelling potential of clay soils. This is the slope of the line that correlates with a PI and percentage finer than 2 m. The PI is the plasticity index that can be calculated by subtracting the plastic limit from the liquid limit of the soil. There are different tests to find these values of a soil. For kaolinite the liquid limit ranges between 35 and 100 and the plastic limit between 20 and 40. This means that the PI of kaolinite can range between 15 and 80. Burmister (1949) made a classification for the plasticity index. From this it can be concluded that kaolinite has a medium plasticity for the values 15-20; a high plasticity from 20-40 and a very high plasticity for values higher than 40. For the current research by the university of Strathclyde non-active clays were chosen over active clays, because adding a pore fluid to a non-active clay does not cause any change in volume of the particles themselves. The pore fluid will only affect the macroscopic behaviour by affecting the particle-to-particle interaction. Thus it is easier to model non-active clay particles.

On macro level non-active clays have several mechanical properties. Two of these are its compressibility and its shear strength. The research of Pedrotti and Tarantino (2017) investigated the compressibility of the non-active clay under influence of different pore fluids. They first did tests using the oedometer to find out the compressibility on macro level. Using a microscope they tried to explain this difference in outcome per pore fluid on macro level by looking at the arrangements of clay particles on micro level. They used the MIP (Mercury Intrusion Porosimetry) method for this. There are two different consolidation states a soil can be in. A normally consolidated (NC) soil is a soil that has never experienced a vertical effective stress greater than its present vertical effective stress. An over consolidated (OC) soil is a soil that does have experienced a vertical effective stress greater than its present vertical effective stress. Figure 3 shows the change in void ratio with respect to the effective pressure for the normally and over consolidated conditions. The path a-c in the graph is the NC state of the soil, in which it is subjected to the maximum stress it has ever experienced. The other path (c-d) represents the OC state in which the soil in subjected to less stress than it has ever experienced.

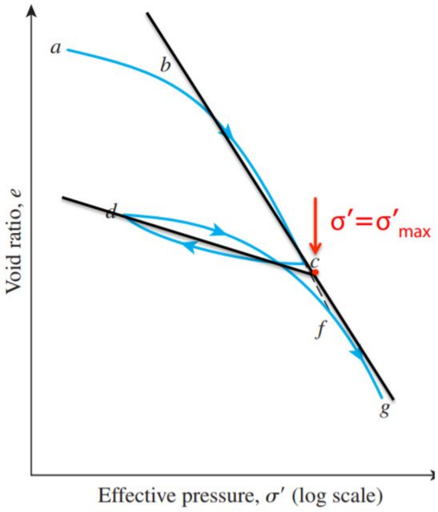


Figure 3: Normally and over consolidated paths (Magnanimo (2017))

Pedrotti and Tarantino (2017) investigated in their research clay in combination with pH 4 water and another combination with pH 9 water. Their research in particular investigated the mechanisms underlying reversible and non-reversible compression. With the methods of MIP (Mercury Intrusion Porosimetry) and SEM (Scanning Electron Microscopy) they could look at micro-level what happened between the clay particles. Previous researches by Sridharan and RAOT (1973) into the mechanisms controlling the volume change of saturated clays found there are two mechanisms controlling the volume change. The first mechanisms is when the compressibility of clay is primarily controlled by shearing resistance at contact points between two clay particles. This is what is also called the edge-to-face configuration. The second mechanism is when the compressibility is primarily governed by electrical repulsive forces. This is called the face-to-face configuration. Their research concluded that in non-active kaolin clay the volumetric change is controlled by the edge-to-face mechanisms, and for active montmorillonite this is the face-to-face mechanism. Pedrotti and Tarantino (2017) used water with pH 4, which is acidic and is comparable with ordinary clay. From literature review they knew that in this configuration the clay has active edge-to-face interaction. They found that the clay is much more compressible than the clay with water of pH 9. The clay with pH 9 water, alkaline water, showed deactivated edge-to-face interactions and had over consolidated-like behaviour. When looking at the pore size distribution the research found that small pore sizes are associated with particles in non-contact configuration, also called the face-to-face configuration. The particles only interact because they overlap and in that way there is a repulsive electrical field generated by the negatively charged faces. The bigger pores are associated with the contact or edge-to-face configuration. This configuration is generated by the attraction between the positively charged edge and negatively charged face of the clay particles. The article also proposed a conceptual micromechanical model for clay. This conceptual model is discussed in more detail in the next subchapter.

In order to investigate the micro mechanics of non-active clays, Pagano et al. (2019b) developed a Discrete Element Method (DEM) model to do virtual experiments into the micromechanics of non-active clays, to validate the conceptual micromechanical model and physical experiments performed by Pedrotti and Tarantino (2017). They modeled the clay particles as rods consisting of small spheres. It was found that the slope of the unloading-reloading lines is depended on the pre-consolidation stress. There were also new discoveries in the overall compression behaviour upon virgin loading and unloading-reloading. With the DEM model they also looked into the conceptual micromechanical model. Further research into the particle orientation found three main mechanisms occurring during compression: (1) the reduction of the distance between particles in face-to-face configuration; (2) the rotation of particles in edge-to-face contact with no contact disengagement; and (3) the permanent loss of edge-to-face contacts. Three different configurations were tested. In the first simulation air was investigated. It was shown that this fabric has the lowest value of anisotropy thanks to the high Coulombian repulsion. For vertical stresses above 40 kPa the fabric deviator increased and particles in edge-to-face configuration started to disengage. Anisotropy means that a material does not have the same characteristics in every direction.(Britannica, 2017) Mechanism 3 occurred and the microstructure of the specimen collapsed. The second simulation investigated acetone. The begin value of the fabric deviator was higher than the value of air. Mechanism 3 already occurred above the vertical stress value of 3 kPa. Above 6 kPa a plateau occurred, after which a stable configuration established and the specimen being insensitive to further stress changes. This behaviour corresponds with the mechanisms 1 and 2. The third simulation looked into the behaviour of the clay with water of pH 9. As was mentioned at this pH-value the edge-to-face contacts are absent, which leads to a high value of initial fabric deviator. The value of the fabric deviator remains almost the same, because the particles rearrange only slightly and there is an absence of irreversible mechanisms.

Next Pagano et al. (2019a) also did a preliminary DEM investigation into the shearing behaviour of non-active clays. Two specimens were investigated: a normally consolidated specimen and an over consolidated specimen. The volumetric behaviour and stress-strain behaviour were shown to be in accordance with literature. (Pagano et al., 2019a) This preliminary simulation study showed that the DEM model is reliable for further experiments in the shear strength of clay, yet the physical experiments on the shear strength of non-active clay are missing. These experiments will help to form a framework based on real data of clay to be able to simulate the behaviour of clay.

As Pedrotti and Tarantino (2017) mentioned in the introduction of their article there has not been any successful research into the micro-mechanics of clays. They investigated experimentally the influence of pH-level and dielectric permittivity in combination with compression on the clay. Pagano et al. (2019b) made a DEM model to virtually test the same settings. Now there is still knowledge lacking about the influence of the micro mechanics on the shearing behaviour of clay.

4.2 Micro mechanisms upon normal stress and shearing stress

First the micro mechanisms that can occur through the appliance of normal stress upon one-dimensional compression and unloading will be discussed. As Pedrotti and Tarantino (2017) mentioned in their article two different forms of contacts between clay particles can occur. When the pH level is below the Point of Zero Charge, the edges of the particles have a positive charge and the faces have a negative charge. Thus in the acidic environment both edge-to-face and face-to-face configurations will occur. For pH levels above the PZC the positive charges of the edges are deleted, thus only face-to-face configurations will occur in this alkaline environment.(Wang and Siu, 2006) Figure 4 shows a schematic overview of the conceptual micro mechanical model that Pedrotti and Tarantino (2017) established after their experiments upon one-dimensional compression and unloading. The contact and non-contact configuration used in the conceptual micromechanical are according to the two mechanisms that Sridharan and RAOT (1973) mentioned of being present in clay at micro level. The model shows the influence of the loading and then unloading of a normal stress on a clay particle.

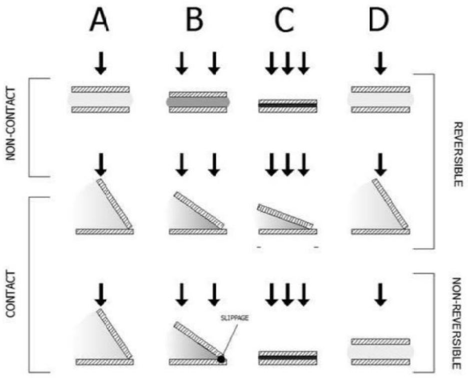


Figure 4: Micro-mechanism associated with contact and non-contact configuration (Pedrotti and Tarantino (2017))

The upper row is in the face-to-face configuration. The only forces that are working between the clay particles are electrochemical forces. That are Van der Waals forces due to temporary dipoles and Coulombian forces due to the permanent charge of the edge and face. Once the vertical stress is removed, the repulsive forces will be the strongest force again. Hence the clay particles will move back to their original configuration. The second and third rows are in the contact configuration. The second row is a reversible mechanism. The two particles will hinge around the contact point when it is put under a vertical stress, and after the stress is removed the two particles will hinge back to their original configuration. The Coulombian forces between the two particles are responsible for this. The repulsive force will push the two particles back to their original position. The third row shows the clay particles that are in contact configuration and are deformed irreversibly. The figure shows that upon vertical stress slippage occurs at the contact point, thus leading to the loss of the edge-to-face contact and the creation of a face-to-face configuration. (Pedrotti and Tarantino, 2017)

The micro mechanisms that occur in kaolin through the appliance of a shear stress are still unknown. The experiments will look on macro level what happens with the shear strength and volumetric behaviour of the kaolin clay mixed with different pore fluids. Looking at the results on macro level, one will suggest what could have happened with the micro mechanisms like the edge-to-face and face-to-face configurations assuming the conceptual micromechanical model of Pedrotti and Tarantino (2017) also applies for clays that are subjected to shear instead of normal stress. The tests on micro level to confirm these suggestions will be out of the scope of this Bachelor Assignment however. It is expected that slippage will also happen when a shear stress is applied on two clay particles in edge-to-face configuration. This means that the edge-to-face configuration is deleted and a face-to-face configuration will form. In the pore fluid with pH 9 there are no edge-to-face configuration anymore, so the mechanism of the deleting of edge-to-face contacts is expected to not happen here.

4.3 Bachelor Assignment

The Bachelor Assignment will try to fill the lack of knowledge about the influence of micro mechanics on the shearing behaviour of kaolin clay by performing physical experiments in a direct shear box. The results can be used to get a better understanding of the micro mechanics under influence of shear and one can add this information to the conceptual micro mechanical model that Pedrotti and Tarantino (2017) already made about the compressibility of non-active clay.

Figure 5 shows the shear strength characteristics of coarse grained soils. (Knappett and Craig, 2012) In the book it is implied that fine soils such as clay will behave in the same way. However, with the knowledge of the previous researches into the microstructure of clay it is expected that clay will show a different behaviour. Clay has a very different microstructure compared to sand. This different behaviour can be tested by performing experiments with different pore fluids.

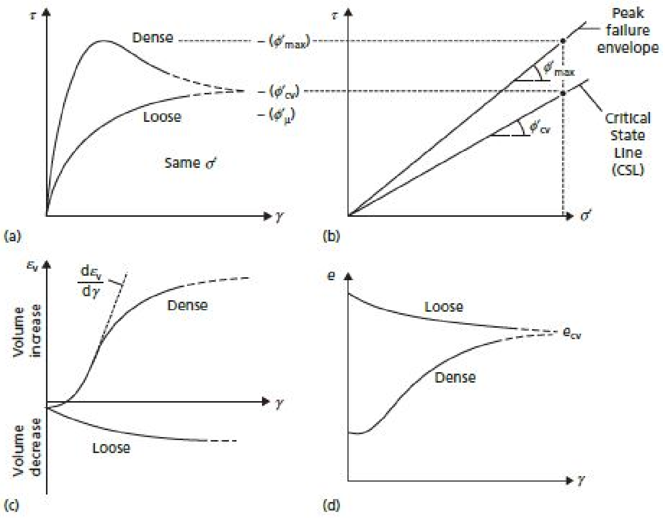


Figure 5: Shear strength characteristics of coarse-grained soils (Knappett and Craig, 2012)

4.3.1 Experimental plan

With the knowledge gained after doing the literature review an experimental plan was established that will support answering the research questions. Table 1 shows an overview of the different pore fluids that will be investigated and their values for dielectric permittivity and pH. Acetone and air are only investigated because they have a different dielectric permittivity than water. Table 1 shows their values for the relative permittivity. The value is relative to the dielectric permittivity of a vacuum, thus they are non dimensional. Their pH value is not of importance for this research. All four different pore fluids will be tested under normally and over consolidated conditions. Three different vertical stress levels will be investigated: 27.25; 95.375 and 299.75 kPa, with a second repetition of every test.

Pore fluid	Dielectric permittivity	pH
Water	80	4
Water	80	9
Air	1	-
Acetone	25	-

Table 1: Overview experimental plan

Figure 6 shows a qualitative compressional curve for the experiments. The blue points are the normally consolidated tests that are on the loading line. The red points correspond to the over consolidated tests that are on the unloading line. The point 763 kPa is the highest stress that was applied on the over consolidated samples before unloading them to the desired vertical stress.

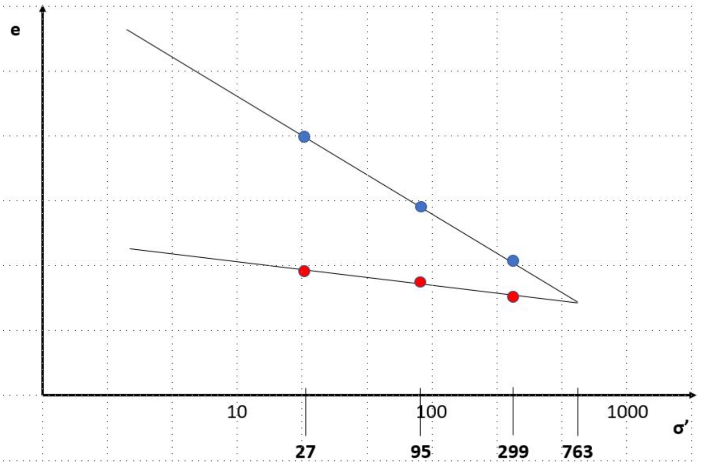


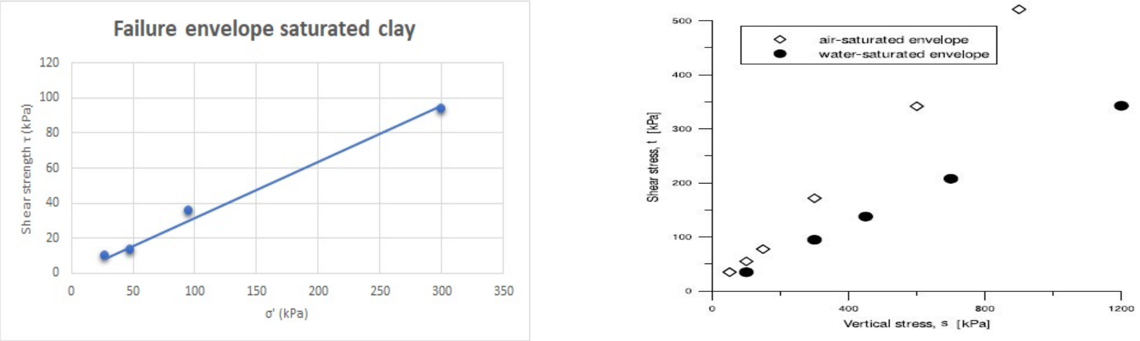
Figure 6: Initial state of NC and OC specimens (before shearing)

5 Preliminary tests

Before performing the actual tests, one has performed some preliminary tests to check the behaviour of the soil. From the results of these tests, an approach was discussed for the actual tests.

5.1 Preliminary tests

During the preliminary tests only water with pH 4 was used as pore fluid. This pore fluid was chosen, because from previous research it was already known what behaviour could be expected. If the results of the preliminary tests would show similar results, one would know that the set up of the experiments is sufficient. The method used for preparing and testing the specimens is the same as the method used for the actual tests. A detailed description of this method can be found in Appendix B. The four tested specimens were in the normally consolidated state. The vertical stresses of 27.25; 47.69; 95.38 and 299.75 kPa were tested. These values lay within the range of vertical stresses which commonly occur in geomechanics. They corresponded to the appliance of a weight of respectively 1.00; 1.75; 3.50 and 11.00 kg. The results produced the graph shown in Figure 7a. The graph shown in Figure 7b is the one from the research of Pedrotti and Tarantino (2018) on effective stresses on the clay microstructure in unsaturated state.



(a) Results preliminary tests

(b) Water-saturated and air-saturated shear envelopes

Figure 7: Graph from test results and graph from Pedrotti and Tarantino (2018)

The research of Pedrotti and Tarantino (2018) tested also higher levels of vertical stress than was tested during the preliminary tests. But when one looks at the second black bullet in Figure 7b it shows that a vertical stress of 300 kPa leads to a shear stress of 100 kPa. Looking at Figure 7a it can be seen that the fourth bullet at a vertical stress of 300 kPa causes a shear stress of almost 100 kPa, 94 kPa to be precise. From these graphs it can be concluded that the set up of the preliminary tests was good and that one can proceed with the actual tests using the same set up. The next step of the research is to perform the actual tests. Since the most difference is expected between the clay with pH 4 water and clay with pH 9 water those two pore fluids will be tested first. Then the acetone and air pore fluids will be tested, but it is expected that they will only slightly differ from the results of the pH 4 water.

5.2 Calibration of load cells

After the preliminary test were finished, the load cells of the shearboxes needed to be calibrated before the actual tests could take place. Calibrating the load cells before the tests would increase the reliability of the results of the tests. For the preliminary tests it was not necessary to have the load cells recently calibrated, since the results were only used to make a rough prediction of what could be expected during the actual tests. One has used a calibration device to calibrate each 500 kg load cell of the shearboxes. The detailed description on the use of the calibration device and the whole calibration procedure can be found in Appendix A. The load cells were dismantled from the shear box and then placed in the calibration device. Weights with a known load were systematically added to the calibration device. For each load it was noted what value of microvolts was produced. A second repetition of adding the same loads was done. Next the microvolts values were plotted against the load in kN and a trendline was plotted. Two other graphs were made in the same way, but now the values of the loading and unloading were separated. The R2 value of the two separate graphs did not differ a lot from the graph with all values, so the trendline of the graph with all values could be used. In excel the formulas for the intercept and slope were added, so one could get a more accurate value with more digits behind the comma. Then these two values were added to the regression file of the load cell to replace the old ones.

6 Method

This chapter will give for each step of the experiments an explanation why this step has to be done. A detailed description of all the steps carried out can be found in Appendix B. There will be four different pore fluids, but for each pore fluid the steps of the experiments will be the same. In the next chapter the results of each pore fluid will be presented and explained individually.

6.1 Oven Drying

The first step of the experiment is oven drying the specimen. In this way, all possible remained water is removed, so each specimen has the same starting condition before it will be mixed with the pore fluid. For the specimen which has air as pore fluid, the second step of mixing the soil is not applicable.

6.2 Soil Mixing

The second step of the experiments is mixing the soil with the pore fluid. For the water pH 4 configuration one needs to make a slurry with a water content of 100%, equal to 1.5 times the liquid limit. For the pH 9 water samples potassium hydroxide needs to be added. For the acetone samples a water content of 100% was used, thus the same weight of acetone should be added to the kaolin powder.

6.3 Shear box - Consolidation phase

Then after the soil is mixed with its pore fluid the first phase of the direct shear test can start. The specimen will be placed in the shear box after which a vertical stress will be applied on it, to start the consolidation. This vertical stress will be applied by placing weights underneath the shear box. After the consolidation phase the soil is completely drained. This will make sure that the excess pore water pressure induced by loading is dissipated, and the applied load is entirely carried by the solid skeleton. All over consolidated samples will be consolidated till a vertical stress of 763 kPa (this corresponds to a weight of 28 kg for the specific shearboxes) and then unloaded to the specific vertical stress: 27.25, 95.375 and 299.75 kPa respectively.

6.4 Shear box - Shearing phase

The fourth step of the experiment is the shearing phase. The normal stress that was applied during the consolidation phase maintains working on the specimen while the shearing takes place. This shearing test will be strain controlled, so one can measure the shear stress applied on the specimen over time. When the shearing phase is done, one can use the data from LabView to derive the shear strength.

6.5 Vacuum packing

After the test was finished the tested specimen needed to be placed in a vacuum back so it could be kept until further research will be done upon them. Keeping the specimen in a vacuum packaging assures that the water content stays the same the whole time, no matter how long ago the actual test took place.

6.6 Water content measurement

The water content of each specimen needs to be measured, so a graph with the initial and final water content can be made.

6.7 Analyzing data from LabView

The last step of the method is to analyze the data that was retrieved from LabView. The consolidation time of each consolidation step has to be found to check whether enough time is left for the consolidation to happen. One has left enough time for consolidation when the slope of the graph starts to get horizontal before adding a new load. Leaving enough time makes sure that the excess pore water pressure induced by loading is dissipated, and therefore the applied load is carried by the solid skeleton only.

7 Results

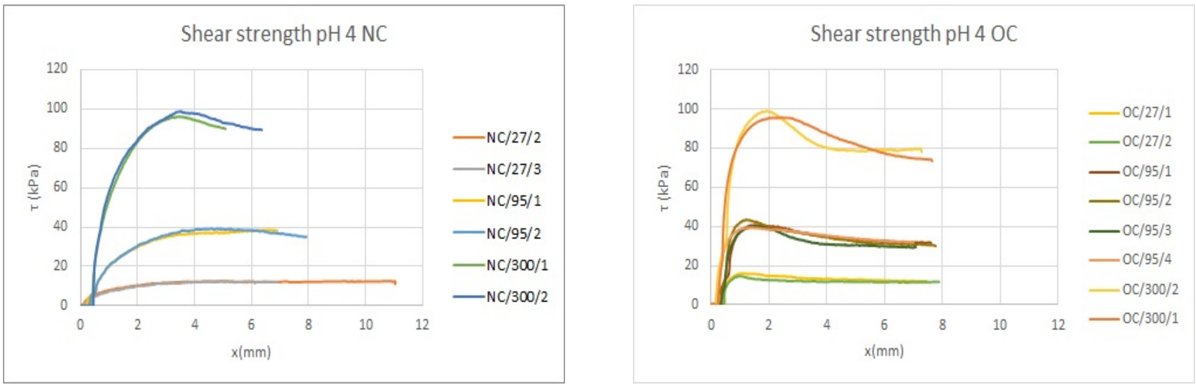
This chapter will show the results of each different pore fluid. To make sure the results of the tests are reliable, one made use of two different shear boxes. Using two different shearboxes would make sure that the behaviour observed was not affected by the experimental set up. The experiments with water of pH 4 were first performed, since one already knew what behaviour could be expected from this configuration. In this way one could check whether the soil was behaving in the way it should and to decide whether the test results were reliable. The results that can be observed from the output graphs will be discussed shortly.

7.1 Water pH 4

Figures 8a and 8b below show the shear stress evolution upon shearing of both normally and over consolidated water pH 4 samples at different vertical stresses. It can be observed in Figure 8a that at all three different stress levels the samples reach a constant value: the critical strength. The samples of 300 kPa show a small decrease in shear strength after a peak is reached. Figure 8b shows the over consolidated samples. They all first reach a peak after which the ultimate strength is reached.

Figures 9a and 9b show the volumetric behaviour of the samples during shearing. As expected the normally consolidated samples show contractile behaviour. All over consolidated samples should have a dilative behaviour, because the vertical stress is decreased right before shearing and thus they should swell. However, not all samples show this behaviour. A lower dilation is observed at higher vertical stresses. This is because there is only a small difference between the point of 300 kPa on the unloading line and the loading line in the compressional curve of Figure 6. It is worth noticing that the peak strength (Figure 8b) occurs after the maximum dilatancy is reached. In Figure 9b one can see that the maximum slope occurs before the peak in the shear strength is observed.

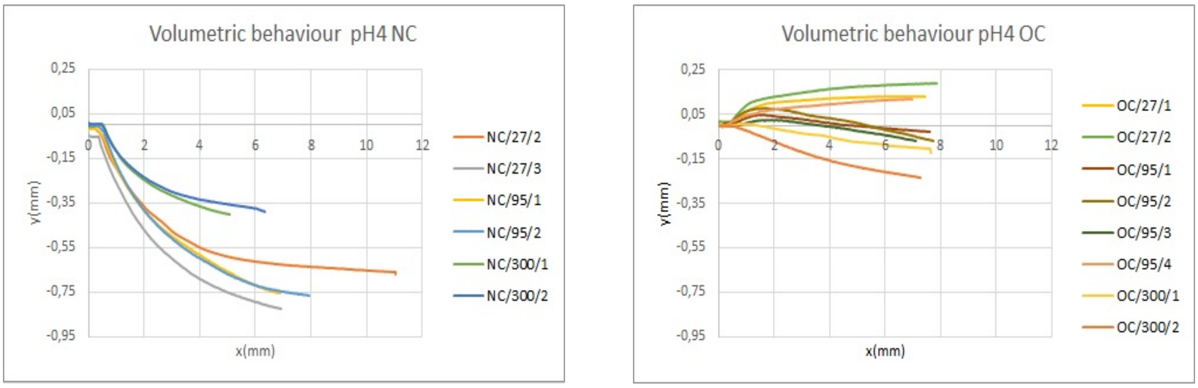
For the sake of comparison, the shear stress evolution of samples OC and NC have been plotted together in Figure 10. It can be observed that this difference between critical and ultimate strength results in different values of the friction angle for over consolidated and normally consolidated samples, thus different slopes of the failure envelopes.



(a) pH 4 water NC

(b) pH 4 water OC

Figure 8: Shear strength graphs pH 4 water



(a) Volumetric behaviour NC (b) Volumetric behaviour OC

Figure 9: Volumetric behaviour graphs pH 4 water

Figure 11 shows the failure envelope of the critical strength, peak strength and ultimate strength of the pH 4 water samples. As can be observed the failure envelope of the ultimate strength (OC) has a friction angle of 15 degrees. The critical strength (NC) however has a friction angle of 18 degrees. This difference in friction angle is not expected: the ultimate strength and critical strength should have the same friction angle and thus the same failure envelope.

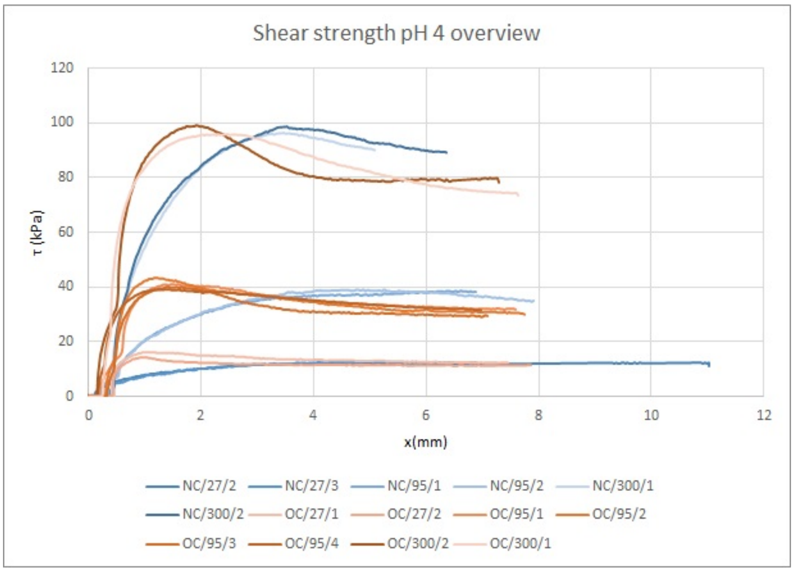


Figure 10: pH 4 water

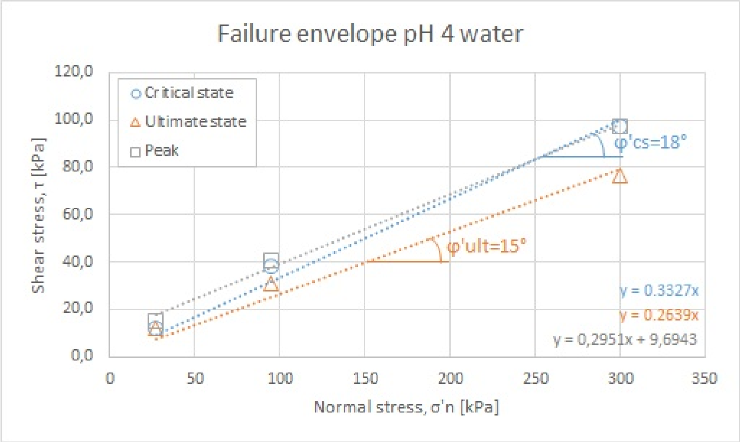


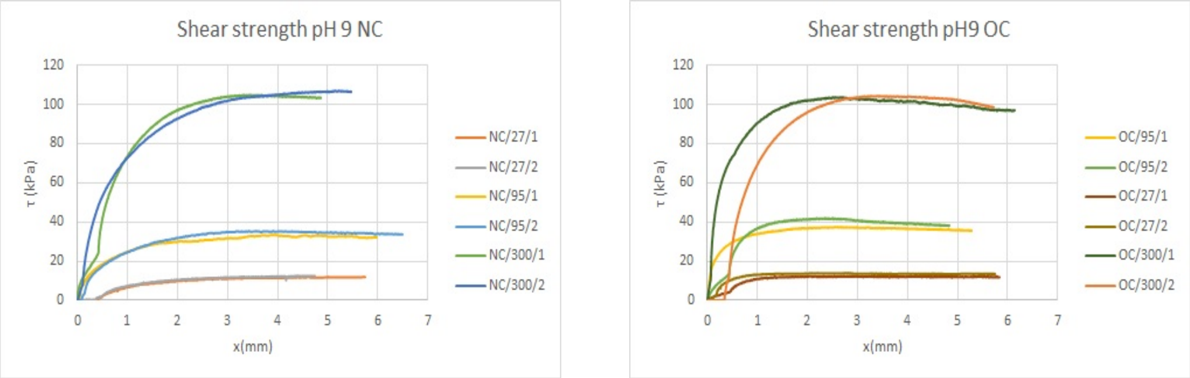
Figure 11: Failure envelopes pH 4 water

7.2 Water pH 9

Figures 12a and 12b show the results in shear strength of both normally and over consolidated pH 9 water samples. The normally consolidated samples reach the critical strength, the over consolidated samples reach the ultimate strength. However, in this case no peak can be observed in the graph of the over consolidated samples. This was not expected.

Figures 13a and 13b show the volumetric behaviour of both normally consolidated and over consolidated samples. The normally consolidated samples show contractile behaviour as expected, however, again the over consolidated samples show both contractile and dilative behaviour. The absence of a peak strength in the samples showing a dilative behaviour is indeed unexpected.

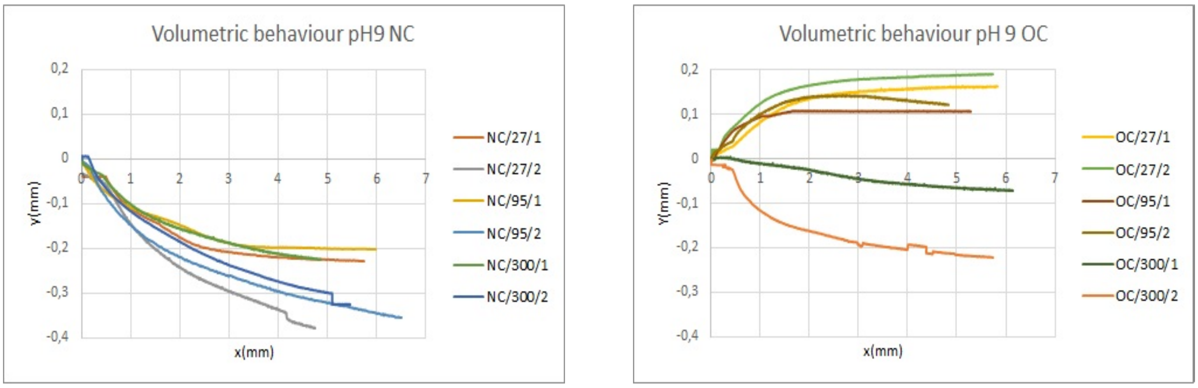
By comparing both normally and over consolidated samples in Figure 14 one can see that both the ultimate and critical strength have the same value.



(a) pH 9 water NC

(b) pH 9 water OC

Figure 12: Shear strength graphs pH 9 water



(a) Volumetric behaviour NC

(b) Volumetric behaviour OC

Figure 13: Volumetric behaviour graphs pH 9 water

Figure 15 shows the failure evelopes of the critical strength, ultimate strength and peak strength of the water pH 9 samples. As can be observed all three failure envelopes have the same friction angle of 19 degrees. This behaviour is expected, because both ultimate and critical strength should end up at the same value and thus at the same failure envelope. However, the peak envelope would normally exhibit a different friction angle (as can be seen in pH 4), which is not the case here due to the absence of a peak strength in over consolidated samples.

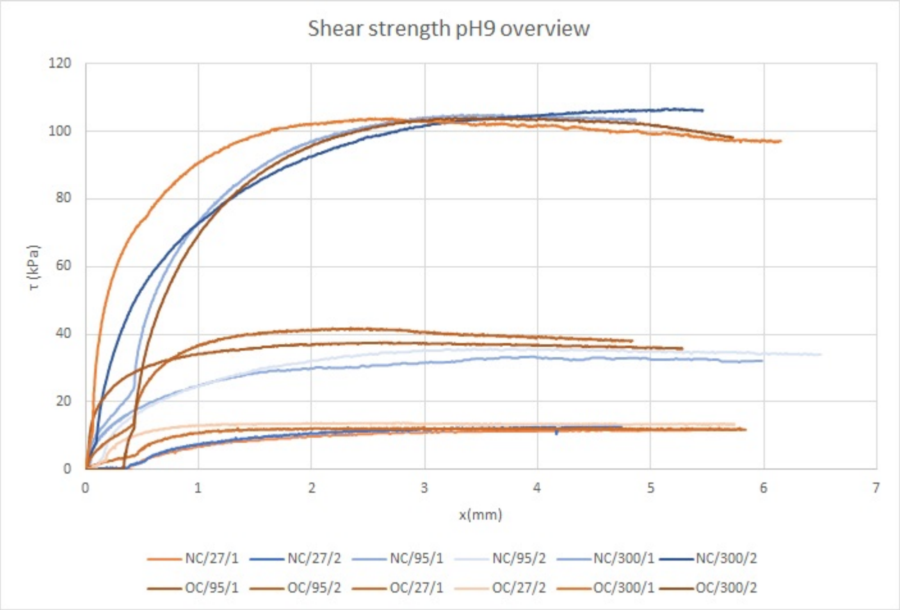


Figure 14: pH 9 water

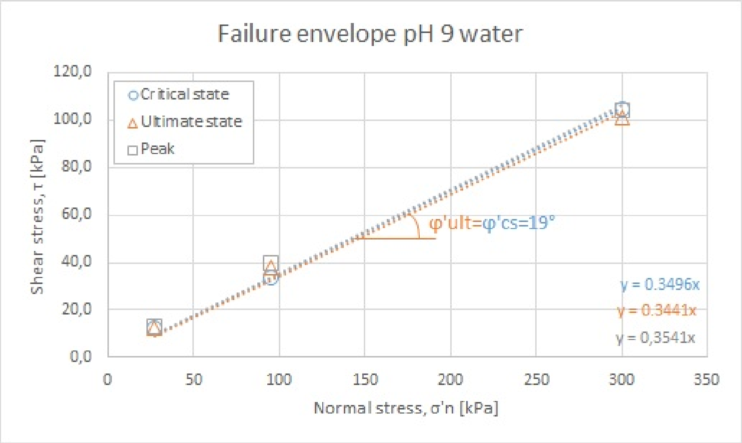
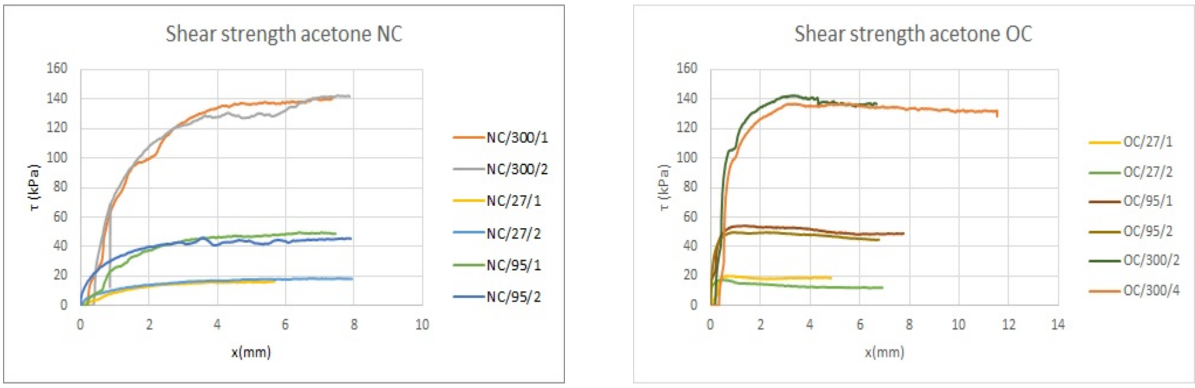


Figure 15: Failure envelope pH 9 water

7.3 Acetone

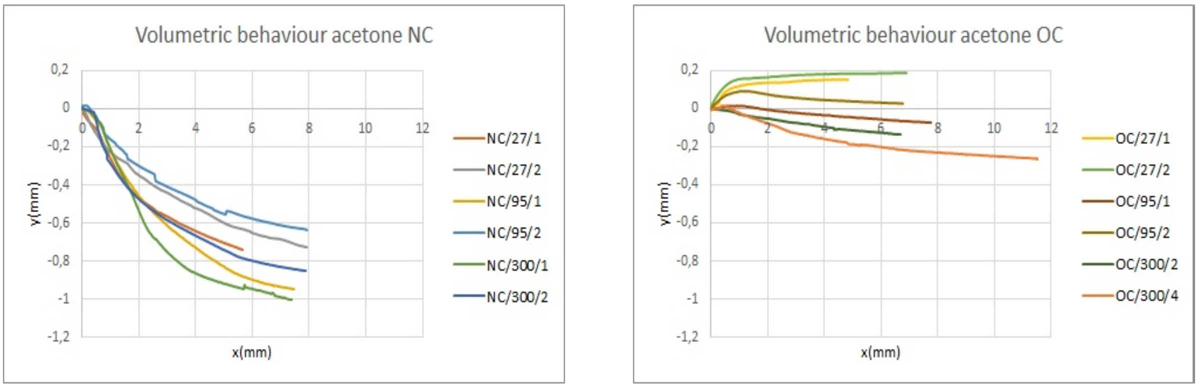
Figures 16a and 16b show the results in shear strength of all acetone tests. It can be observed that all normally consolidated samples reach the critical strength. For the over consolidated samples it can be observed that there is a small peak, after which the ultimate strength is reached. Figures 17a and 17b show the volumetric behaviour of the acetone samples. Again, the normally consolidated samples have contractile behaviour and the over consolidated samples show both contractile and dilative behaviour, even though only dilative behaviour was expected.

Looking at Figure 18, when one assumes that the lines still would continue for a while, it can be observed that the ultimate strength (OC) is smaller than the critical strength (NC), as was also observed with the water pH 4 samples.



(a) Acetone NC (b) Acetone OC

Figure 16: Shear strength graphs acetone



(a) Volumetric behaviour NC (b) Volumetric behaviour OC

Figure 17: Volumetric behaviour graphs acetone

Figure 19 shows the failure envelope of the critical strength, ultimate strength and peak strength of acetone. The failure envelope of the ultimate strength is 23 degrees and 25 degrees for the critical strength. This is a small difference in angle, which is not expected.

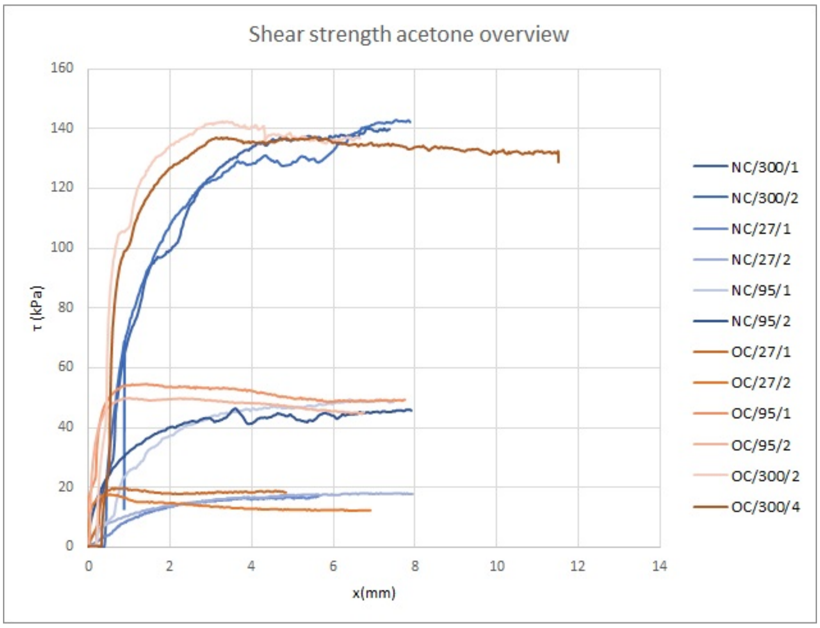


Figure 18: Acetone

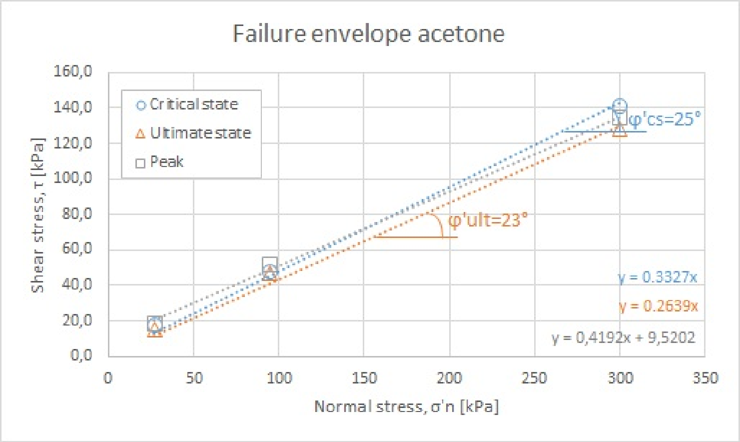
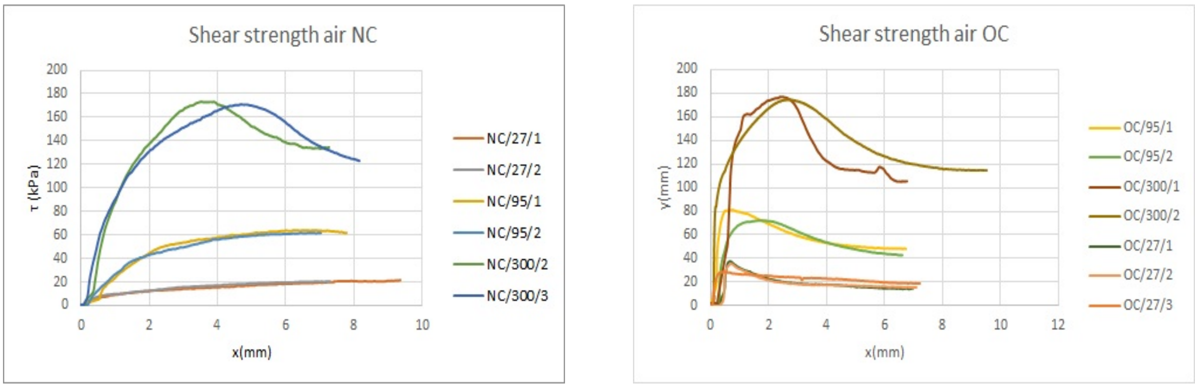


Figure 19: Failure envelope acetone

7.4 Air

Figures 20a and 20b show the results of the shear strength of the air samples. For the normally consolidated samples, the 27 and 95 kPa ones show the expected behaviour, but the 300 kPa samples seem to reach a peak and then decrease in shear strength. The overconsolidated samples show the expected behaviour, however the difference between the peak strength and ultimate strength for the 300 kPa samples is really big. Figures 21a and 21b show the volumetric behaviour of both normally and over consolidated samples. Also with this pore fluid, the normally consolidated samples show the expected behaviour. However again, the overconsolidated samples show both contractile and dilative behaviour.

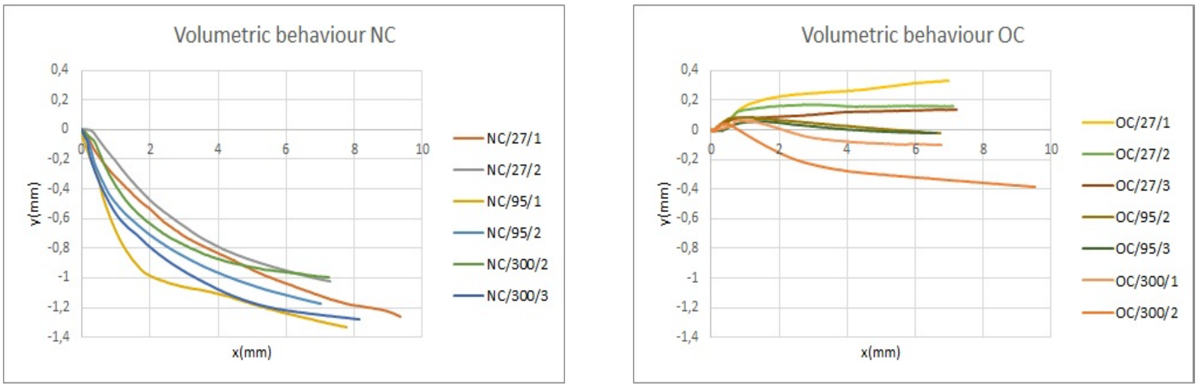
Figure 22 shows a comparison between the normally and over consolidated samples. It can be observed that at a vertical stress of 300 kPa the peak of the over consolidated samples almost corresponds with the peak of the normally consolidated sample.



(a) Air NC

(b) Air OC

Figure 20: Shear strength graphs air



(a) Volumetric behaviour NC

(b) Volumetric behaviour OC

Figure 21: Volumetric behaviour graphs air

Figure 23 shows the failure envelope of the critical strength, ultimate strength and peak strength of the air samples. The failure envelope of the ultimate strength has a friction angle of 20 degrees. The friction angle of the critical strength is 30 degrees. This is a very big difference. Both should have the same failure envelope, but instead the failure envelope of the critical strength is 50 percent bigger than the ultimate strength failure envelope.

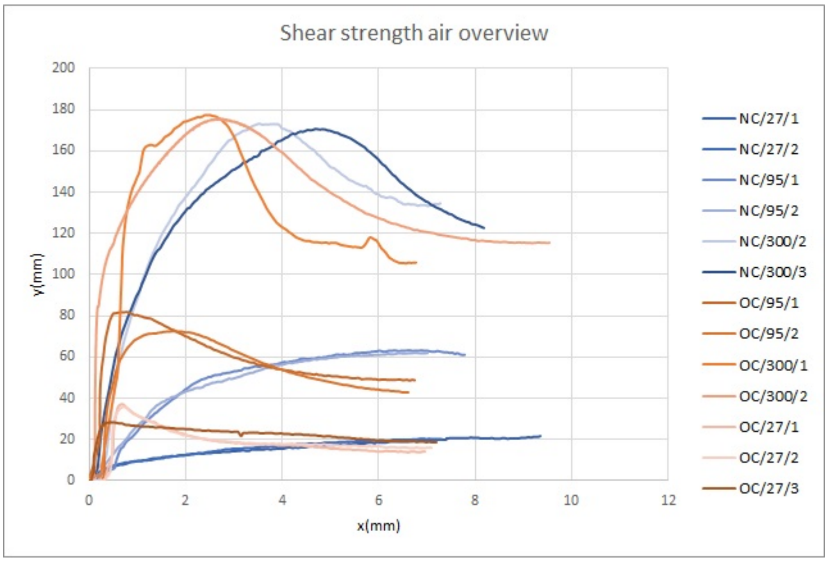


Figure 22: Air

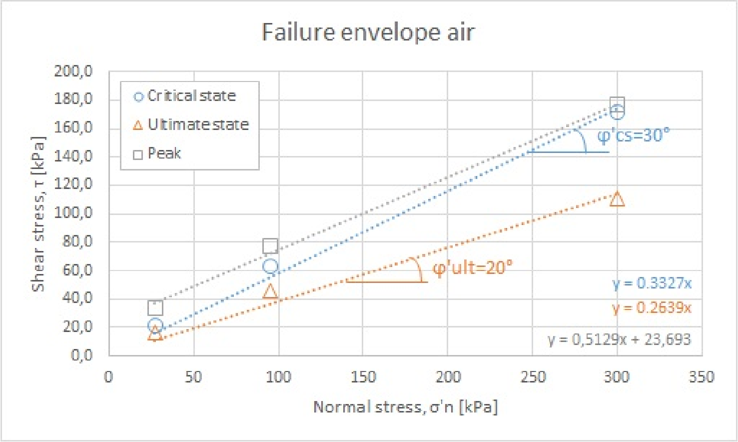


Figure 23: Failure envelope air

8 Discussion

This chapter will discuss the results that were found and what one might suggest is the explanation for the observed behaviour. A short recap of the most unexpected results:

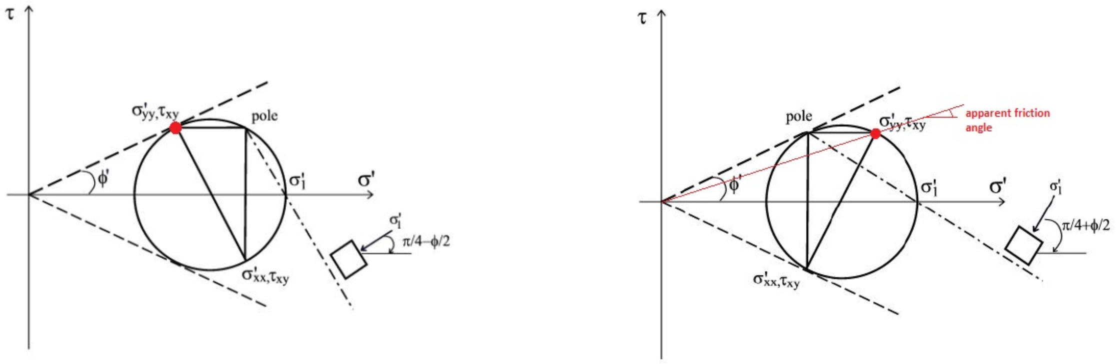
- a difference between the ultimate strength and the critical strength for the pH 4 water samples
- for over consolidated pH 4 water samples the peak strength was reached after the maximum dilatancy occurred
- for over consolidated pH 9 water samples there was no peak in shear strength, even though they showed dilative behaviour
- different pore fluids showed different failure envelopes. This is expected under the assumption that the microstructure of the clay is changed by changing the pore fluid

Some possible explanations for these observations will be discussed in the following subsections.

8.1 Interpretation of direct shear test using the Mohr circle

For the pH 4 samples the failure envelope of the critical strength has a friction angle of 18 degrees, whereas the failure envelope of the ultimate strength has a friction angle of 15 degrees. This difference in friction angles might be explained by the way one interprets the failure envelope in the Mohr Circle. A short explanation about the Mohr Circle follows.

Figures 24b and 24a show the different Mohr circles for either a failure on the horizontal plane or vertical plane. Generally, one would assume that the critical values of tau and sigma are lying on that specific point of the Mohr Circle that touches the failure envelope. In this case Figure 24a would apply and the failure would have happened on the vertical plane. However, for the ultimate strength is was observed that the friction angle was smaller than the known friction angle of kaolin. This smaller friction angle could be explained by the fact that one assumes that the critical values of tau and sigma are lying on the failure envelope as in Figure 24a. But in fact, the critical value point would be lying somewhere else on the Mohr Circle, as is shown in Figure 24b. As can be seen there is still a critical value lying on the failure envelope, the lower failure envelope. This value could not be measured by this research, because one only knew the vertical sigma that was acting on the soil. This could explain why the friction angle of the ultimate strength (OC) is lower than the friction angle of the critical strength (NC). Instead of touching the upper failure envelope, it touches the lower failure envelope. Thus this would imply that the over consolidated samples fail on the horizontal plane. The value of the critical strength would be corresponding with the critical failure value in Figure 24a. Thus this would imply that the normally consolidated samples failing on the vertical plane.



(a) Mohr's Circle for failure on vertical plane (Tombolato, 2007)

(b) Mohr's Circle for failure on horizontal plane (Tombolato, 2007)

Figure 24: Mohr Circles for failure on horizontal and vertical plane

Looking at the Mohr circle explains the difference between the critical strength and ultimate strength for the pH 4 samples, but it does not explain the occurrence of the peak strength after maximum dilatancy for pH 4, and the absence of a peak strength for the pH 9 samples. The following subsection might give an answer for that.

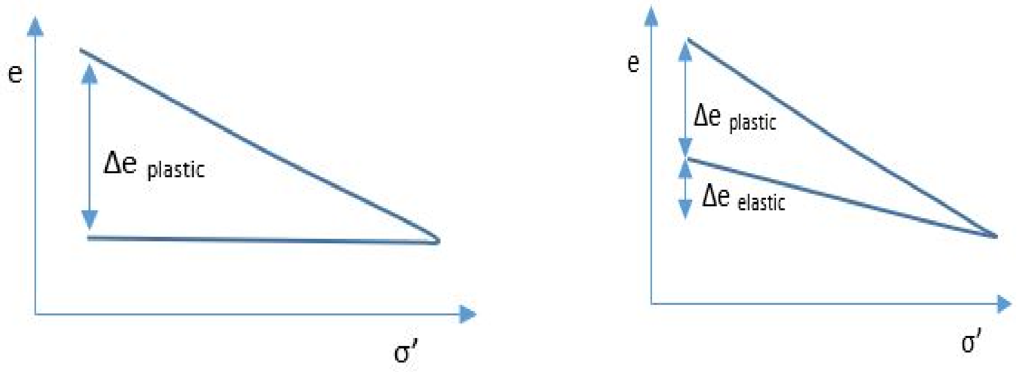
8.2 Dissipation of energy upon shearing: sand versus clays

Another explanation might be given by looking at the Taylor's equation. This equation has been developed for sandy soils by Taylor (1948), and explains the relationship between volumetric behaviour upon shearing and the shear stress evolution. Taylor's equation (see Formula 8.1) assumes that all energy is dissipated from friction only. The equation comes from the an equilibrium between the work done by the external forces and the work done by the internal forces. This equation holds for sands, but it is not sure whether it also holds for clays. The formula says that the internal friction angle μ is constant over time. Now when $\frac{\delta y}{\delta x}$ is maximum, the value of $\frac{\tau}{\sigma}$ is maximum, thus one should observe a peak in the shear strength. When $\frac{\delta y}{\delta x}$ is 0, $\frac{\tau}{\sigma}$ is equal to μ, thus the friction angle can be retrieved by looking at that part of the graph. This is the part where the critical strength for normally consolidated soils and ultimate strength for over consolidated soils are observed.

$$\frac{\tau}{\sigma'_n} = \mu + \frac{\delta y}{\delta x} \tag{8.1}$$

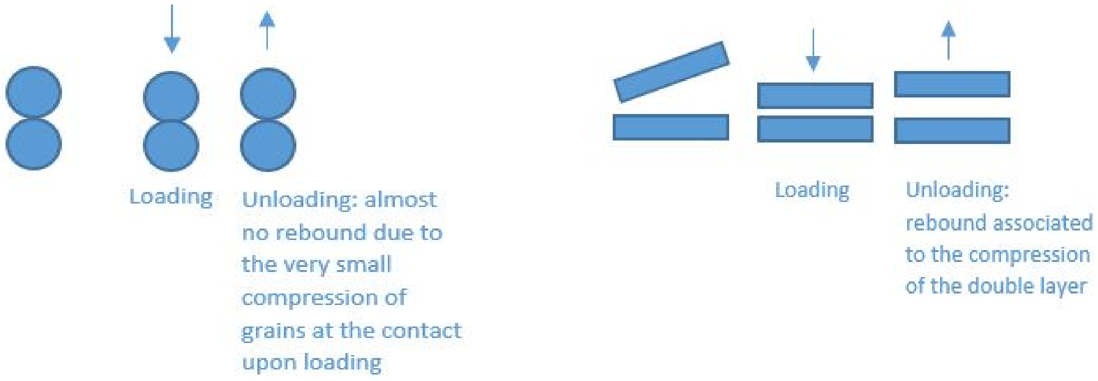
Now for clay this equation might not hold. When looking at the shearing and volumetric behaviour of the over consolidated pH 4 water for example (see Figures 8b and 9b) one can see that the volumetric behaviour already reached its maximum dilatancy before a maximum could be observed in the shearing graph. One might argue that for clayey soils both plastic and elastic energy needs to be taken into account in the equation, since elastic energy is not negligible for clays. If this assumption were to be true this could also explain the absence of a peak for the pH 9 overconsolidated samples and the difference in ultimate strength and critical strength for pH 4 water. Because one needs to add the elastic energy this could also explain why the peak of the shear strength is shifted.

The fact that elastic energy is not negligible in clay can be explained by comparing sand and clay on micro and macro level. Figure 25 shows the macro and micro level behaviour of both sand and clay.



(a) Sand (macro)

(b) Clay (macro)



(c) Sand (micro)

(d) Clay (micro)

Figure 25: Micro and macro level behaviour sand and clay

At the macroscopic level, sands that have been subjected to one-dimensional first loading exhibit negligible elastic deformation upon unloading, thus there is almost no recovery of void ratio. (Figure 25a) This is associated to the fact that, at the microscale, the elastic rebound would only be associated with the decompression of sand grains at the contact: because of the high stiffness of the grain-to-grain contact, the resulting rebound upon unloading is almost negligible. (Figure 25c)

On the other hand, clays exhibit higher elastic rebound upon unloading at the macroscopic level. (Figure 25b) This is associated to the decompression of the double layer between clay particles at the microscale (Figure 25d), which is almost a fully reversible (elastic) mechanism and, non negligible elastic rebound. This difference could be taken into account by adding a term to the Taylor's equation. Prof. Tarantino derived a new equation, taking into account the hypothesis of linear elasticity. (Formula 8.2)

$$\frac{\tau}{\sigma'_n} = \mu + \frac{\delta y}{\delta x} + [\frac{\tau}{\sigma} * \frac{\tau}{G} - \frac{\sigma}{E_oed} - \mu * \frac{\tau}{G}] * \frac{\delta h}{\delta x} \tag{8.2}$$

The elastic term between brackets can be neglected for sand, since G and E are very high for sand. Meaning the Taylor's equation would still look like it is known now (Formula 8.1) for sand. However, for clay this elastic energy term must be taken into account, since G and E are smaller. It can also be observed in the proposed formula that the elastic energy decreases when dx increases. This could explain why the peak strength is shifted to the right: one needs to add the elastic energy. This is visualized in Figure 26.

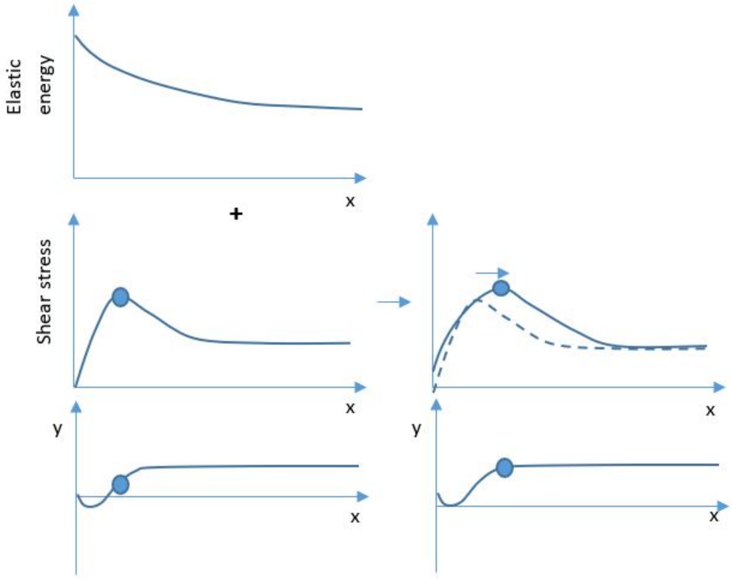
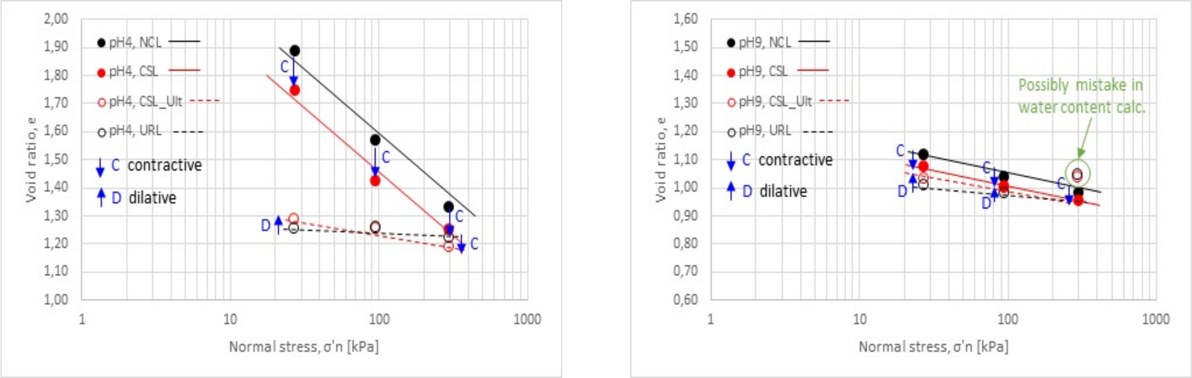


Figure 26: Addition of elastic energy to shearing stress

If this new formula would be correct, this means it is also expected that the strength should still decrease at very high horizontal displacement, also for normally consolidated samples and samples at pH 9, since the elastic energy term would not play a role anymore.

8.3 Non uniqueness of the critical state line: the effect of microstructure

As was mentioned before the shearing behaviour of sand and clay cannot be interpreted in the same way, since they are controlled by different mechanisms. To prove this further, the results of the direct shear tests for pH 4 water and pH 9 water have been plotted. Figures 27a and 27b show the normal consolidation line (NCL), critical state line (CSL) and unloading-reloading line (URL) in the plane e-sigma'. Looking at Figure 27a, for normally consolidated samples one starts at the black normally consolidation line. Upon shearing, the void ratio changes and moves to the red critical state line. This line should be unique for the same soil. Now when over consolidated tests are performed at the beginning of shearing one starts from the dotted black line: the unloading reloading line. According to theory during shearing the over consolidated samples should swell and move upwards in the graph until they reach the critical state line. However, as can be observed in Figure 27a, the void ratio only moves up till the red dotted line. This is unexpected behaviour. Now when one looks at Figure 27b, one can see that for pH 9 water the critical state line still is unique and shows the expected behaviour.



(a) Water content pH 4 water

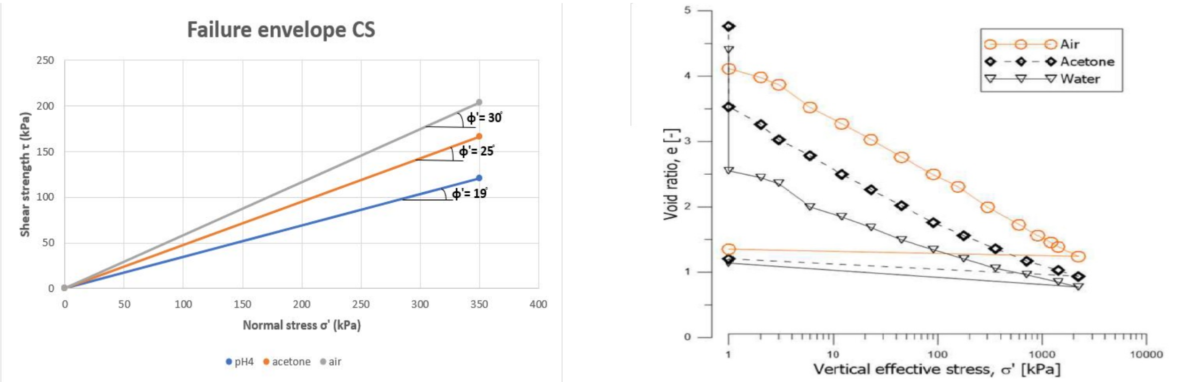
(b) Water content pH 9 water

Figure 27: Difference in initial and final water content pH 4 and pH 9

This difference in CSL for normally and over consolidated pH 4 samples could be explained as follows. The over consolidated samples should dilate a lot in order to move back to the original CSL. This is however not possible, since the edge-to-face contacts have been permanently destroyed when applying the pre-consolidation stress of 763 kPa. This could explain why the void ratio only has a small recovery: if the edge-to-face contacts were still active they would be responsible for the big increase in void ratio back to the CSL. This might also explain the fact that the ultimate strength is lower than the critical strength. One does not see this behaviour in pH 9 water, where both normally and overconsolidated samples only contain face-to-face contacts.

8.4 Effect of dielectric permittivity on the shear strength

The differences between the behaviour of samples saturated with water at pH 4 and pH 9 have been extensively described so far. A few points about the influence of dielectric permittivity will be discussed in this section. Samples saturated with air and acetone also exhibited a behaviour similar to water at pH 4, with ultimate strength being lower than critical strength. However, the values of friction angle corresponding to the same state for the three different pore fluids were found to be extremely different. For the sake of comparison, the failure envelopes at critical state of samples saturated with water (pH4), air and acetone have been plotted together in Figure 28a. It can be observed that air had the biggest friction angle, followed by acetone and then water. This is consistent with the change in dielectric permittivity. This is similar to the behaviour that was observed by Pedrotti and Tarantino (2017) during their 1D compression tests on the same fluids mixed with kaolin. This demonstrates that the difference in behaviour happens not only upon 1D compression, but also upon shearing. Why this difference in dielectric permittivity causes a sample to be stronger or weaker could be explained by looking at the microstructure. The higher the dielectric permittivity, the higher the repulsion and thus on microlevel the angle between the two particles in edge-to-face configuration.



(a) Failure envelopes for fluids with different dielectric permittivity

(b) Void ratio for different fluids upon 1D compression (Pedrotti and Tarantino, 2017)

Figure 28: Difference between pore fluids upon shearing and 1D compression

All these explanations of the observed behaviour on macro level are done by suggesting behaviour that might have happened on micro level. However, this research did not look into the micro level. Thus to be sure about the suggested behaviour on micro level one needs to perform tests looking into the behaviour on micro level. This is outside the scope of this Bachelor Assignment.

9 Conclusion

This chapter will draw some short conclusions that can be made from the previous chapter and the results of the experiments. The answers on the research questions will be discussed shortly.

The first research question was to find out the influence of the different OCR states on the behaviour of non-active clay. For the pH 4 water samples it was observed that the normally consolidated samples reached a critical strength. The over consolidated samples however first reached a peak and then reached the ultimate strength, as expected. There was a difference in the value of the critical strength and ultimate strength, even though these two should have been the same. The previous chapter suggested that this difference in strength is not due to the difference in OCR states, but the difference in microstructure between the normally consolidated and over consolidated samples.

The second research question was to look into the influence of the different pH-levels on the shear strength of the non-active clay. As can be seen in the graphs, the samples with pH 9 water never show a peak in the over consolidated state. However, in the pH 4 samples, which still have edge-to-face configurations, there is a peak observed in the over consolidated state.

The third research question was to look into the influence of the dielectric permittivity on the shear strength of non-active clay. As was discussed in the previous chapter the shear strength of the air samples turned out to be the biggest, whereas the shear strength of the water samples was the smallest. The dielectric permittivity thus indeed influences the shear strength of the non-active clay. It was also suggested that the dielectric permittivity on itself might not be directly responsible for the difference in shear strength. The dielectric permittivity might influence the angle between the two particles in edge-to-face configuration, and this change in microstructure would cause the difference in shear strength.

The fourth research question was to find out the micro mechanical mechanisms responsible for the difference in shear strength. Combining the answers of the previous three research questions can explain the answer on this research question. As was suggested in the previous chapter the two different configurations of edge-to-face and face-to-face and their presence or absence might be responsible for the difference in shear strength observed. It was suggested that samples with both edge-to-face and face-to-face contacts would fail on the vertical plane and samples with only face-to-face contacts would fail on the horizontal plane. Whether this is true must be confirmed by performing experiments on micro level on the clay samples.

The last thing that can be concluded from what is mentioned in the discussion is that the relationship between the volumetric behaviour and shear stress evolution are not linked by the same relationship that governs the shearing mechanisms in sand. This is a speculation up until now, which needs further analysis at a quantitative and experimental level.

10 Recommendations

This chapter will give some recommendations for further research.

The first recommendation is to investigate the pore size distribution of the tested samples making use of the MIP (Mercury Intrusion Porosometry) method. All samples were vacuum packed after shearing, so they are still in the same condition as at the end of the shearing phase. By checking the pore size distribution one can check how much edge-to-face and face-to-face configurations are left in the clay. Thus it could be confirmed that indeed all edge-to-face configurations were deleted in the pH 9 water samples or destroyed for the over consolidated samples. If indeed no edge-to-face configurations are left in these samples the suggestions made in the previous chapters would be confirmed.

Another recommendation for further research is to use the MIP method at different shearing stages. So one would stop the shearing when the peak strength is reached for the over consolidated samples and see what the pore size distribution is at that specific value of horizontal displacement.

The third recommendation is to perform more tests, but on higher values of vertical stress. As was said before the presence of the edge-to-face contacts also depends on the amount of vertical stress that is applied. It is expected that the edge-to-face contacts are destroyed above a certain value of vertical stress. This would mean that also normally consolidated samples would show a decrease shear strength after reaching a peak strength, because edge-to-face contacts are destroyed.

And the last recommendation is to perform tests at a higher horizontal displacement. The tests that were performing during the bachelor assignment were stopped around a displacement of 7 mm. But to check whether the assumption made about the Taylor's equation and the addition of an elastic energy term is true, one needs to perform the tests till a higher horizontal displacement. As was mentioned in the discussion it is expected that at a higher vertical displacement the influence of the elastic energy term will be smaller, thus leading to a decrease in shear strength. In the end all samples should get the same failure envelope.

11 Relevance

This chapter will briefly describe the relevance of this research for civil engineering.

Knowing the influence of different pore fluids on the microstructure and thus strength and behaviour of clay can give insight in what failure of the soil can be expected. This is the case for quick clay. Several articles discuss landslides in quick clay. The article of Geertsema et al. (2006) explained the occurrence of landslides in glacial marine sediments. These sediments occur in eastern Canada and Scandinavia. When the land was glaciated around 20 000 years ago the clay was submerged into the sea and through absorption of seawater and its salts it gained a flocculent structure. As can be seen in Figure 1 of the literature review this structure is very stable. After the glaciers disappeared and the vertical stress was decreased the land rose again, thus leaving the clay not submerged anymore. During heavy rainfall the salts in the clay can be washed out, thus changing the microstructure of the clay. With the knowledge of the bachelor assignment one knows that the microstructure of the clay changes from a both edge-to-face and face-to-face configuration to a face-to-face configuration. This matches the dispersed structure in Figure 1. Because only the face-to-face configuration is left, the clay quickly fails. In the past a lot of landslides occurred in Canada and Norway because of the change of the microstructure of the clay. The problem with quick clay is that nowadays it is mostly covered with normal soil, thus people do not know there is quick clay in the subsurface. Only soil tests can prove this.

To prevent more accidents happening to people after landslides in quick clay it is thus of importance to know the influence of different pore fluids. As explained one already know what happens to the microstructure after heavy rainfall, but one does not know yet what happens when the clay is submerged in a fluid with a different dielectric permittivity or pH level, for example because of contamination. This ongoing research of the University of Strathclyde could provide more knowledge on this subject.

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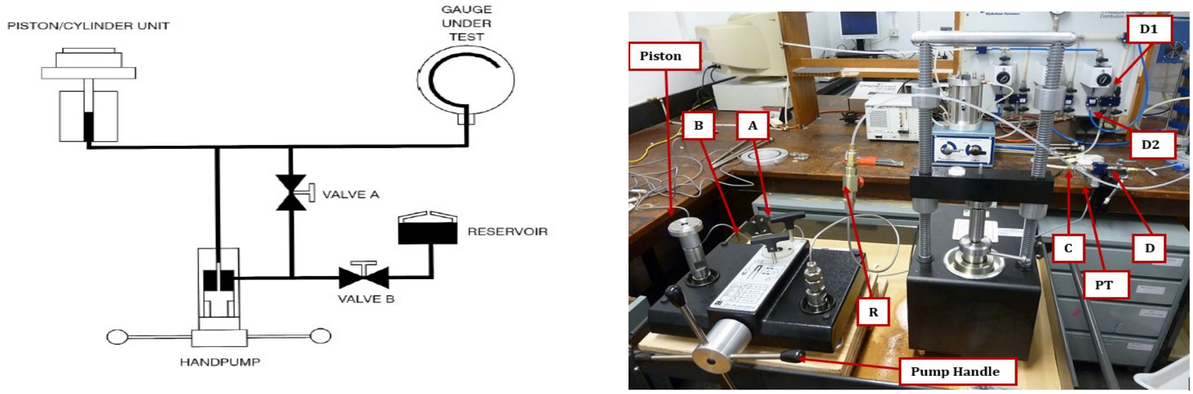
A Preliminary tests detailed description

This appendix contains a more detailed description of the calibration process performed after the preliminary tests. The same method was used for the preliminary tests as was used for the actual tests. The detailed description can be found in Appendix B. During the preliminary tests the water content test wasn't performed. Next a detailed description of the calibration process is given. These steps are retrieved from the manual of the device. (Budenberg, 2019)

A.1 Calibrating method

A.1.1 Preparing calibration machine



(a) Schematic overview DH Budenberg Hydraulic Dead-Weight Tester

(b) Parts of calibration machine

Figure 29: Schematic overview and real life picture of calibration machine

1. First the system needs to be primed. Open valve A and B and fill the system with oil from the reservoir. A schematic overview of the device can be seen in Figure 29a.
2. Close valve B and keep valve A opened so one can perform the lower test pressures.
3. Connect the oil outlet to the pressure transducer. Make sure the tube is fully saturated with oil before attaching it to the pressure transducer. This can be done by opening the pump handle slightly. When a drop of oil comes out of the tube, one can move the pump handle back to its original place and attach the tube to the pressure transducer.
4. Dismantle the load cell from the shear box and check which channel the load cell is at by pressing a bit on the load cell and see what channel changes in value.
5. Next change the units on the computer from Engineering Units to microVolts.
6. Place the load cell on the pressure transducer by using two metal balls on the bottom and top and adjust the black beam, so it fits the load cell.

A.1.2 Calibration of load cell

1. Check the initial value in microVolts of the channel that the load cell is at and write this down.
2. Check the maximum load of the load cell. The load cell can have 500 kg, which is equal to a load of 5 kN. The guideline of the calibration machine mentioned that 1 bar is equal to 0.4 kN in this case. The maximum load is thus 25 bar.
3. It was chosen to apply a maximum load of 22.5 bar. Start with 1 bar. Place the load on the piston and open the pump handle, keep opening it until the load starts to float on the oil. So the piston may not be moved to its highest position.
4. When the load floats on the oil, start rotating the load slightly and look at the value on the computer. The value changes a bit, but try to find the average and write this down.
5. Repeat step 3 and 4 for the other loads until one reaches 22.5 bar. Start with 1, then 1.5, then 2.5, then 4.5, then 8.5, then 12.5 and then 13.5. Then start unloading the piston and also write down these

values for the different loads. When one's at 1 bar again, replace the load for 20 bar, write the value down and add another 22.5 bar and write the value down. These two values don't need to be unloaded.

6. Repeat steps 3,4 and 5 for two to three times. Now one can take the data to analyze it in Excel.

A.1.3 Analyzing data

1. Put all the values in an Excel sheet. Calculate the values in bar to kN. As was said, 1 bar is equal to 0.2 kN. The values in kN will be placed on the y-axis. The values in microVolts will be placed on the x-axis. Mark the values that were acquired during the unloading of the piston.
2. First plot all values in a graph with the kN on the y-axis and the microVolts on the x-axis. Plot a trendline through the points and make the formula of the trendline and the R2 value visible.
3. Next plot another graph, but now with only the values of the unloading. Do this also for only the loading values.
4. Use the commands in Excel to find the slope and intercept of the trendline for all values. Now one gets as much digits behind the comma as possible.
5. With those values calculate the calculated value of y using the x-value in microVolts. Calculate the difference between the calculated y and the measured y for each x-value.
6. Calculate the average difference in kN. The load cell has an allowed difference of 0.1 percent of the total load it can bear. This means a difference of 0.005 kN is allowed. Compare the allowed difference with the actual difference and when the actual difference is higher than the allowed difference one should redo the calibration.
7. The last step is to process the calibrated values of the slope and intercept in the computer. Look for the regression document that is the input for the load cell and change the values of the slope and intercept. Just in case something went wrong, make sure to write down the old values.

B Detailed description method

This appendix will give a more detailed description of the steps performed during the experiment, so one could replicate the experiments done.

B.1 Oven Drying

1. Check whether the scale is properly balanced.
2. Put a glass jar on the scale and press 'tare', so the scale shows 0 weight.
3. Get kaolin powder out of the storage box with a spatula and put it in the glass jar. Each sample needs 60 grams of kaolin, but a little extra kaolin is desirable, since more could be needed for making in the high water content slurry.
4. Put the glass jar in the oven by making sure one is wearing gloves.
5. When the kaolin is left in the oven overnight, all the water that may be left should be removed. Since it is already a powder the water content should be really minimal already.

B.2 Soil Mixing

1. Check whether the scale is properly balanced.
2. Put a plastic beaker on the scale and press 'tare'.
3. For pH4: Put in another plastic beaker deionised water. For the pH 9 samples one weighs 45 grams of deionised water and adds 3 flakes of KOH to it. Then in a small tray 1 gram of deionised water should be added with one flake of KOH. For the acetone samples one fills the beaker with acetone instead of water.
4. Get the glass jar from the oven by making sure one is wearing gloves.
5. Put the oven dried kaolin in the plastic beaker up until 60 gram. For the pH 9 samples 100 grams should be added.

6. For pH4: Add the same amount of deionised water in the plastic beaker with kaolin making use of a syringe. When the amount of kaolin wasn't exactly 60.00, one should add the same extra amount of deionised water. For the pH9 samples one should first add the 45 grams of deionised water, mix this, then add the 1 gram of deionised water with one flake. The last step is to add another 2 grams of deionised water and mix it all together. For the acetone samples one adds the exact same amount of acetone to the kaolin powder as the weight of the powder.
7. Mix the kaolin with the pore fluid making use of a spatula or spoon. Make sure no lumps are left.

B.3 Shear box - Consolidation phase

1. Make sure all parts of the shear box are cleaned and dried before use, except for the two metal porous plates. They must be kept in water when they are not used for testing. Once a month they must be boiled in water to make sure all remaining air in the pores is removed.
2. Place the bottom part of the shear box on a leveled underground.
3. Place the bottom part with the groves on top in the bottom part of the shear box. Follow with a porous metal place, then the silver metal plate which has wholes in it. Make sure the groves of both bottom part and silver metal plate are places perpendicular to the shearing direction.
4. Place a paper sheet on top of the silver metal plate. If it doesn't fit, make it fit by using a scissor.
5. Rub some silicone grease on the parts of the bottom and upper part that will touch each other. This will make the metal frictionless, so all the friction measured comes from the upper and lower layer of clay moving past each other.
6. Place the upper part of the shear box on top of the bottom part by making sure the letter shown on both parts are on top of each other. Put the two screws in the wholes and tighten them with either a screwdriver or allen key.
7. Put the prepared specimen on top of the paper sheet, making sure no air is left in the box. Make sure the surface of the clay is straight. Try to fill up the box to just below the two holes in the upper part.
8. Place another paper sheet on top of the specimen. Make sure it fits.
9. Place the other silver metal plate on the specimen, but now with the groves facing down. Then place the other porous metal plate on it. Don't put pressure on the specimen! The top cap also has to be placed on top with the lines perpendicular to the direction of the shear stress.
10. Place the shear box in the carriage making use of the two bigger screws at the side, making sure one tightens the different parts of the bigger box till the shear box is fixed on its place.
11. Check the calibration equipment.
12. Fill the bigger box with normal tap water until the water touches the side of the cap of the shear box. For the pH 9 samples deionised water with KOH should be added. For the acetone samples acetone should be added and the air samples don't have any liquid in the carriage.
13. Balance the lever arm below the shear box by making sure it is horizontal at the same time that the unit above the cap of the shear box only slightly touches the cap of the shear box. It may not apply any load at that point!
14. Put the screw below the arm up til it just touches the arm. This will make sure the load is not applied yet when one places a weight on the arm.
15. Place the chosen weight on the unit below the arm. It is recommended to place the load in multiple steps. Start with 0.25 or 0.50 kg and double the amount of weight one applies with each step.
16. Check the number of the shearbox. In LabView, make sure the button under 'Consolidation' of the chosen shearbox number says 'ON'. A notification in Italian will pop up asking if one wants to keep the previous acquired data. Click 'NO'. Then a notification will pop up asking if one wants to reset the vertical displacement measurement to 0. One has to press 'OK' for quite some time. Then a new notification pops up asking if one wants to start the measurements. Before pressing 'OK', make sure the screw below the arm isn't touching it anymore, then press 'OK'.
17. One can activate the (t,y) graph showing the amount of vertical displacement (y-axis) against the time that has past (x-axis). When the slope of the graph starts to change to a horizontal slope, the consolidation is finished. One now can add the additional weight or stop the measurements to start the shearing phase.

18. When the consolidation is finished, press on the ’ON’ button below ’Consolidation’ so it shows ’OFF’ and the measurement stops. Put the screw back up til it slightly touches the arm. For the shearing phase the screw needs to stay down.

B.4 Shear box - Shearing phase

1. Remove the two screws in the shear box.
2. Check the calibration equipment for the horizontal displacement.
3. Set the desired strain rate. For the pH 4 and acetone samples 0.033 mm/min can be used, for the pH 9 samples 0.005 mm/min. The dry samples have a strain rate of 0.05 mm/min.
4. Click on ’ON’ below Monitor on the screen and check whether ’Box 1 - Loading Cell 500 kg’ and ’Box 2 - Loading Cell 500 kg’ show approximately 0 as value.
5. Make sure the button below ’Shearing’ shows ’ON’ of the chosen shearbox number. A notification will pop up asking if one wants to keep the previous acquired data. Click ’NO’. Then a notification will pop up asking if one wants to reset the horizontal displacement measurements to 0. One has to press ’OK’ for quite some time. Then a new notification pops up asking if one wants to start the measurements. Before pressing ’OK’, make sure the screw below the arm isn’t touching it anymore and press ’RUN’ on the box with the strain rate. Then press ’OK’.
6. One can activate the (x,T) graph showing the amount of horizontal displacement (x-axis) against the shear stress applied (y-axis). When the slope of the graph starts to change to a horizontal slope, the shearing is finished. Make sure to wait till a vertical displacement of around 7 mm is reached before stopping the measurements.
7. Press on the button below ’Shearing’ so it shows ’OFF’. Turn the shearbox of by pressing the ’STOP’ button.
8. Remove all the water in the bigger box before unloading the shear box. This prevents the specimen absorbing water when it is unloaded.
9. Be very careful when getting the specimen out of the shearing box. Place it in a plastic bag and seal it vacuum.
10. Clean all the parts of the shearingbox before starting the next experiment.

B.5 Vacuum packing

1. Retrieve the tested sample from the shearbox. Make sure to be very gentle when removing the upper part, so one won’t harm the sample.
2. Take a small sample of the specimen that can be used for the water content measurement.
3. Keep the paper sheets on the sample and place the sample in a small plastic bag that can be retrieved from the shelve under the vacuum machine.
4. Make sure the place where the seal will come is completely clean and all possible clay is removed.
5. Set the time of the vacuum machine to 15 seconds. Remove the metal rod holding the cap of the vacuum machine up. When one closes the cap the vacuuming will immediately start!
6. Place the plastic bag with the specimen in the vacuum machine. Make sure the end of the plastic bag is tucked under the rod on which the seal will be made.
7. Press the cap of the vacuum machine firmly on the bottom part.
8. When the vacuuming is done make sure the metal rod holding up the cap of the machine is placed back to its original state when leaving the machine.

B.6 Water content measurement

1. Weigh the tray that will be used to place the small sample on and write it down.
2. Place a small sample of the tested specimen on the tray and weigh the tray again. Also write this value down. The saturated weight can now be calculated.

3. Place the tray in the oven and leave it overnight.
4. Take the tray from the oven and weigh the tray with the sample again. Now the dry weight can be calculated.

B.7 Analyzing data from LabView

1. Copy the consolidation data retrieved from LabView from a text document into Excel. If one's Excel works with commas instead of points, make sure to replace all points by commas before copying the data into Excel.
2. Plot the time with a logarithmic scale on the x-axis and the vertical displacement y on the y-axis.
3. Draw manually a line through both inflection points and look at the intersection of both lines.
4. Look at what time the lines intersect. This is the consolidation time for this phase.
5. Repeat steps 1 till 4 for the remaining consolidation phases and find their consolidation time.
6. Add all the consolidation times up, so one gets the total consolidation time. Check this value with the actual consolidation time. If the actual consolidation time is bigger than the calculated time, the results are reliable.
7. Then multiply the total consolidation time by 12 and check whether the actual shearing time is bigger than the calculated shearing time. If the actual shearing time is bigger than the calculated time, the results are reliable.
8. Copy the shearing data retrieved from LabView from a text document into Excel. If one's Excel works with commas instead of points, make sure to replace all points by commas before copying the data into Excel.
9. Add five new columns: the first two should show the negative value of the measured x and y. The third column shows the effective area. The value of x must be subtracted from the original width. So the formula will be: =(0.06-x/1000)*0.06. x is gathered in mm, so it must be converted to metres. The fourth column shows the measured horizontal force. Since the horizontal force has a value at time 0, this value must be subtracted from all T's to get the actual value of T. The fifth column shows the shear stress, which is the horizontal force divided by the effective area. So the formula will be: =T/A.
10. Plot two graphs: the first graph shows the horizontal displacement x against the shear stress in kPa. Where the line becomes completely horizontal the sample has reached its shear strength. The second graph shows the horizontal displacement x against the vertical displacement y. This shows the volumetric behaviour of the sample. A negative slope means the sample is contracted.
11. For each pore fluid and consolidation state, one should make a graph with the shear strength on the y-axis and the vertical stress applied on the x-axis. These graphs can then be compared with each other.

C Planning

This appendix will compare the planning from the proposal report with the actual performance of experiments and discuss why things were changed during the Bachelor Assignment.

C.1 Planning v Reality

The first thing that one did not take into account when making the planning was the performance of preliminary tests. One did not know that these would take place, so one could not have included them in the planning. The second difference between the planning and the reality was that the order of the tests was altered. Instead of starting with the over consolidated air samples, one started with the pH 4 water samples, then the pH 9 water, then acetone and then air. Also, the normally consolidated tests were performed first and then the over consolidated tests. The reason for this deviation was that the most difference in results was expected between the pH 4 and pH 9 samples. The pH 9 samples would only have face-to-face configurations left, which would mean a significant difference in results of the shear strength test could be observed.