Micro-Macro study of the cracks in clays related to desiccation
Xin Wei

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DOCTEUR DE L’ECOLE CENTRALE PARIS
par
Xin WEI
Spécialité : Géotechnique
Laboratoire d’accueil : MSSMat CNRS UMR N°8579

ETUDE MICRO-MACRO DE LA FISSURATION DES ARGILES SOUMISES A LA DESSICCATION

Soutenue le 31 janvier 2014
Devant le jury composé de :

Yujun Cui Rapporteur
Said Taibi Rapporteur
Nadine Bourgeois Examinateur
Odile Ozanam Examinateur
Nadia Saiyouri Examinateur
Hanene Souli Examinateur
Mahdia Hattab Directeur de thèse
Jean – Marie Fleureau Directeur de thèse

Ecole Centrale Paris
Grande Voie des Vignes – 92290 – Châtenay Malabry
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Financial support for this study, which was provided by China Scholarship Council, is gratefully acknowledged.
ABSTRACT

The objective of this research is to analyze the appearance and propagation of cracks related to desiccation and to provide a better understanding of the relation between the macroscopic and microscopic behavior of five clays, a kaolinite, a montmorillonite and three mixtures of kaolinite and montmorillonite. At the macroscopic scale, the method is based on (1) measurements of water content, void ratio and degree of saturation versus suction during drying, which allows to specify the relationship between shrinkage and desaturation and highlights the characteristic phases of behavior; (2) measurements of water contents and global deformations in free desiccation tests in order to study their homogeneity; (3) the determination of the local deformations and displacements during drying using the softwares VIC-2D and VIC-3D; (4) a classical study of the parameters of cracks; (5) traction tests in order to identify the tensile properties of the clays involved in the formation of cracks.

At the microscopic scale, the study is based on a thorough microstructure analysis using in particular scanning electron microscope, coupled to an adapted method for the identification of preferential orientations of particles. This microscopic study is complemented by mercury intrusion porosimetry analysis, a method which allows to quantify the poral space and to characterize the local void ratio.

In addition to the research on clay soils, the effects of decompression and suction on the formation of cracks in a clay rock were analyzed too. The relationship between macroscopic changes and the changes in the microstructure and porosity was investigated.

During free desiccation tests, two-dimensional strains and displacements maps are obtained with Vic-2D. The zones of the sample where cracks appear are identified as well as the evolution of strains and displacements before the appearance of cracks. At the end of desiccation, the cracks form a kind of network. Bifurcation of cracks can be observed in some cases. Two modes of cracks are detected during the tests: traction mode and tearing mode. When a crack is caused by traction, the propagation direction follows the direction perpendicular to maximum extension. If there are shear strains in the vicinity of the crack, then its direction changes. In the early time of desiccation, the displacements and strains on the boundaries are larger than those in the other parts of the model. In the vicinity of cracks, displacements and strains are relatively larger than those in the other parts. In most parts of the model, the principal strains are mainly longitudinal and transversal.

For a given material, tensile strength increases when water content decreases. Plotting the results versus the liquidity index allows highlighting the effect of mineralogy on tensile strength. At the same liquidity index, the maximum tensile stress decreases when the montmorillonite content increases. The effect of suction on
tensile strength seems qualitatively similar to what is observed in the case of unconfined compression strength with tensile strength being a linear function of the logarithm of suction.

Analysis with SEM of four slurries submitted to several suctions highlights the global isotropy of the microfabric, with a random orientation of the particles, while a finer analysis reveals that the fabric may present locally some anisotropy.

**Key words**: clays, clayey stone, digital image correlation, free desiccation, shrinkage, suction, microstructure, isotropy, scanning electron microscope (SEM), mercury intrusion porosimetry (MIP)
L'objectif de cette recherche est d'analyser l’apparition et la propagation des fissures de dessiccation et de mieux comprendre la relation entre les comportements macroscopique et microscopique de cinq argiles, une kaolinite, une montmorillonite et trois mélanges de kaolinite et montmorillonite. A l’échelle macroscopique, la méthode est basée sur (1) la mesure de la teneur en eau, de l’indice des vides et du degré de saturation en fonction de la succion au cours du séchage, qui permet de préciser la relation entre le retrait et la désaturation et met en évidence les phases caractéristiques du comportement; (2) des mesures de teneur en eau et des déformations globales dans les tests de dessiccation libre afin d'étudier leur homogénéité ; (3) la détermination des déformations et des déplacements locaux pendant le séchage avec les logiciels VIC-2D et VIC-3D ; (4) une étude classique des paramètres des fissures ; (5) des essais de traction pour déterminer les propriétés des argiles impliquées dans la formation de fissures.

A l'échelle microscopique, l'étude est basée sur une analyse approfondie de la microstructure en utilisant en particulier le microscope électronique à balayage, couplé à une méthode adaptée pour l'identification des orientations préférentielles des particules. Cette étude microscopique est complétée par l'analyse de porosimétrie au mercure, une méthode qui permet de quantifier l'espace poral et de caractériser l’indice des vides local.

Outre les recherches sur les sols argileux, les effets de la décompression et de la succion sur la formation des fissures dans une roche argileuse ont été analysés également. La relation entre les changements macroscopiques, les changements dans la microstructure et de la porosité a été étudiée.

Pour un matériau donné, la résistance à la traction augmente lorsque la teneur en eau diminue. Le tracé des résultats en fonction de l’indice de liquidité permet de mettre en évidence l’effet de la minéralogie sur la résistance à la traction. Au même indice de liquidité, la contrainte maximale de traction diminue lorsque le pourcentage de monmorillonite augmente. L’effet de la succion sur la résistance à la traction semble qualitativement similaire à ce qui est observé dans le cas de la résistance à la compression simple : on observe que la résistance à la traction est une fonction linéaire du logarithme de la succion.

L’analyse au MEB de quatre suspensions soumises à plusieurs succions met en évidence l’isotropie globale de la microstructure, avec une orientation aléatoire des particules, tandis qu’une analyse plus fine révèle que la microstructure peut présenter localement une certaine anisotropie.

**Mots clés:** argile, argilite, corrélation d’images numériques, dessiccation libre, retrait, succion, microstructure, isotropie, microscopie électronique à balayage (MEB), porosimétrie au mercure (PAM)
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<tr>
<td>c'</td>
<td>Effective cohesion intercept</td>
</tr>
<tr>
<td>Cc/Cs</td>
<td>Compression index and swelling index</td>
</tr>
<tr>
<td>d</td>
<td>Equidistance between the crystallographic planes;</td>
</tr>
<tr>
<td>d_{mean}, d_{inter}</td>
<td>Mean pore diameter; intermediate pore diameter</td>
</tr>
<tr>
<td>e</td>
<td>Void ratio</td>
</tr>
<tr>
<td>e'</td>
<td>Global void ratio</td>
</tr>
<tr>
<td>e_{SL}</td>
<td>Shrinkage limit void ratio</td>
</tr>
<tr>
<td>E</td>
<td>Elastic modulus</td>
</tr>
<tr>
<td>ε</td>
<td>Strain of the sample</td>
</tr>
<tr>
<td>ε_v</td>
<td>Volumetric deformations</td>
</tr>
<tr>
<td>ε_{xx}</td>
<td>Strain in the X-direction</td>
</tr>
<tr>
<td>ε_{yy}</td>
<td>Strain in the Y-direction</td>
</tr>
<tr>
<td>ε_{xy}</td>
<td>Shear strain</td>
</tr>
<tr>
<td>ε_1</td>
<td>Maximum principal strain</td>
</tr>
<tr>
<td>ε_2</td>
<td>Minimum principal strain</td>
</tr>
<tr>
<td>F</td>
<td>Load measured during the traction tests</td>
</tr>
<tr>
<td>G</td>
<td>Elastic energy release rate per crack tip</td>
</tr>
<tr>
<td>G_s, ρ_s</td>
<td>Grain density</td>
</tr>
<tr>
<td>K</td>
<td>Stress intensity factor</td>
</tr>
<tr>
<td>I_p</td>
<td>Plasticity index</td>
</tr>
<tr>
<td>Δl</td>
<td>Displacement during the traction test</td>
</tr>
<tr>
<td>p</td>
<td>Order of reflection (XRD);</td>
</tr>
<tr>
<td>p'</td>
<td>Mean effective stress</td>
</tr>
<tr>
<td>Q_o</td>
<td>Compressive strength</td>
</tr>
<tr>
<td>R</td>
<td>Crack resistance</td>
</tr>
<tr>
<td>Ra, Rp</td>
<td>Profile roughness parameters</td>
</tr>
<tr>
<td>σ_t</td>
<td>Minor total stress at tensile failure, tensile strength</td>
</tr>
<tr>
<td>σ_n</td>
<td>Effective normal stress</td>
</tr>
<tr>
<td>σ'<em>{1}, σ'</em>{3}</td>
<td>Effective principal stresses</td>
</tr>
<tr>
<td>σ'_{v}</td>
<td>Vertical effective stress (oedometer, 1-D consolidation)</td>
</tr>
<tr>
<td>σ_{H_g}</td>
<td>Surface tension of mercury</td>
</tr>
<tr>
<td>Θ'</td>
<td>Effective friction angle</td>
</tr>
<tr>
<td>θ_{nw}</td>
<td>The contact angle between mercury and the pore wall</td>
</tr>
<tr>
<td>S_r</td>
<td>Liquid degree of saturation</td>
</tr>
<tr>
<td>s</td>
<td>Matrix suction</td>
</tr>
<tr>
<td>S</td>
<td>Total pore surface area</td>
</tr>
<tr>
<td>S_{0}</td>
<td>Surface of section of the tensile test</td>
</tr>
<tr>
<td>Sa, Sp</td>
<td>Area roughness parameters</td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
</tr>
<tr>
<td>--------</td>
<td>-------------</td>
</tr>
<tr>
<td>( \tau_f )</td>
<td>Shear strength</td>
</tr>
<tr>
<td>( T )</td>
<td>Transmittance</td>
</tr>
<tr>
<td>( w )</td>
<td>Gravimetric water content</td>
</tr>
<tr>
<td>( w_L )</td>
<td>Liquid limit</td>
</tr>
<tr>
<td>( w_p )</td>
<td>Plastic limit</td>
</tr>
<tr>
<td>( w_{SL} )</td>
<td>Shrinkage limit water content</td>
</tr>
<tr>
<td>( u_a )</td>
<td>Pore air pressure</td>
</tr>
<tr>
<td>( u_w )</td>
<td>Pore water pressure</td>
</tr>
<tr>
<td>( U )</td>
<td>Elastic energy release</td>
</tr>
<tr>
<td>( \nu )</td>
<td>Poisson’s ratio</td>
</tr>
<tr>
<td>( \lambda )</td>
<td>Wavelength of the radiation used;</td>
</tr>
<tr>
<td>( \tilde{\nu} )</td>
<td>Wavenumbers</td>
</tr>
<tr>
<td>( U )</td>
<td>Displacement along the X-axis</td>
</tr>
<tr>
<td>( V )</td>
<td>Displacement along the Y-axis</td>
</tr>
<tr>
<td>( W )</td>
<td>Energy required for crack growth</td>
</tr>
<tr>
<td>( Z )</td>
<td>Displacement along the Z-axis</td>
</tr>
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GENERAL INTRODUCTION

BACKGROUND OF THE STUDY

It is common knowledge that clayey soils can crack during desiccation. Cracks occur when soils are restrained while undergoing volume change produced as a result of the soil suction generated within the desiccating soil matrix. The desiccation cracking of clay soils can have a severe impact on the performance of clayey soils in various geotechnical, agricultural and environmental applications. In many circumstances it is the cause of damages in earthen and soil supported structures. For example, desiccation cracking has the potential to render a low conductivity barrier constructed of clayey soil virtually ineffective. The desiccation of soils and its most visible consequence, cracking, are universally observed in soils, as shown for instance in figure 1.1.

![Desiccation crack pattern in dry mud, Anhui Province of China](image1.jpg)

Figure 1.1 Desiccation crack pattern in dry mud, Anhui Province of China

Beyond such observational aspects, the general process of desiccation cracking in soils, as well as in other kinds of materials, is of real importance to the engineer. When it affects the soil on which infrastructures are founded, or the soil that constitutes man-made earth structures themselves, the process may have major consequences. We should also stress in this introduction that desiccation intervenes in many activities and stages in the manufacturing of products (e.g. food, wood, powders, ceramics, polymers, etc.) and is a subject studied in the associated fields of engineering.
Despite this significance, the essential mechanisms of desiccation cracking are not well understood, and consequently, predictive tools are inadequately developed. The mechanisms of drying shrinkage and associated cracking in soils, and the ways to control or avoid such cracking, are still elusive.

It is noteworthy that the classical theories of soil mechanics (initially developed for saturated soils) of effective stress, stresses and strains, shear strength, earth pressure, consolidation, and their application to settlement, bearing capacity, and stability problems imperfectly address the occurrence and the consequences of desiccation cracking. Even with the more recent development of unsaturated soil mechanics, which, by definition address drying and shrinkage, little was done to understand the effect of microstructural desiccation cracks, for example, local deformations and displacements etc.

In geotechnical engineering practice, desiccation cracks are widely reported in soil, especially in clayey soils. The general understanding is that cracks are the consequence of the drying shrinkage process. Historically, shrinkage itself was related to a change in effective stress. It is also recognized that desiccation cracks are mainly tensile cracks, and that the soil, as it gains strength during desiccation, should provide increased resistance to crack formation (Mitchell and Soga 2005).

However, most of the time, the studies on the subject are still reduced to behavioral and qualitative descriptions of shrinkage characteristics and desiccation cracking potential of soils based on experimental results. More quantitative works are scarce; they stem from a classical approach, addressing desiccation either as an elastic deformation problem or a plastic equilibrium problem. The problem is often considered in simple situations, such as in two-dimensional geometry.

Only very recently studies have been undertaken with a more rational approach, taking into account the effective processes as a whole (or trying to do so). However, in 1993, Abu-Hejleh stated that “the constitutive relationships during desiccation of soft soils (were) not described in the literature”. To our knowledge, this statement remains valid today.

It is believed that a robust prediction of the occurrence of desiccation cracks could only really be achieved through a global comprehension of the processes, a unified conception, and a rational modeling approach. There are several researchers who have established different constitutive models for soil subjected to desiccation, in order to predict the beginning and development of cracks (Lau 1987; Péron 2008).

This study includes an experimental and phenomenological study of desiccation, characterizing the drying shrinkage and the cracking of soils, trying to offer a better understanding of the mechanisms of appearance and evolution of the cracks related to desiccation in clay material. The approach combines a classical study of cracks and the determination of the local deformation and displacement fields during drying, taking into account the real physical phenomena occurring at the microscopic scale in clayey media submitted to desiccation (suction effect, temperature etc.).
OBJECTIVES AND ORGANIZATION OF THE THESIS

The aim of this study is to try to understand how, in clayey materials submitted at macroscopic scale to a given drying path, cracks and microcracks appear, and how the phenomenon evolves.

The investigation consists in conducting a literature review, a laboratory experimental program and an analytical program. The principal objectives of the investigation are:

a) To study the cracking patterns of desiccating soil and to identify the parameters that influence the cracking pattern;

b) To study the effect of soil suction on the desiccation cracking in soils;

c) To provide a better understanding of the mechanisms of appearance and evolution of the cracks related to desiccation in clay material.

d) To associate to the global approach, a local approach, which is focused on an experimental characterization of the strains.

The approach that is proposed is based on experimental investigations and consists in following the microstructural evolution in the material, especially the orientation of clayey particles, as a function of its hydric state. The study is based on a thorough microstructure analysis using in particular scanning electron microscope, coupled to an adapted method for the identification of preferential orientations of particles. This microscopic study will be complemented by mercury intrusion porosimetry analysis, a method which allows to quantify the poral space and to characterize the local void ratio.

And the approach also combines a classical study of cracks and the determination of the local deformations and displacements during drying. For this purpose, photos of the soil surface are taken at regular intervals and analyzed by correlation of digital images using the software CORRELIT3Q and VIC2D and VIC3D, which identifies the cracks at an early stage.

A more detailed organizational list of the thesis is as follows:

A general introduction to the desiccation cracking in soils is provided.

Chapter 1 contains a summary of the existing state-of-the-art on the desiccation cracking of soils, including macroscopic studies and microscopic studies.

Chapter 2 presents the clayey materials and experimental methods which are used in this study.

Results of macroscopic experiments are provided in Chapter 3, which includes drying/wetting paths of the materials, macroscopic experiments of desiccation, digital image correlations of displacements and strains, analysis of cracks and traction tests.

Chapter 4 proposes the results of the microscopic experiments. Two kinds of tests are presented: Scanning electron microscopy and mercury intrusion porosimetry analyses.
Macroscopic and microscopic results of a clayey rock in Bure are presented in Chapter 5. Conclusions derived from this study and recommendations on future research are given.
CHAPTER 1 LITERATURE REVIEW

Research literature was reviewed from the geology, soil science and engineering disciplines concerning the problem of shrinkage and desiccation cracking in soils. Most of the information provided by geologists deal with the physical observation of the aerial patterns of desiccation cracks. The information provided by the soil scientists concerns the physical characterization of soil shrinkage and cracks. There is also a large amount of information derived from the engineering field which considers the mechanical characteristics of soils. Cracking has received considerable attention in the literature of soil science and agriculture (for example, Frydman, 1967; Ritchie and Adams, 1974; Blake and Jones, 1973; Fredlund and Morgenstern, 1977; Chen et al., 1980; Ravina, 1984; Bagge, 1985; Ringesten, 1988; Péron et al., 2007). It appears that, as yet, the problem of shrinkage and desiccation cracking of soils has not been completely assessed or addressed. The treatment of cracks in soils remains largely behavioral and qualitative; the literature review given in this chapter provides some insight into the behavior of desiccation cracking in soils.

1.1 MACROSCOPIC STUDIES OF CRACKS

1.1.1 Definition of cracks in a clayey material

When a horizontal layer of soft, initially saturated soil begins to dry, decrease in water content largely results in reorganization of the soil particles into successively closer arrangements. This involves shear straining, since the loss of water is largely one-dimensional towards the drying surface, where it is removed by evaporation. Experiments have shown (Croney and Coleman, 1953) that the void spaces between the particles do not remain indefinitely filled with water, and successively larger proportions of air gradually enter the void spaces. Surface-tension effects at air-water-soil interfaces inside the soil generate negative pressures (or matrix suctions) below atmospheric pressure in the remaining pore water. These matrix suctions (measured by the difference (u_a - u_w) between the pore-air pressure and the pore-water pressure) produce two counteracting effects. First, at a selected point, the soil tends to contract more or less isotropically, assuming at this stage that the pore water remains interconnected and the pore air is discontinuous. This shrinkage produces vertical cracks below horizontal drying surfaces. Then, the soil gains strength and provides increased resistance to crack formation. Suctions can also arise from osmotic effects related to soil chemistry. However, matrix suction dominates below the immediate surface of a soil deposit, and most workers assume that it drives shrinkage and cracking processes during drying. Most of the subsequent discussion will relate to the effects of matrix suction.
Cracks affect soil compressibility, its time rate of consolidation its strength and the rate at which water can reenter. Thus, many geotechnical constructions are affected directly or indirectly by the presence of cracks in a soil mass. A soil with cracks is more compressible than an intact version of the same soil at the same water content. However, wetting and drying cycles lead to increasing overconsolidation towards the surface, and this reduces the compressibility. The transition with depth from overconsolidated states close to the surface to normally consolidated states below the water table is relatively smooth.

1.1.1.1 The patterns of cracks

It is commonly considered that desiccating clayey soils crack when the tensile stress developed in the soil due to the matrix suction exceeds the tensile strength of the soil (Kodikara et al., 2000). Tensile stresses develop only when the soil is restrained in some way against shrinkage. The restraints can be external (e.g. rough layer interfaces) or internal (e.g. sections of soil undergoing non-uniform drying, heterogeneity in water content or density, etc.).

Observed 3D fields cracking patterns can be divided into two broad categories, namely orthogonal patterns and non-orthogonal patterns. In orthogonal patterns, cracks tend to meet at right angles. In the evolution of these patterns, cracks usually occur sequentially. First, primary cracking develops dividing the clay surface into blocks and subsequent drying tends to further sub-divide these blocks. In non-orthogonal cracking patterns, the cracks do not meet at right angles. Hexagonal patterns (cracks meeting at angles of 120°) and their deviations fall into this category. The non-orthogonal cracks appear to originate simultaneously and connect up to form a blocky pattern. During subsequent drying, however, (secondary and tertiary) cracking and opening of the cracks can occur over larger blocks encompassing a number of smaller blocks. Theoretically, this secondary and tertiary cracking behavior can be considered to be a bifurcation from the primary cracking pattern (Bazant and Cedolin, 1991; Kodikara et al., 1999). As postulated by Bazant and Cedolin, cracks appear to originate simultaneously and connect up to form a blocky pattern. The parallel cracks shown in Figure 1.1 can be considered as two-dimensional whereas other crack patterns are essentially three-dimensional.
1.1.1.2 Observation of cracks in-situ and in the laboratory

**In-situ observations**

Cracked clays are widely met all over the world, including many countries in Asia, Europe and Africa etc. For example, in the south-west of China, there are large scale distributions of cracked clays.

*Kong (1994)* tried to find the geological characters and engineering geological properties of fissured clay from 5 different Provinces of China (Fig. 1.2).
After in-situ investigations of these cracks, Kong also discussed the origin of fissures in the clay mass. At last, the effects of these cracks on the mechanical properties of clay mass were discussed.

Desiccation cracks have also been observed in salt lakes (Fig. 1.3, Longwell, 1928), in clayey till deposits after heavy rain (Fig. 1.4), in varved sequence of lacustrine clays and in dried-out hydraulic fills of intermixed and inter layered silty fine sand and moderately to highly plastic clayey silt (Fig. 1.5). Desiccation cracks are characterized by their aerial patterns, spacings and depths of the cracks.
Studies of the effect of desiccation on the rehabilitation of tailing ponds at open-pit coal mines in Queensland, Australia were conducted by Morris et al., 1992 (Fig. 1.6). They began by reviewing the factors that influence the occurrence of cracked soils in Australia and Canada and the morphology of crack development. Subsequent sections develop calculations
of cracking in unsaturated soils using (a) elastic theory, (b) linear elastic fracture mechanics, and (c) a rupture analysis based on the transition between tensile and shear failure.

![Cracking in coal mine tailings](image)

Figure 1.6 Cracking in coal mine tailings, New Hope Colliery, Bundamba District, West Moreton Coalfields, Queensland, Australia.

In the following section, available laboratory experiments on the desiccation of soil layers are discussed. The data include those reported by Corte and Higashi (1960), Lau (1987), Péron (2008), Tang et al. (2010). The process of desiccation is strongly dependent on the local climatic conditions. The climatic conditions include temperature, relative humidity (RH), wind velocity and solar radiation. In these laboratory experiments, the temperature and RH are controlled.

**Observations in laboratory**

i. Corte and Higashi (1960)

Corte and Higashi (1960) carried out these experiments as part of an early research on patterned ground by the US Corps of Engineers. These tests appear to be the most comprehensive series of laboratory tests undertaken on the desiccation of soil layers, but seemed to have not been published except in their original report. It is noted that about 60 tests have been conducted spanning a period of one year. The test parameters were divided into two categories, namely extrinsic and intrinsic. The extrinsic parameters controlled were
temperature, relative humidity, thickness of the soil layer and the base material. The relevant intrinsic test parameters were initial moisture content and density or initial state of the soil.

Most of the experiments were carried out in flat wooden containers of 600 mm×840 mm plane area and 70 mm depth. The soil was prepared in two initial states: (a) as a slurry with initial water content of 60% and dry density of 1800 kg/m$^3$; and (b) as a loosely compacted soil with initial water content of about 45% and a dry density of about 1500 kg/m$^3$. Various materials were used at the base of the containers in order to provide differing base adhesion characteristics. The base materials used included plain wood, greased wood, and sheet of glass. A 20 mm thick base layer of sand in the container having a wood base was also tested. For the majority of the tests, the room temperature was kept at about 22°C and the relative humidity was in the range of 30 to 40%.

ii. Lau (1987)

These tests have been carried out in the soil laboratory at the University of Saskatchewan Canada. Two types of local soils were used in the tests. The cracking tests were carried out in a flat wooden container of 610 mm×610 mm plane area and 76 mm deep. The soils were prepared so that their initial moisture contents were close to their liquid limits and the soils were close to a slurry state at the beginning of the tests. An additional aspect of these tests was that the soil was instrumented with four embedded ceramic-cup tensiometers to measure the soil suction during the tests. Furthermore, the vertical deformation of the top soil surface was also measured using dial gauges that were mounted at the four quadrants of the container. After the cracking began, the soil moisture contents during the tests were measured by taking small samples of soil. No special attention was paid to the base condition of the wooden container. Photographs were taken during the tests to record the development of the crack pattern (Fig. 1.7).

Unfortunately, in most of these tests, the initiation of cracking was influenced by the presence of the instruments in the soil.

![Figure 1.7 The layout of equipment for the cracking test (Lau, 1987)](image_url)

The research of Tang presents the results from laboratory experiments (Fig. 1.8) conducted on Romainville clay from a slurry state to investigate the shrinkage and desiccation cracking behavior at different temperatures. Image processing technique was applied to characterize the crack pattern. Surface crack ratio (RSC), which is the ratio of the surface cracks area to the total surface area of a specimen, was determined at different water contents to quantify the cracking extent on the soil surface. Some critical water contents during drying were identified. In addition, the relationship between cracking behavior and shrinkage properties were discussed.

![Figure 1.8 Schematic drawing of set-up (Tang, 2010)](image)


Péron did two different kinds of desiccation test of soil: free desiccation test and constrained desiccation test.

Free desiccation tests were conducted, avoiding as much as possible any mechanical constraints at the boundaries due to cohesive-frictional effects during shrinkage. The drying was undertaken on a Teflon support treated with a hydrophobic substance, containing silicon and ethanol. A device (Fig. 1.9) was designed to obtain regular and reproducible bar-shaped samples. The initial slurry was poured in a 295 mm × 49 mm, by 12 mm high, aluminum mould, lying on the Teflon plate.
Constrained desiccation tests were all performed on a support creating an axial restraint. After about 17 hours drying time on this support (scheme in fig. 1.10), a series of 6 to 8 cracks (generally 7) appeared always in the direction parallel to the notches.

![Sliding mould and Teflon support](image)

Figure 1.9 Set up for bar fabrication (Péron, 2008)

![Diffuse cracking at cake tip](image)

Figure 1.10 Detail of the bar extremity showing slight cracking between the support and the rest of the bar (Péron, 2008)

1.1.1.3 The parameters which influence the formation and evolution of cracks

The process of desiccation is strongly dependent on different factors, either external (e.g. rough layer interfaces, climatic conditions etc.) or internal (e.g. sections of soil undergoing non-uniform dying).

Most of the earlier works on the shrinkage and cracking of soils were performed by
geologists and soil scientists. Their research was mainly focused on the aerial patterns and on the spacing of desiccation cracks. Kindle (1917) conducted small scale laboratory experiments using a 150 mm diameter porcelain vessel. The experiments were performed on a slurried Ontario lake clay. He concluded that temperature and tenacity of material are two primary factors in controlling the spacing of mud cracks. Rapid desiccation was found to produce widely spaced cracks and the addition of marly material or sand gave polygons which were much smaller than those formed in clay mud. In the case of a sandy mud, a sufficient amount of sand entirely prevented the formation of mud cracks.

Longwell (1928) studied the mud cracks in Nevada and questioned whether Kindle's conclusion on the effect of desiccation rate was valid in the field. He suggested that there were other factors governing crack spacing. In 1950, Twenhofel pointed out that crack spacing depended on the character of the mud, the rate of drying, the thickness of the mud, the character of the water in which the mud was deposited, the nature of the underlying material, and the presence of foreign matters. He did not present any quantitative relationships between crack spacing and these physical factors.

Washburn (1956) studied the origin of different patterned ground and suggested that non-sorted polygonal patterns in temperate regions are due to the contraction resulting from desiccation or drying. He pointed out that once a fissure pattern is formed it tends to persist or reform in the same places after later wet/dry cycles. The fissures keep pace with the reduction of the land surface and lowering of the water table.

Further experimental work on desiccation cracks in soils was performed by Corte and Higashi (1960). They concluded that crack patterns depend largely on the thickness of soil layer and the bottom material of the container. Widely spaced cracks and large polygons were formed in "thick" layers of mud, and closely spaced cracks and mud curls in thin-bedded muds. The maximum thickness of soils used was about 46 mm. Corte and Higashi (1960) criticized Kindle's conclusions saying that the results were incorrect due to the small vessel used in his experiments.

Lachenbruch (1962) studied the ice-wedge polygons in permafrost and theorized that crack spacing of any polygonal patterned ground is a function of the frequency of flaws (or in-homogeneities) in the ground. The in-homogeneities include non-uniform strength within the soil mass and the non-uniform drying conditions or thermal conditions in case of frozen patterned ground caused by local variation in thickness and shrinkage properties. He further suggested that flaws with an average separation of several polygon diameters have little effect on either uniformity or magnitude of crack spacing. Those flaws with an average separation of the order of a polygon diameter primarily affect the uniformity of spacing, and those whose separation is an order of magnitude less affect the size of crack spacing.

Lachenbruch (1961) also pointed out that shallow, closely spaced, cracks relieve surface tension developed by rapid desiccation after a rain. Deeper cracks, more widely spaced, might represent seasonal desiccation. Profound cracks with spacing of the order of 3 m to 30 m
could correspond to the draining of a marsh or perhaps to a regional climatic trend toward aridity (Willden and Mabey, 1961).

Discussions in geological papers consist mostly of physical observations and the authors do not investigate measurable parameters which control soil cracking. A model of soil cracking for use in engineering application requires measurable parameters and therefore, papers from the geological literature will not be discussed in further details.

Croney and Coleman (1953) and Aitchison and Woodburn (1969), among others, showed that the volume and water content of most soils decrease as soil suction increases. Conversely, the soil volume and water content increase as soil suction is reduced.

Means and Parcher (1963) suggested that the shrinkage of clays is caused by tension in the pore-water, through the action of menisci, reacting against the soil grains.

Blight and Williams (1971) applied the theories of shear strength and Griffith failure criterion to explain the formation of fissures in South African soils. Pore-water tension was incorporated in the estimation of the shrinkage stress. However, they considered cracking in terms of a stress criterion.

Papers presented to the symposium on "The Influence of Vegetation on Clay" (Driscoll, 1984, Ravina, 1984 and Wakeling, 1984) suggested that soil suction should be used as a control variable to describe the tendency of a clay soil to shrink or swell.

Picornell (1985) and Picornell and Lytton (1987) used soil suction as the parameter to estimate the shrinkage and crack depth for the design of vertical moisture barriers in Texas.

The factors that could influence the shrinkage and cracks of the soils are summarized by Cauley and Kennendy (1972) as follows:

a) Crack ratio (area of cracks divided by total area) increases with the percentage of clay particles and depends on the type of clay.

b) Large aggregates (i.e., greater than 25 mm) increase crack ratio.

c) Crack spacing and width are dependent upon tensile strength, tensile modulus of elasticity and coefficient of friction between base and subgrade of pavement. Higher cement contents will result in greater shrinkage and greater tensile strength but wider crack spacing. Higher coefficients of friction can decrease crack ratio but this parameter is not as important as cement content.

d) Rate of evaporation is very important with high rate yielding large shrinkage stresses and substantial cracking.

e) Total amount of shrinkage is appreciably higher at compaction water contents larger than optimum.

1.1.2 Water retention characteristics of soils during desiccation

The desiccation cracks developed in structures are governing factors for their hydraulic
properties; water conductivity is drastically increased by cracking, leading to dysfunction of the barrier systems. Albrecht and Benson (2001) found that the hydraulic conductivity of some clay liner materials caused by desiccation cracks was almost 500 times that of intact soil. The experiments of Boynton and Daniel (1985) and Rayhani et al. (2007) showed that soil cracking increases the hydraulic conductivity of several orders of magnitude. In addition, cracks induced by shrinkage also create weak zones in a soil body with reduced overall mechanical strength and bearing capacity, and increased compressibility. Desiccation cracks were also observed in natural expansive soil slopes and vertical cuttings, considerably affecting their stability (Bagge, 1985; Silvestri et al., 1992). In the case of earth dams, the presence of cracks may also turn into piping leaks, leading to dam failures as for the Stockton and Wister dams (Sherard, 1973). In the agricultural field, as desiccation cracks control the rate and velocity at which water, solutes and micro-organisms are transported in the soil, they can significantly affect the crops growth and production (Bronswijk et al., 1995; Kelly and Pomes, 1998).

To common knowledge, the water content or water loss is the key parameter that controls the cracks initiation and propagation. On the other hand, for a given soil, the water loss or evaporation rate is strongly dependent on temperature and relative humidity.

1.1.2.1 Drying of soils with relationship of water content and suction

Soils shrink in response to a change in the stress state. The stress state depends on various environmental factors such as land use and annual climatic conditions.

The water retention behavior deserves deeper investigation regarding the involved mechanisms of air penetration and shrinkage cessation. In the thesis of Péron (2008), water retention curves of the studied soils are shown in figure 1.11. Essentially, drying shrinkage of such remoulded, initially saturated, non-consolidated samples includes two main stages. In domain 1, of drying shrinkage ($S_r = 1$ and $s = 0$ kPa), deformations can be seen as elasto-plastic (mostly irrecoverable). Within such domain, suction change is equivalent to Terzaghi effective stress change. Deformations occurring during the first drying follow then the NCL line (the normal consolidation line). In Domain 2 of drying shrinkage (decreasing $S_r$ and $s$), deformations are much smaller, and are assumed to be reversible.
Haines (1923) studied blocks of soil molded from a paste and distinguished three main phases of soil shrinkage that accompanied water withdrawal: normal, residual and no shrinkage as shown in figure 1.12. Normal shrinkage occurs when the change in soil volume equals the water loss. Residual shrinkage occurs when air enters the soil and the reduction in soil volume is less than the volume of water lost. At the no shrinkage phase, soil does not shrink upon further drying.
Stirk (1954) defined a fourth phase of shrinkage termed structural shrinkage (Fig. 1.13). It has similar characteristics to residual shrinkage (i.e., water loss larger than volume change) but occurs at the wet end of the moisture range and is associated with the removal of water from coarse pores.

Large volume changes due to shrinkage (i.e., up to 34% of original soil volume) in compacted clayey soils have been reported by various researchers (Stirk (1954) and Chang and Warkentin (1968)). The actual amount of shrinkage due to drying depends on factors such as type and amount of clay minerals, soil fabric arrangements, initial water content and confining pressure (Mitchell, 1976).
The amount of shrinkage in soil increases with the plasticity of the soils (i.e., the more plastic the soils, the more the potential shrinkage). It would be expected that, for an equal amount of clay minerals in a soil, montmorillonites undergo a greater volume change on drying and wetting than kaolinites do. If two samples of a given clay are at the same initial water content, but have different soil fabrics, the one with the dispersed structure shrinks most. Lambe (1958) reported that "clay with oriented particles shrinks the most in a direction perpendicular to the plates and the least in a direction parallel to the plates and a clay with randomly oriented particles shrinks equally in all directions."

Several investigators were concerned with other parameters which affect shrinkage of clay. Gokhale and Anandakrishnan (1970) mixed clay (i.e., either kaolinite or montmorillonite) with sand and noted that the addition of sand generally reduces shrinkage. Kleppe and Olson (1985) conducted shrinkage tests on compacted clay-sand mixtures and concluded that the addition of sand to clayey soil reduces the amount of drying shrinkage. They further concluded that shrinkage strains were essentially linear functions of compaction water content and did not depend on dry density for the range of compaction effort and water content used in their investigation. Sridharan and Rao (1971) found that the void ratio of a kaolinite mixed with organic fluid (i.e., carbon tetrachloride) was higher than that of the soil mixed with clear water. It was suggested that shrinkage was dependent on the dielectric constant of the pore fluid.

A normally consolidated drying path was obtained by Taibi (1994) starting from a very liquid state and then following a drying path. Comparison was made between drying and oedometric or isotropic NC paths. Figures 1.14 shows that for a clay-kaolin P300, where the void ratio is plotted versus the negative pressure (drying) or mean effective stress (isotropic or oedometric tests), the paths are nearly superimposed; in the latter case, the calculations were made taking $K_0 = 0.5$.

![Figure 1.14 Comparison between the oedometric and isotropic compression paths (depending on $p' = p-u_w$) in saturated kaolin P300 (Taibi, 1994)](image-url)
Fleureau et al. (1993) did experiments with 11 different clayey materials to determine the main characteristics of the drying and wetting paths and the influence of initial state and other factors.

Figure 1.15 represents the first drying-wetting cycle on a slurry of kaolinite, with an initial water content \( w_i = 1.5w_L \). Figure 1.15(B), (D) and (E) show the changes in void ratio, degree of saturation, and water content, respectively, with the negative pore-water pressure or the pF.

Figure 1.15 Synthesis of drying-wetting paths on the white clay (Fleureau et al., 1993)
1.1.3 Cracking mechanisms

1.1.3.1 Tensile strength of soils

Soils, in general, are weak in tension. In the analysis and design of earth structures, it is usually reasonable to neglect the tensile strength of soils. However, knowledge of the stress-strain relationships of the soils in tension is of importance for understanding cracking in soils. A limited amount of research has been conducted on the tensile properties of soils. The findings obtained from these studies are summarized by Krishnayya et al (1974) as follows:

a) Soils have a low tensile strength ranging from zero to a few kPa;
b) Soils of high plasticity are, in general, more flexible (i.e., they can undergo a higher tensile strain) than the soils of low plasticity.
c) In the case of compacted soils, an increase in the molding water content from 2% to 3% dry of Proctor optimum to nearly optimum substantially increases the flexibility of soil.
d) The rate of strain has a considerable influence on the tensile characteristics of soils.

It seems reasonable to assume that soil cracking is a result of the application of tensile stresses. Leonards and Narain (1963) presented data from beam flexion tests and found the tensile strains-at-failure (at rupture or cracking) for compacted clay samples ranged from 0.05% to 0.33%. Krishnayya et al. (1974) defined failure as the maximum tensile stress in the indirect (Brazilian) test. It was found that the maximum tensile stress for a compacted low plastic till was about 3.5 kPa with tensile strain-at-failure of 0.2% to 3.0%. Ajaz and Parry (1975) performed both beam flexion and direct tension tests on two clays and found that tensile strain at failure (defined at maximum tensile stresses.) was from 2.0% to 15.0% for flexion tests and from 1.0 to 5.0% for direct tension tests. In general, all the above tests showed that tensile strains-at-failure increased with an increase in water content. Other conclusions on tensile strains-at-failure are impossible to draw as values of strain appear to vary with test type, loading rates and definition of failure.

Fang and Chen (1972) showed that tensile strength increases but that the unconfined compressive strength to tensile strength ratio decreases as plasticity index increases (Figs. 1.16 and 1.17). They found that the range of unconfined compressive strength to tensile strength ratio for compacted silty clay varied from 6.0 to 13.0.
Bishop and Garga (1969) conducted drained tension tests on London clay and found that the tensile stress at failure lied in the range 26.2 kPa to 33.1 kPa for intact samples and unconfined compressive strength to tensile strength ratio ranged from 5.6 to 6.9. They also found that the tensile strength for remolded London clay was small (close to zero). They concluded that remolding almost completely destroyed cementation or other bonds that were capable of withstanding tensile stresses.

Avila (2004) presented in his thesis about the direct measurement of tensile strength of clays at different scales, with double triangular-shaped specimens (Fig. 1.18). The mold is split at the narrowed portion where displacements can be measured.
Figure 1.19 shows the stress-strain curves of tensile tests on 9 samples prepared at different initial suctions. The stress-strain curves for initial water contents between 40.9% and 44.8% (tests a to f) are presented in figure 1.20.

Figure 1.19 Stress-strain curves of traction with different initial suctions (controlled load tensile tests) (Avila 2004)

Figure 1.20 Stress-strain curves of the tests (f) with controlled tensile deformations (Avila 2004)

1.1.3.2 Failure criterion of soils under tension

If the stresses or strains in a soil could be estimated, then prediction of cracking would then depend on the choice of a failure criterion. In cohesive soils, failure situation can be defined either at the beginning of loss of shearing resistance or at a relatively advanced state in the loss of shearing resistance (Newmark, 1960). The best known and most widely used failure criterion is the Mohr-Coulomb theory as illustrated in fig. 1.21. The shear strength of a soil at a point on a particular plane in term of effective stresses can be expressed as a linear function of the normal stress.

\[
\tau_f = c' + \sigma'_n \tan \phi'
\]

(Equation 1.1)

Where: \( \tau_f \) = shear strength.

\( c' \) = effective cohesion intercept.

\( \sigma'_n \) = normal stress.

\( \phi' \) = effective friction angle.
Figure 1.21 Mohr-Coulomb failure criterion

From Figure 1.21, the relationship between the effective principal stresses at failure and the shear strength parameters can also be obtained.

\[
\sin \phi' = \frac{(\sigma' - \sigma_3')/2}{c'\cot \phi' + (\sigma_1' + \sigma_2')/2} \quad \text{(Equation 1.2)}
\]

\[
(\sigma_1' - \sigma_3') = 2c' \cos \phi' + (\sigma_1' + \sigma_3') \sin \phi' \quad \text{(Equation 1.3)}
\]

where: \(\sigma_1', \sigma_3'\) = effective principal stresses.

Equation 1.3 is referred to as the Mohr-Coulomb failure criterion. It is seen that the Mohr-Coulomb theory predicts a ratio of unconfined compressive strength \(Q_u\) to tensile strength \(\sigma_t\), which varies according to the frictional properties of the material:

\[
\frac{Q_u}{\sigma_t} = \frac{2 \sin \phi'}{1 - \sin \phi'} \quad \text{(Equation 1.04)}
\]

Although the Mohr-Coulomb failure criterion is widely used with success in engineering practice to describe the soil behavior under compressive forces, the criterion is a poor representation of the behavior of soils in tension, since the yield conditions in the zone of negative (tensile) stress is highly non-linear (Brace, 1960, Frydman, 1967).

There are other failure criteria, such as Mohr-Paul, Griffith, Griffith-Brace and Modified Mohr-Coulomb theories (Figs. 1.22 and 1.23, Lee and Ingles, 1968 and Fang and Chen, 1971, 1972) which have been proposed to describe the soil behavior under tensile forces.

The Mohr-Paul theory is less satisfactory than the Mohr-Coulomb theory as there is no relationship whatsoever between the unconfined compressive strength and tensile strength, though any observed ratio will be not less than that given by the Mohr-Coulomb theory.
The derived Mohr envelope for the Griffith theory is,

\[ \tau_c^2 - 4\sigma_t \sigma_n - 4\sigma_t^2 = 0 \]  

(Equation 1.5)

The predicted tensile strength is equal to one half of the apparent cohesion. Also, Griffith theory requires that the ratio of unconfined compressive strength to tensile strength be a constant equal to 8.0.

The Griffith-Brace Theory, better known as the Modified Griffith Theory (Brace, 1960), is a combination of Mohr and Griffith theories. The compressive portion of Griffith-Brace theory is essentially equal to the Mohr-Coulomb criterion, whereas the tensile portion is equal to the Griffith criterion. Again, the tensile strength must be equal to half of the "Apparent Cohesion". However, the Modified Griffith theory requires that the unconfined compressive strength to tensile ratio be a function of the frictional properties of the soils (Lee and Ingles, 1968).

The Modified Mohr-Coulomb Criterion (Fig. 1.23), proposed by Fang and Chen (1971, 1972), does not require any condition with respect to the ratio of apparent cohesion to tensile strength and the ratio of unconfined compressive strength to tensile strength.
As there is not enough experimental information to allow a definition of the non-linear part of the failure criterion (Baker, 1981), the Modified Mohr-Coulomb failure criterion seems to be superior to other criteria as it does not specify any “apparent cohesion” to tensile strength ratio. Furthermore, a different ratio of unconfined compressive strength to tensile strength is allowed in the criterion.

1.1.3.3 Fracture mechanics and desiccation cracking of soils

Although fracture mechanics developed mainly in the 60s and 70s, the basic concept of crack propagation was established by Griffith in 1921. Griffith (1921, 1924) stated that crack propagation will occur if the energy released upon crack growth is sufficient to provide all the energy that is required for crack growth. Let us consider a crack, as shown in figure 1.24, in a uniform tensile stress field. If we consider the work done by external forces (or change of strain energy per unit thickness) when the crack extends by a distance $\delta a$, then for propagation, we have,

$$\frac{\delta U}{\delta a} = \frac{\delta W}{\delta a}$$

(Equation 1.6)

$$\frac{\delta U}{\delta a} = G$$

(Equation 1.7)
\[
\frac{\delta W}{\delta a} = R \quad \text{(Equation 1.8)}
\]

where: 
- \( U \) = elastic energy release.
- \( W \) = energy required for crack growth
- \( R \) = crack resistance.

\( G \) = elastic energy release rate per crack tip or also called “crack driving force”. \( G \) is a function of Poisson’s ratio \( \nu \), elastic modulus \( E \), and stress intensity factor \( K \).

---

Let us examine the details of an elastic stress distribution in the vicinity of the crack tip. The predominant terms of the stress and displacement distributions are given in equations 1.9 (Zienkiewicz, 1977). \( K_1 \) is called the stress intensity factor, which depends on the mode of cracking, orientation of cracks and size of cracks. In three-dimensional problems, three possible modes of crack propagation are illustrated in figure 1.25. In each case the local stress distribution is similar in form to those given in equation 1.9.

The energy condition of equation 1.7 states that \( G \) must be at least equal to \( R \) before crack propagation can occur. If \( R \) is a constant, this means that \( G \) must exceed a certain critical value, \( G_{ic} \). Accordingly, \( K \), the stress intensity factor, would approach a critical value \( K_{ic} \) which is called the "fracture toughness".
Figure 1.25 The three modes of cracking

\[
\sigma_x = \frac{K_1}{\sqrt{2\pi r}} \cos \frac{\theta}{2} (1 - \sin \frac{\theta}{2} \sin \frac{3\theta}{2})
\]

\[
\sigma_y = \frac{K_1}{\sqrt{2\pi r}} \cos \frac{\theta}{2} (1 + \sin \frac{\theta}{2} \sin \frac{3\theta}{2})
\]

\[
\tau_{xy} = \frac{K_1}{\sqrt{2\pi r}} \sin \frac{\theta}{2} \cos \frac{\theta}{2} \cos \frac{3\theta}{2}
\]  
(Equation 1.9)

\[
u = \frac{K_1}{4G \sqrt{2\pi}} \left[ (2\kappa - 1) \cos \frac{\theta}{2} - \cos \frac{3\theta}{2} \right]
\]

\[
v = \frac{K_1}{4G \sqrt{2\pi}} \left[ (2\kappa + 1) \sin \frac{\theta}{2} - \sin \frac{3\theta}{2} \right]
\]

where \( \kappa = (3 - 4\nu)/(1 + \nu) \) for plane strain

\( \kappa = (3 - \nu)/(1 + \nu) \) for plane stress

For brittle materials, \( R \) consists of surface energy only, while for ductile materials, plastic deformation occurs at the crack tip. Hence, much work is required to produce a new plastic zone at the tip of the advancing crack in ductile materials.

Broek (1984) stated that as "high strength materials usually have low fracture toughness \( (K_{ic}) \), plane strain fracture problems in these materials can be successfully solved by means of fracture mechanics Low strength materials usually have a high fracture toughness \( (K_{ic}) \) and the size of the plastic zone may be so large that, at present, a versatile method to treat crack problems in high toughness materials is not yet available".

Based upon Muskhelishvili (1953) stress perturbation analysis of a crack in an elastic
medium and the Modified Griffith theory of macroscopic fracture (Irwin, 1948 and Orowan, 1950), Lachenbruch (1961) derived a set of graphs for the estimation of depth and spacing of tension cracks in brittle media, such as permafrost. Reasonable numerical values of crack depth and spacing for ice-wedge polygons in permafrost were obtained by Lachenbruch (1962). In spite of the apparent success in using the theory of macroscopic fracture to the ice-wedge polygons in permafrost, Lachenbruch (1961) admitted that the theory of brittle fracture cannot be applied to the desiccation cracking of soils, owing to their plasticity.

In several studies (Blight and Williams, 1971, Briones and Uehara, 1977 and Raats, 1984) the Griffith brittle fracture theory has been applied to the desiccation cracking of soils, with little success, however. Briones and Uehara (1977) performed beam flexion tests on soil samples at several stages of drying to obtain strain release parameters and elastic constants. They concluded that the fracture theory was not sufficient to predict cracking even with a knowledge of the soil parameters.

Within the field of fracture mechanics, the cracking problem is essentially treated as a mechanical process, and the failure (cracking) criterion is based on the critical stress field. However, Corte and Higashi (1960) stressed that "cracking by desiccation is entirely different from mechanical cracking in the sense that the material loses mass during the process. Furthermore, fracture mechanics deals only with the propagation of cracking; the initiation or on-set of cracking has not been addressed.

Based on the foregoing discussions, it appears that fracture mechanics is not adequate when dealing with the problem of desiccation cracking in soils.

1.1.4 Different techniques for the study of cracks

1.1.4.1 Direct measurement on CT specimens

Some researchers have extended classical experiments used to study fracture behavior of metallic materials to soils and rocks. The tests with CT specimens allow creating a cracking mode in a precise area of a specimen and monitoring crack growth by various means (for example, image analysis, replicas, microscopy...). This approach has been adopted by Avila (2004) in his thesis in the case of clay (Fig. 1.26), and by Bompard (2010) in the case of the Vosges sandstone. It leads to quantitative results which are interpretable in the framework of fracture mechanics.
1.1.4.2 Indirect characterization of the cracks by measuring the monophasic or polyphasic permeability

It is customary to indirectly characterize cracks in a specimen of almost saturated rock by means of permeability measurements by pulse or harmonic tests. These tests can be carried out with a mechanical loading of the sample.

Pulses

The pulse test consists in imposing a limited increase of pressure at the entrance of the sample for a relatively short time, and then measuring the dissipation of this pressure gradually as the fluid penetrates into the sample. Different methods of interpretation are used to derive the permeability of the material, making the assumption that the amount of fluid injected at each pulse being small, it does not change the state of sample. There is a rapid increase in permeability when there is creation of a traversing crack (Fig. 1.27).

The same procedure can be applied to measure multiphasic permeability, by imposing pulses of air pressure in the sample. In this case, the permeability increases when the degree of saturation decreases and when there is creation of cracks.
Harmonic method

The harmonic tests consist in submitting the sample to a sinusoidal variation of pressure at the entrance and measuring the attenuation and the phase change of the pressure wave at the output. Such studies have been performed at the University of Montpellier and at the University of Havre. They showed that, with this method, it was possible to obtain representative values of single-phase permeability. This method can also be used to characterize the cracking of material.

1.1.4.3 Indirect characterization of cracks by measuring the speed of wave propagation (elastic modulus)

Most of the mechanical and physical properties of materials is affected by the formation of cracks. It is customary to measure the change in elastic characteristics characterized, for instance, by the speed of wave propagation that is to say by the time it takes for a wave to cross the sample (Fig. 1.28).
Figure 1.28 Example of device for measuring speed of wave propagation in a sample of rock and argillite and results obtained with respect to the relative humidity of the samples. (Pham 2007 and Yang 2008)

1.1.4.4 Indirect characterization of cracks by acoustic emission

Crack formation in a sample is accompanied by the emission of acoustic signals that can be captured. This method was used by Valès 2008 in the case of Bure argillite (Fig. 1.29).
1.2.4.5 Analysis using digital image correlation

Many researchers (Avila 2004, Péron 2008, Maison 2011) have studied the cracks formed on the surface of a soil sample by direct observation with the naked eye or under a device, taking pictures at regular intervals and eventually with the analysis of images for identify different parameters of cracks, for example, length, width, area of cracks, etc.

The most common method to analyze the parameters of cracks from photos is Digital Image Correlation (DIC).

The digital image correlation consists in capturing a series of pictures of a specimen with a digital camera during the deformation process. With computer software the images are analyzed to create contour maps of displacements and strains. The specimen needs to be prepared with a random dot pattern (speckled pattern) so that the software can be able to calculate the displacements. During the tests, while the specimen is subjected to external loads, the camera takes one picture before and others after deformation: then the software analyzes the difference between pixels of the different images and correlates them to show the strain map.

This task is performed by means of various numerical techniques. The finite element method (Zienkievicz and Taylor, 1989) is one of the classical tools to analyze civil engineering structures. 2D (Kwak and Filippou, 1997; Ile et al., 2002) or even 3D (Rashid et al., 2001; Kwon and Spacone, 2002) codes are run. Very detailed analyses are possible and require full 3D constitutive models.

There are various softwares which can realize digital images correlation, for example Correl\textsuperscript{i}Q4 which is developed by Hild and Roux (2008) and VIC-2D and VIC-3D.
2D digital images correlation

For example, VIC-2D is a system that uses the digital image correlation technique to make strain calculations. This system is able to provide two dimensional strain maps of any entire planar specimen. The equipment consists of computer software, proper lighting and a digital camera (Fig. 1.30) with appropriate lens and resolution.

![Figure 1.30 The digital camera used with VIC-2D](image)

Stereophotogrammetry method

Much like human vision, two imaging sensors provide enough information to perceive the environment in three-dimensions.

![Figure 1.31 The digital cameras used with VIC-3D](image)

Recovering the three-dimensional structure of the environment using two imaging sensors is called stereo-triangulation.
Stereo-triangulation requires computing the intersection of two optical rays (Fig. 1.32). It is feasible only if these rays are formulated in a common coordinate system. We need to model and calibrate the stereo-rig (Fig. 1.33).

1.2 MICROSCOPIC STUDIES OF CLAYS

Microscopic and mesostructural studies are increasingly used to improve understanding of the macroscopic behavior and physical properties of soil materials and crack formation. These studies involve the determination of the properties of clays at microscopic level and the use of techniques at particles/aggregates scale (< 100 μm) to analyze the arrangement and distribution of particles, particle assemblies and pores – and their contact and connectivity in different soils (Collins & McGown 1974; Delage & Lefebvre 1984; Delage et al., 1996; Al-Rawas & McGown 1999; Mitchell & Soga 2005; Baker & Allmaras 1990; Fukue et al., 1999; Borsic et al., 2005; Desrues et al., 2006).

1.2.1 Mineralogy of clays

Clay includes different types of natural materials: a set of species minerals, a family of rocks, soil category or class size. A wide variety of materials with various compositions, physico-chemical properties and mechanical behaviors is contained in the general term (Homand & Duffaut, 2000; Millot, 1964; Meunier, 2005; Velde, 1995; Sparks, 2003).

Clays can be divided into two structural general forms: clayey rocks and soils. Their common feature is the proportion of clay minerals they contain. It is generally considered that a clayey soil is made up of more than 30% of fine particles whose size is less than 2 μm (Lambe & Whitman, 1969). And they are distinguished by the level of consolidation experienced during their geological history. Rocks are consolidated and indurated (Osipov & Sokolov, 1978).
1.2.1.1 Structure of clayey soils

Clay minerals are constituted by a stack of sheets. The crystalline structure of clays is important because it determines the physical properties of the mineral (Caillére & Hénin, 1959). Figure 1.34 clarifies the terminology used to define the structure of clays. There are 3 levels of organization:

- Sheets, tetrahedral or octahedral;
- Particles that are combinations or arrangements of sheets;
- Aggregates which result from the association of several particles.

![Figure 1.34 Schematic representation of the organization of a clay material](image)

1.2.1.2 Fabric of clayey soils

In addition to the crystalline structure of clays, fabric is an important parameter because it determines the physical properties of the mineral (Caillére & Hénin, 1959). The texture concerns the mineral assemblage (clay or not) as well as aggregates or arrangement distribution space ($\epsilon$) of the said units (Audiguier 1979). Aubouin et al. (1968) define texture as “the shape, size and arrangement of a number of minerals naturally grouped in a population within the rock”.

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These classifications are valid for clay soils. Gens and Alonso (1992) established a classification for expansive soils, based on the work of Collins and McGown (1974). This classification is based on the organization of elementary clay particles:

- “Matrix” Texture (Fig. 1.35(a)): texture whose base is a homogeneous mixture with elementary particles dispersed in the material. This texture is characteristic of natural soil or compacted soil on the wet side of the optimum Proctor (Fig. 1.36 (a)).

- “Aggregate” or “compacted” Texture (Fig. 1.35(b)): texture packed with assemblies of elementary particles forming clumps or aggregates. This texture is characteristic of compacted soil on the dry side of the Proctor optimum (Fig. 1.36(b)).

Figure 1.35 Typical fabric of swelling soils

Figure 1.36 SEM photos of matrix texture: (a). Green clay of Romainville and aggregate texture ; (b). Clay of Bavent (Vincent et al., 2009)
1.2.1.3 Arrangement of soil particles

Among the clay minerals, the most common are (Mitchell, 1976):

- Kaolinite ($1/1^2$, $d^3 = 7\text{Å}$) (Fig. 1.37). No substitution happens within the layers. The layer is neutral. Kaolinite forms in well–drained soils with acidic pH, especially in tropical and subtropical climate. Its crystals are often large up to 15 μm.

- Illite ($2/1$, $d = 10\text{Å}$). Association of O layer (alumina) and two T layer (silica). There may be substitutions (replacement of Si by Al). Cations ($K^+$) are absorbed in the interlayer space to compensate for the charge imbalance. This is the most frequent mineral. Its structure is similar to muscovite (with more water and less $K^+$).

- Smectites ($2/1$, $d = 14 \text{Å}$) (Fig. 1.37). The stacking of the layers is disordered; each layer is rotated in its plane compared to the previous. Substitutions atoms are important. This disorder and weakly charged layers facilitate their gap(separation) and the adsorption of various molecules (water, cations, organic molecules) in the interlayer space ($d = 18 \text{Å}$). Smectite, or montmorillonite, is usually calcic, seldom sodic. They are formed in poorly drained alkaline soils. Layers of smectites can intercalate regularly or irregularly with other clay layers, often illitic. The overall shape is interstratified.

- The glauconite: Green mineral ferroan close to illite, exclusively formed in relatively shallow marine medium.

- The chlorites ($2/1$, $d = 14 \text{Å}$): The interlayer space is lined by a layer composed of Mg and OH. The Al is replaced locally by Fe, chlorites exist in larger crystals in igneous rocks. They are also formed during diagenesis of sedimentary rocks.

- The vermiculite ($2/1$, $d=12 \text{Å}$): It is very common in soils of the temperate zone. The octahedral layer contains Fe and Mg. Vermiculite is close to illites and chlorites but shows swelling properties.

- The fibrous clays: The layers are discontinuous and form ribbons. The main types are the sepiolite and attapulgite or paligorskite. They are found in confined environments.
(1) Shapes of mineral crystals

Using the electron microscope, clay mineralogists have made excellent progress in determining the sizes and shapes of the clay mineral crystals.

Shapes of some of the most common clay minerals are (Fig. 1.38):
Kaolinite – sheets (often regular hexagonal sheets)
Halloysite – tubes of curled sheets
Montmorillonite – flaky sheets
Nontronite – laths
Illite – irregular flaky sheets
Attapulgite – narrow fibers
Chlorite – sheets
Vermiculite – sheets

Thus, most of the mineral particles which contribute to the plasticity of natural clays are not equidimensional (with the same dimension in every direction); they are usually sheets or plates and sometimes rods or laths.

The other minerals (i.e., quartz, feldspars, carbonates, and oxides), common in natural clays, are irregularly shaped, but their particles are usually not far from equidimensional. These particles can be considered, with reasonable accuracy, as rough-edged shapes approaching sphere.

(2) Possible arrangements of particles

Since the size and shape of particles in a natural clay vary, and especially since the most important particles are far from equidimensional, there are many possible arrangements of particles. Very irregular and very complex patterns are possible. The patterns of arrangements are less complex in compacted clays than in sedimentary clays, because stratifications are less important. Sedimentary clays usually have layers of different sized particles resulting from different conditions of deposition.

Mitchell (1956) worked out a semi-quantitative system for describing particle arrangement, or fabric. The items in his system are:

- Texture: regularity of components; transition between components;
- Particle orientation: within small areas, i.e., $< 1500 \, \mu m$; within large areas, i.e., $> 1500 \, \mu m$; clay around silt.

Mitchell used scales to describe these factors, as:

- Zero for irregularity to 90 for a high degree of regularity
- Zero for poor transition to 90 for no discontinuities
- Zero for perpendicular to 100 for parallel plates.

By means of a complete microscopic study, including measurements of interference fringes of thin sections under polarized light, Mitchell assigned numbers to the fabric of several clays. He enjoyed some success in relating his fabric measurements to engineering behavior; his technique is, however, too time-consuming and complex for routine work.
(3) Arrangements of particles occurring in soils

The arrangement of silt-sized particles can be, and has been, easily determined by means of microscopic examination. The preparation of clays for examination with an electron microscope may destroy the structure in a natural soil; in fact, some researchers think that the severe pretreatment (especially drying) given specimens for the electron microscope actually changes the size and shape of some clay crystals, e.g., nontronite. In some cases, the replica techniques may give reliable electron photo-micrographs of soil without disturbing the structure. The limitation of microscope resolution prevented Mitchell from making reliable determination of particles orientation in zones smaller than 10 μm wide. He had to assume, therefore, that the orientations he measured within aggregates, 10 μm and larger in size, existed between individual particles. His works on specially prepared specimens of known particle orientation indicate his technique does give correct results (Lambe, 1958).

1.2.1.4 Surface area and binding capacity of clay particles

The size of clay particles, which is relatively small and thin gives them a high specific surface with respect to the volume of the particles that they define. The relative area increases with decreasing diameter. The thickness/width ratio is in the range of 20 for clays. The physical properties of clays are mainly controlled by the surface area.

Table 1.1 gives typical values of the surface areas of the main families of clays. The total area comprises the outer surface, between the clay particles, and the inner surface, corresponding to the interlayer space (Fig. 1.39). Smectites have the maximum total area:

Surface: Smectites > Vermiculites > Illites > Kaolinites = Chlorites.

<table>
<thead>
<tr>
<th>Clays</th>
<th>Specific surface (m²/g)</th>
<th>Cation exchange capacity (meq/100 g) of clayey minerals</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Internal</td>
<td>External</td>
</tr>
<tr>
<td>Smectite</td>
<td>750</td>
<td>50</td>
</tr>
<tr>
<td>Vermiculite</td>
<td>750</td>
<td>&lt; 1</td>
</tr>
<tr>
<td>Illite</td>
<td>5</td>
<td>25</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>0</td>
<td>15</td>
</tr>
<tr>
<td>Chlorite</td>
<td>0</td>
<td>15</td>
</tr>
</tbody>
</table>
The cation exchange capacity (CEC) measures the ability of a clay to exchange cations with the water environment. It measures the concentration of cations in the mobile diffuse layer and depends on the total cation charge (that is to say, surface charge). The CEC is pH dependent and is usually given for a neutral pH (pH $\approx 7$) (Laribi et al., 2006).

The CEC values for the major clay families are shown in Table 1.1.

1.2.2 Different techniques for studying the microstructure of clays

The methods which were used in this thesis will be presented in detail in Chapter 2. We will only present here some methods often found in the literature for the study of the microstructure of clays but not used in the thesis.

1.2.2.1 X-ray diffraction

X-ray diffraction is a non-destructive analytical technique which reveals information about the crystal structure, chemical composition, and physical properties of materials and thin films. This technique is based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident angle, polarization and wavelength or energy.

When a sample is bombarded by incident X-ray, the condition for observing a maximum intensity in the direction $\theta$ of the incident ray is that the wave diffused by the nodes of different planes are in phase, thus verifying the law of Bragg (Fig. 1.40):

$$2ds\sin(\theta) = p\lambda$$

Note: $d$: equidistance between the crystallographic planes;
\( \theta \): angle between the incident and diffracted rays;

\( p \): order of reflection;

\( \lambda \): wavelength of the radiation used;

Figure 1.40 Schematic diagram of XRD

X-ray diffraction is a nondestructive technique to identify crystalline phases and orientation. The other applications of XRD are:

- Determination of structural properties (Lattice parameters (10^{-4} \text{ Å}), strain, grain size, epitaxy, phase composition, preferred orientation (Laue) order-disorder transformation, thermal expansion) (Souli, 2006).

- Measurement of thickness of thin films and multi-layers;

- Determination of atomic arrangement;

- Detection limits: 3% in a two phase mixture; can be reduced to 0.1 % with synchrotron radiation.

The XRD is an important means of identifying clay species and non-clay minerals constituting the samples. In the case of a clayey material, following the evolution of the positions of the reflections allows to know the distance between two successive sheets. In swelling material, the interlayer distance is also representative of the state of hydration of the clay (Souli, 2006). The increase in the intensity of this reflection is representative of a stack of large sheets. Peak width at half-height also gives information on the number of stacked layers. Indeed, a narrow line reveals a stack of a large number of sheets whereas a small number of layers results in a broad line. Hk bands provide information on the organization of the material, increasing the intensity of these bands shows a disorientation of clay layers (Guillot, 2001).
1.2.2.2 Infrared spectroscopy

Infrared (IR) spectroscopy is one of the most common spectroscopic techniques used by organic and inorganic chemists. Simply, it is the absorption measurement of different IR frequencies by a sample positioned in the path of an IR beam. The main goal of IR spectroscopic analysis is to determine the chemical functional groups in the sample. Different functional groups absorb characteristic frequencies of IR radiation. Using various sampling accessories, IR spectrometers can accept a wide range of sample types such as gases, liquids, and solids. Thus, IR spectroscopy is an important and popular tool for structural elucidation and compound identification.

IR absorption positions are generally presented as either wavenumbers ($\tilde{\nu}$) or wavelengths ($\lambda$). Wavenumber defines the number of waves per unit length. Thus, wavenumbers are directly proportional to frequency, as well as the energy of the IR absorption. The wavenumber unit (cm$^{-1}$, reciprocal centimeter) is more commonly used in modern IR instruments that are linear in the cm$^{-1}$ scale. In the contrast, wavelengths are inversely proportional to frequencies and their associated energy. At present, the recommended unit of wavelength is $\mu$m (micrometers).

Wavenumbers and wavelengths can be interconverted using the following equation:

$$\tilde{\nu} \text{ (in cm}^{-1}\text{)} = \frac{1}{\lambda \text{(in} \mu\text{m)}} \times 10^4 \quad \text{(Equation 1.10)}$$

Infrared radiation spans a section of the electromagnetic spectrum having wavenumbers from roughly 13,000 to 10 cm$^{-1}$, or wavelengths from 0.78 to 1000 $\mu$m. It is bound by the red end of the visible region at high frequencies and the microwave region at low frequencies.

IR absorption information is generally presented in the form of a spectrum with wavelength or wavenumber as the x-axis and absorption intensity or percent transmittance as the y-axis (Fig. 1.41). Transmittance, $T$, is the ratio of radiant power transmitted by the sample ($I$) to the radiant power incident on the sample ($I_0$). Absorbance ($A$) is the logarithm to the base 10 of the reciprocal of the transmittance ($T$).

$$A = \log_{10} \left( \frac{1}{T} \right) = -\log_{10} T = -\log_{10} \frac{I}{I_0} \quad \text{(Equation 1.11)}$$

The transmittance spectra provide better contrast between intensities of strong and weak bands because transmittance ranges from 0 to 100%, whereas absorbance ranges from infinity to zero.
1.2.2.3 SEM and ESEM

The classical SEM observations provide images of high quality but with the drawback of having to completely dry, and most often, metalize the soil samples. Coupled with the analysis of the orientation of the particles (refer to chapter 2), the method has the advantage of being able to quantify the phenomena with great finesse.

The peculiarity of the environmental SEM (ESEM) is to have a differential pumping system separate for the chamber and the column; the column is subjected to high vacuum \(10^{-5}\) Torr while, in the chamber, a pressure in the range \((0.1 – 20\) Torr) is sufficient. The pressure in the environmental SEM is controlled by a gas flow at different apertures of the system. In the study on geo-materials, the gas used is water vapor which plays the role of both ionization gas for forming the image and hydration fluid for the sample.

Thus, the environmental SEM can reconstruct environmental (or in-situ) conditions of water content by controlling temperature and pressure within the chamber. The samples can be studied in a quasi-saturated state with their natural or desired hygrometry. Concurrently, a chemical microanalysis by energy dispersive X-ray can be performed during the observation of the entire field of vision or a given particle. When this analysis is carried out on several successive images, it provides a visualization of the evolution of chemical components on the surface of the sample (mapping X). After the acquisition, image processing is difficult, especially in environmental mode in which the quality of images is somewhat degraded. The analysis of images acquired in the ESEM can be used to define many parameters such as the size of particles in two perpendicular directions of the observation plane, the apparent diameter, surface, etc. 3D modeling from stereoscopic images can be also used to characterize...
the evolution of the surface topography of a sample during cycles of wetting / drying (Maison, 2011).

There are some limits in this method; the pressure range may seem quite low (0.1-20 Torr) based on experimentations that are done. The accuracy of the relative humidity around the sample is a function of the precision on the measures of temperature and pressure, and is not sufficiently high, especially in the range of RH between 90% and 100%. Indeed, in this range, a small change in relative humidity induces a strong variation of suction and deformation, hence the importance of precision. The full saturation of the sample (including internal) cannot be verified, given that the observation is surfacic. The field of observation which relies on the environmental detector limits the observable surface, especially if we increase the magnification to observe small aggregates, which prevents an overview of the sample. In order to get photos and videos of the highest quality possible, the adjustment of contrast and brightness is important and must be settled automatically and continuously.

1.2.2.4 Pore size distributions (PSD) in clays

Delage and Lefebvre (1984) used the porosimetry technique to investigate fabric changes of a sensitive natural clay during consolidation and to define entrapped and free porosity on clay structure, Griffiths and Joshi (1989) studied changes in PSDs due to consolidation of different clay types, and Al-Mukhtar (1995) analyzed pore space results on a kaolinite clay relative to the effects of mechanical and hydraulic (partially saturated) states. Prapaharan et al. (1985) predicted the soil moisture characteristic curve up to a matric suction value of 0.7 MPa based on porosimetry data. Enrique and Simms et al. (2008) studied the microstructure of partially saturated soils with two widely used techniques – MIP and SEM. Penumadu and Dean (2000) quantitatively evaluated the compression that can occur during the evaluation of pore-size distribution of cohesive soil using MIP. Monroy and Zdravkovic et al. (2010) carried out a study to investigate the evolution of fabric in compacted natural clay during drying and loading with MIP and SEM in order to observe and quantify the change in fabric associated with each path.

1.2.3 Influence of stresses and suction on the porosity, the arrangement of particles, their orientation, etc.

A complete physical description of the state of a clay soil or an experimental clay mass is often of considerable interest. Usually the information available is confined to measurements of gross parameters, such as moisture content, total porosity etc. Less frequently additional descriptors such as particle-size distribution, surface area and information on mutual orientation of particles is supplied.

Morgenstern & Tchalenko (1967) analyzed the effect of direct shear tests on specimens of kaolin using polarized light microscopy. They highlighted the strong orientation of the clay
particles in the direction of the movement. Several investigators used various techniques to study the evolution of clay fabric during one-dimensional compression tests: Delage & Lefebvre (1984); Pusch (1970), for example, showed by means of scanning electron microscopy that the structural anisotropy of remoulded materials progressively develops itself with the loading. Bai & Smart (1997) used scanning electron photographs to highlight the change in the microstructure of kaolin on undrained axisymmetric triaxial paths. The authors observed first an increase in the anisotropy of the samples with strain, followed by a decrease. However, they reported some difficulties, suggesting a possible instability in the microstructural response to the strain increase, since similar samples showed opposite responses under the same conditions. Hicher et al. (2000) used scanning electron micrographs to study the changes in the microfabric of saturated kaolin and bentonite after drained triaxial tests: they observed the creation of structural anisotropy during loading and concluded that the mechanical behavior is largely dependent on the changes that occur at the particle level. Using petrographic investigations on a kaolinitic matrix, Dudoignon et al. (2001) came to the same conclusion, showing that the percentage of oriented particles increases from a sub-isotropic matrix induced by the isotropic loading to an anisotropic matrix for the normally consolidated sheared samples. The research of Holtz and Kovacs (1981) and Mitchell (1993) showed that capillary forces associated with the loss of water content in soils is the environment which causes soil shrinkage. During desiccation, suction develops and increases the effective stresses in the soil. In return, the volume of soil begins to decrease and cracks develop in soils. Cracks propagate with increasing suction. Soils with fine grains are more susceptible to the development of cracks than those with large grains. This is due to the presence of small pores which allow the development of high suctions.

Hammad (2010) analyzed the orientations of five clayey materials on oedometric and isotropic paths, using the same method as that presented in chapter 2. In the oedometric tests, two vertical stresses (200 and 600 kPa) were applied to the materials. The author concluded that the initial oedometric loading led to a preferential orientation of the particles which was progressively erased by the subsequent isotropic loading when the applied stress was sufficiently high (Fig. 1.42).
Hattab et al. (2010) analyzed the relation between the behavior of a clayey material at the macroscopic level and its local deformation properties (Fig. 1.43). The orientation of the clay particles by SEM images analysis after different phases of triaxial loading is studied. In the initial state (one-dimensional compression), the observations highlight the microstructural anisotropy of the slightly overconsolidated specimens with a preferential orientation of the particles normal to the loading direction.

Figure 1.43 Microfabric in initial state after oedometric consolidation to $\sigma'_v=120$ kPa, example of a region observed on a vertical plane
SEM observations of Bure clay-rock on the planes perpendicular to bedding (Fig. 1.44 (a)) carried out by Wright (2001) showed a preferred orientation of the particles in the clay matrix (Fig. 1.44 (b)) along the bedding planes. Some planes present a large spacing between the particles and clayey platelets which can accommodate non-clayey minerals (Fig. 1.44 (c)). The author concludes that the texture is characterized by a lamellar porosity mainly located between the clayey particles.

![Figure 1.44 SEM Observations of clay rocks (Wright, 2001)](image)

### 1.2.4 Formation and evolution of cracks

#### 1.2.4.1 Cracks in metal and concrete

The microprocesses of crack initiation and propagation in a ductile iron have been investigated by Dai et. al. (2004) using a scanning electron microscope with a microtensile holder. It was revealed that microcracks always initiated at, and propagated along, the graphite-matrix interface. Microcracks in the matrix initiated after the interface cracking. The graphite nodules in this cast iron could not be regarded as voids with no strength because internal fracture of the graphite was observed. When the cracks propagated in the matrix, they often grew along the bainitic ferrite-austenite interface.

Desiccation also affects concrete. In some aspects, concrete behavior is similar to that of the soil. They are both commonly classified as geomaterials. Drying of concrete is restricted to the porous matrix and slowly spreads from the exposed surface to the core of the structure. At the beginning, before concrete setting, induced shrinkage occurs relatively rapidly, and is irreversible (Favre, 2004), but may last several years. Actually, the desiccation process (drying shrinkage) in concrete coexists with inside (inner) shrinkage. Drying shrinkage, including inside shrinkage, may induce non-negligible shrinkage. When the shrinkage is constrained, which is almost always the case in concrete due to gradients or reinforcements, cracking is bound to appear. Many times cracks are thin and cannot be seen by eyes. However, they can affect the structural properties of the concrete, increase the permeability of the
medium and cause a loss of pre-stressing (Bazant & Raftshol, 1982; Bazant & Wittmann, 1983).

1.2.4.2 Cracks in clayey rocks

The difference in the mechanical properties, especially deformability, of the minerals constituting the clay rocks leads to high local deformations in the clay and low relative values in the quartz grains and calcite matrix. Shear stress develops at the interfaces, resulting in microcracking (by detachment) between the clay/quartz/calcite particles (Fig. 1.45). This is observed by Bornert (2001) on sample images taken by SEM.

(a) (b)

Figure 1.45 SEM images of the Callovo-Oxfordian clay rock sample (a) before (b) after the triaxial test: appearance of microcracks (Bornert, 2001)

The SEM observations carried out by Noiret (2009), combined with a method of digital image correlation, on Tournemire clay rock under uniaxial loading, show that the average strain field is rather homogeneous in the linear phase of the macroscopic (stress/strain) behavior. In the case presented in figures 1.46 and 1.47, however, a crack appears to propagate from the base of the sample into the clay matrix and bypass the stronger elements of the material. Microcracks are also observed but they close during loading without much impact on the average field deformation. Two questions are raised: the first one concerns the role of freeze-drying in the observed behavior. The second is about the scale seen in the observations and the issue of VER. This is clearly stated in figure 1.47 through the variation curves of the average strain versus load, which show linear trends and become almost superposed when the study area is enlarged (i.e. magnification is less important).
Figure 1.46 In-situ compression tests in SEM on Tournemire clay rocks (Noiret, 2009)

Figure 1.47 (b) Axial strains/stress curves of two zones observed at different scales
Characteristics of the equivalent average strain field of the Tournemire clay rock (Noiret, 2009)
Montes (2002) investigated the microstructural changes of clay rocks subjected to drying/wetting cycles. For this purpose, he performed ESEM observations on different samples, which allowed him to analyze the influence of the cracks caused by clay swelling and to observe the location of the cracks. The observations are done in two planes (the plane parallel to the lithology, and the perpendicular plane).

When the fraction contained in the clayey material is swelling (smectite), the material is more susceptible to crack when subjected to drying/wetting cycles (Figs. 1.48 and 1.49). These cycles result in the opening and closing of the pores and cracks, particularly visible on the planes perpendicular to bedding. It seems that, after a few cycles, the crack may be partially reversible or irreversible, and that this depends on the clay family contained in the clay rocks. Finally, the presence of large rigid minerals seems to produce cracking (also observed after mechanical testing) and the presence of smectite influences the opening/closing of cracks.

![Figure 1.48 ESEM photos presenting the evolution of a crack during cycles of water condensation/evaporation (Montes, 2002)](image)

Figure 1.48 ESEM photos presenting the evolution of a crack during cycles of water condensation/evaporation (Montes, 2002)
It appears in different studies that cracking (and fracturing) under mechanical (and thermomechanical) loading depends strongly on the minerals present in the material and their microstructural organization. The presence of pyrite, even less than 2% in the material, results in a significant decrease in global strength and is responsible for the weak points where the first crack is initiated. The work of Wright (2001) conducted at different scales (macro-meso-micro) on the analysis of behavior of clay rocks in Bures also shows different causes and types of cracks. These studies also indicate that the failure is due to shear fractures that generally develop along bedding planes. They will constitute weak zones where the shear mechanism tends to appear. Under the conditions of the tests carried out by Wright (Fig. 1.50), the macroscopic deformation is related to the opening of cracks caused by tension and shear strength (local path), primarily depending on the fabric heterogeneities. The tests consist of compression tests under controlled temperature and relative humidity conditions.
The brutal contact of the clay rock with water leads to a rapid disintegration of the material (Wright, 2001). Therefore, caution should be taken with core sampling on site (core made using an oil slurry) or in the laboratory (dry drilling). In the case of prolonged immersion under axial loading such as in contact with drilling mud in the water during the extraction of rock carrots, it develops microcracks which are oriented in the plane of stratification or without preferred orientations. The disintegration, (Fig. 1.51) the degree of brittleness and also the deformation of the samples depend on the type of ions present in the solution (Fig. 1.52) (Freissmuth, 2002; Wakim, 2005).
Figure 1.51 Micro-X-ray samples of clay rocks (in Bures at 482 m depth) after different times of immersion in solutions of different concentrations (Freissmuth, 2002)

Figure 1.52 Swelling pressure versus ions concentration in the solution (C) and load at the surface of the clay (σ) which depends on its mineralogy (Gaombalet, 2004)

1.2.4.3 Cracks in clays

Avila (2004) carried out tests on clays to observe the initiation, propagation and stabilization of cracks (Fig. 1.53). One of the aims of this research was also to test the applicability of fracture mechanics in clays. The conclusion is that, at the beginning of desiccation, the cracks are very small and superficial. These microcracks are usually not the cause of the final cracking, since this is mainly related to the limit conditions imposed by the geometry of the sample (Fig. 1.54). The propagation of cracks is not continuous but happens for very short sections. The cracks are usually interconnected by larger particles or cluster of soil. With increasing desiccation, the cracks extend and coalescence of the small cracked sections occurs. They do not follow a straight line (Fig. 1.55). The morphologies observed in cracking clays are similar to those found in mortar and concrete, where the presence of grains with larger size influences the orientation and shape of the cracks (Fig. 1.55).
Figure 1.53 Appearance of secondary microcracks and the propagation of microcracks:
A. Advance of secondary microcrack; B. Appearance of secondary microcracks; C. Soil filaments remain between the faces of the microcracks (Avila, 2004)

Figure 1.54 Discontinuous microcracks observed at the beginning of desiccation:
A. Details of two overlapping microcracks
B. Particle surrounded by microcracks and tips
C. Separation between the sample and a mold wall (Avila, 2004)

Figure 1.55 The coalescence of microcrack sections to form a continuous microcrack (compared with fig. 1.54) (Avila, 2004)

Several studies have led to a better understanding of the morphological description of the cracks during desiccation. Perrier et al. (1995) characterized the structure of cracks which is very important for the evolution of the geotechnical properties of the soil-water system. If the cracks form a network (size, connectivity, connection), the soil behavior on wetting-drying path can be predicted.
In order to quantify the cracks in soils, different techniques have been used. The first method is to measuring the cracks directly (Zein El Abidine and Robinson, 1971; Lima and Grismer, 1992). It is common to measure the larger cracks with a rule. Otherwise, the cracks with irregular form and complicated geometry are not easy to quantify. Another approach is used. Imaging techniques have been developed to quantify the characteristics of microcracks. Hallaire (1994) describes the microcrack orientation in a clayey soil using image analysis.

1.3 SUMMARY

This chapter contains an outline of the existing literature concerning macroscopic and microscopic researches of cracks in clayey materials. At the macroscopic level, the definition of cracks in a clayey materials, in-situ and in the laboratory observations of cracks, shrinkage and desiccation of soils, water retention characteristics of clayey materials, physical behavior between fracture mechanics theory and the soil cracking, and the factors affecting soil shrinkage and cracking are presented. The mineralogy of clays, different techniques for studying the microstructure of clays, the influence of stress and suction on the porosity, the arrangement of particles, their orientations, evolution and formation of cracks in microscopic level are introduced.

Although the solution of the problem of desiccation cracking is not obvious, the following conclusions were drawn:

1.3.1 Macroscopic study of cracks

(1) Soils shrink in response to a change in the state of stress and especially, suction. The state of stress depends on different environmental factors, such as land use and climate (for in-situ cracks), support-layer friction, thickness of soil layers (for laboratory tests).

(2) The amount of soil shrinkage depends on the type and content of clay minerals, soil fabric, initial water content and confining pressure.

(3) Desiccation cracking is the result of volume reduction in soils due to shrinkage (or contraction) during the drying process.

(4) In nature, orthogonal patterns are usually observed in desiccation cracking. Orthogonal intersections suggest that one of the cracks predates over the other cracks.

(5) Recent studies conducted by soil scientists and geotechnical engineers have used soil suction as the stress state variable to relate to the shrinkage behavior of soils.

(6) Soils have a low tensile strength ranging from zero to about several kPa. The tensile strains at failure for compacted soils increase with the decrease in water content.
1.3.2 Microscopic study of cracks

(1) Clay includes different types of natural materials. Structure of clays, their fabric, arrangement, surface area and binding capacity influence the appearance of cracks and the properties of clays.

(2) From the experimental point of view, many works investigated the microstructure of clayey materials under various stress conditions. These studies require the use of several techniques at particles and aggregates scales (<100 μm) to analyze the organization and orientation of particles, particle assemblies and pores, e.g. (i) optical microscopic observation of thin sections, (ii) scanning electron microscopy, (iii) X-ray diffraction, (iv) mercury intrusion porosimetry etc.

(3) Cracking (and fracturing) under various stress conditions depends strongly on the minerals present in the material and their microstructural organization.

(4) Microcracks in metal, concrete and clayey rocks have been widely studied by many investigators. However, in the case of clays, the literature is scarcer and the subject needs more attention. With different techniques at the microscopic level, the appearance, propagation and stabilization of microcracks can be detected.
CHAPTER 2 MATERIAL PROPERTIES AND EXPERIMENTAL METHODS

2.1 MATERIAL PROPERTIES

Six materials were studied in this research:

- kaolin P300, a little swelling “nearly pure” kaolinite;
- Ca-montmorillonite, a very swelling “nearly pure” montmorillonite;
- three mixtures of kaolin P300 and Ca-montmorillonite containing 35%, 50% and 65% of montmorillonite;
- a claystone taken from an experimental nuclear waste depository in the east of France (Bure).

Kaolin P300 is an industrial clay containing about 95% of pure kaolinite, marketed by Dousselin (Rhône, France). Montmorillonite is a natural clayey soil coming from the Milos island in Greece.

The characteristics of the remoulded clays (kaolin P300 and Ca-montmorillonite) and their mechanical behavior have been the subject of several studies in MSSMat laboratory, Ecole Centrale Paris and other laboratories. In this study we try to understand the behavior of the clays, especially the crack phenomenon and its propagation during drying, at two levels: at the macroscopic level using digital image correlation analysis, suction-controlled drying-wetting tests, traction tests, etc. and, at the microscopic level, by a study of the evolution of the microstructure under different suctions using SEM images analyses and MIP tests.

Another objective is to explore the effect of mineralogy on the behavior of these soils. It is the reason why we chose 5 mixtures of kaolin and montmorillonite where liquid limits vary ranging from 40% to 170%). The microscopic approach that we propose consists in following the microstructural evolution of the materials, especially the orientation of clay particles, as a function of their hydric state. This microscopic study will be complemented by mercury intrusion porosimetry, a method which allows to quantify the pore space and to characterize the void ratio.

This approach will allow us to better understand the role of each component of the mixture in the behavior of the clays.

The other material used in this research is a claystone taken from the east of France.
(Bure, Meuse - Haute-Marne) from the site of the Andra laboratory. During the previous years, studies of these claystones were conducted by several researchers, which led to a series of physical and mineralogical data and the determination of their thermo-hydro-mechanical properties. These parameters permit a better characterization of the material and prediction of its behavior under different loads. The samples were taken from the site at approximately 500 m depth and kept under stress and isolated from the atmosphere until they were tested.

2.1.1 Mineralogy and physical properties

2.1.1.1 The clays

In this research, 5 mixtures of kaolin and montmorillonite were made by varying the proportion of the two components (by weight) (Table 1). Thus, the kaolin P300 is noted K, the montmorillonite M100, the mixtures Mx, where x indicates the percentage of montmorillonite.

The physical properties of kaolin P300 (K), M35, M50, M65 and montmorillonite (M100) are summarized in Table 2.

The mineralogical analysis of kaolin P300 carried out by X-ray diffraction (Hammad, 2010) reveals the presence of various minerals, including kaolinite, illite and quartz. The observation by scanning electron microscopy (Figure 2.1) confirms this analysis and shows that the clay is mainly kaolinite with the presence of some illite and quartz particles.

The montmorillonite is a calcium montmorillonite with a dioctahedral character (Souli et al., 2007, 2008). The photo taken with an electron microscope on a saturated sample Hammad, 2010) shows a complex texture. The clay particles of montmorillonite present a three-dimensional structure (Hammad, 2010; Tessier, 1984) with pore spaces between the particles much larger than in the case of kaolin P300 (Figure 2.1 (b)). The kaolinite particles, compared to those of montmorillonite, are better defined in terms of platelet shape: flatter, smaller and more compact.

Table 2.1 The mixture clays

<table>
<thead>
<tr>
<th>Materials Mx</th>
<th>% of kaolinite</th>
<th>% of montmorillonite</th>
</tr>
</thead>
<tbody>
<tr>
<td>K</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>M35</td>
<td>65</td>
<td>35</td>
</tr>
<tr>
<td>M50</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>M65</td>
<td>35</td>
<td>65</td>
</tr>
<tr>
<td>M100</td>
<td>0</td>
<td>100</td>
</tr>
</tbody>
</table>
Table 2.2 Physical properties of kaolin P300, M35, M50, M65 mixtures and montmorillonite, *(Hammad, 2010)*

<table>
<thead>
<tr>
<th></th>
<th>Liquid limit (w_L(%))</th>
<th>Plasticity index (I_p(%))</th>
<th>Density of the solid grains (\rho_s(\text{cm}^2/\text{s}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kaolin P300</td>
<td>40</td>
<td>19</td>
<td>2.65</td>
</tr>
<tr>
<td>M35</td>
<td>82</td>
<td>33</td>
<td>2.67</td>
</tr>
<tr>
<td>M50</td>
<td>100</td>
<td>37</td>
<td>2.69</td>
</tr>
<tr>
<td>M65</td>
<td>118</td>
<td>42</td>
<td>2.70</td>
</tr>
<tr>
<td>Montmorillonite</td>
<td>170</td>
<td>119</td>
<td>2.73</td>
</tr>
</tbody>
</table>

Figure 2.1 a) Kaolin P300 *(Hammad, 2010)*
2.1.1.2 The claystone

(1) Mineralogical properties

Bure claystones consists of 40% to 50% clay, primarily smectite, with a small content of kaolinite, 25% to 30% quartz and 20% to 30% of carbonates (Bauer-Plaîdoux et al., 1998). The water content is between 3% and 9%; the void ratio is in the range of 0.15 to 0.2.

The clayey minerals in Bure claystones appear in the form of clusters of a few micrometers, which often take the shape of the grains which they surround (Su, 2005) as can be seen in figure 2.2. Thus the geometry of the structure at the mesoscopic level appears to be controlled by non-clayey minerals (Sammartino et al., 2003).
Identification of clayey minerals was conducted using specific procedures (by X-ray diffraction with non-oriented and oriented powders and microscopic observations) on the whole claystones and on claystones grains smaller than 2 μm. The semiquantitative estimation took into account, in addition to the X-ray diffraction, the chemistry of the rock and the cation exchange capacity (CEC) to establish a normative calculation. The accuracy of this approach is about ± 5%.

The mineralogical composition of the claystone varies with depth. According to the mineralogical analysis of some samples, the following proportions were established:

- 30 to 60% of clays, mainly interstratified illite-smectite (50 to 90% of the clayey fraction, according to the results of X-ray diffraction). The proportion of smectite is greater in the upper part. This result is also reflected by cation exchange capacity (CEC), which is higher in the highest levels than in the lowest levels;
- Quartz whose percentage is slightly less than that of the clay;
- 20 to 40% of carbonates, primarily CaCO₃;
- Less than 5% of accessory minerals such as pyrite.

(2) Physical properties

The natural density of claystones is between 2.32 and 2.61 with an average value of 2.42.

The natural water content, calculated by differential weighing before and after oven drying at 110°C for 48 hours is between 2.8 and 8.7 % with an average value of 6.7 %.

The porosity was determined from the density value; its value is between 9 and 18%
with an average value of 14%.

Figure 2.3 presents the evolution of physical (water content, density, carbonate content, porosity and CEC), acoustic (compressional and shear wave velocities), mechanical (traction and compression) and thermal (conductivity and expansion) properties with depth.
2.1.2 Grain size distribution curve

2.1.2.1 The clays

The grain size distribution of kaolin P300 and montmorillonite was studied by laser granulometry. The curve of figure 2.4 shows that the fine fraction of kaolin consists of nearly 59% of particles whose size is less than 2 μm, whereas for montmorillonite, there are 80% smaller than 2 μm.

Laser granulometry is more accurate than sedimentometry measurements presented in table 2.3, (Montmorillonite by Fleureau et al., 1992; kaolin P300 by Hicher et al., 2000) which gives systematic underestimation of the proportions of particles less than 2 μm.

![Figure 2.4 Grain size distribution of kaolin P300](image)

For montmorillonite, 100% of the particles are smaller than 80 μm, 40% of the particles are less than 2 μm and \(d_{50}\) is 4 μm.

Table 2.3 Identification of grain size distribution of kaolin P300 and montmorillonite

<table>
<thead>
<tr>
<th>Granulometry</th>
<th>Kaolin P300</th>
<th>Montmorillonite</th>
<th>Method of analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 80 μm (%)</td>
<td>100</td>
<td>100</td>
<td>Laser</td>
</tr>
<tr>
<td>&lt; 2 μm (%)</td>
<td>83</td>
<td>80</td>
<td></td>
</tr>
<tr>
<td>&lt; 80 μm (%)</td>
<td>100</td>
<td>100</td>
<td>Sedimentometry</td>
</tr>
<tr>
<td>&lt; 2 μm (%)</td>
<td>59</td>
<td>40</td>
<td></td>
</tr>
</tbody>
</table>
2.1.3 Compressibility of the materials

2.1.3.1 The clays

Oedometric path: the following oedometric tests were performed by Hammad (2010) on the clays (kaolin P300, M35, M50, M65 and montmorillonite) from different initial states of the material:

- From mud prepared at \( w_i = 1.5 \, w_L \) and poured directly into the mold previously greased.
- On samples pre-consolidated in a consolidometer up to a vertical effective stress \( \sigma'_V = 100 \, \text{kPa} \)

The same procedures were followed for all the materials, the loading being done step by step.

(1) Kaolin P300

Figure 2.5 shows the evolution of the void ratio as a function of effective vertical stress: the purple line corresponds to the correlations of Biarez and Hicher (1994) with the Atterberg limits.

![Figure 2.5 Oedometric loading (kaolin P300) (Hammad, 2010)](image)

(2) M35

The M35 mixture contains 65% kaolinite and 35% montmorillonite. Figure 2.6 shows the results of oedometric tests on the mixture: the correlation is still representative of the mixture with a mean value of \( C_c \) equal to 0.65 and an average
value of Cs of 0.092. Cc/Cs for this mixture is equal to 7, which is close to the expected value for remolded clays. Between the test performed on a pre-consolidated sample and that on a sample prepared from the mud, we do not see a noticeable difference, Cc values of these two tests are very close.

(3) M65

For the M65 mixture (Figure 2.7), the path is not completely linear. The stress at which the slope changes is around 700 kPa; for stresses < 700 kPa, the compression index is equal to Cc₁ (average) = 1.38, while the compression index for stresses > 700 kPa is equal to Cc₂ (average) = 1.11.

The swelling index does not seem to change, but is important enough, of the order of 0.16.

It can be seen that the correlation is not able to represent this behavior, but that the curves comes close in the area of high stresses above 700 kPa.
A behavior similar to that of the M65 mixture was observed in the case of montmorillonite (Figure 2.8). There is the existence of two slopes in the \((\sigma' - e)\) plane: \(C_{c1}\) (average) = 1.81 and \(C_{c2}\) (average) = 1.32, swelling index \(C_s = 0.192\).

The difference between the M65 mixture and the montmorillonite is that the stress at which the slope changes is smaller in the case of montmorillonite, at about 500 kPa. After this stress, the experimental curve and the correlation eventually tend to join.
2.1.4.2 The compressibility of claystone

For isotropic compression tests, it is noted that the behavior of claystone is nonlinear. Figure 2.9 shows an example of the results of isotropic compression tests for the first loading cycle. For each loading cycle, there is a decrease in the value of the modulus of compressibility.

With the evolution of damage, microcracks develop in the form of different families, which leads to a decrease in the modulus and a decrease in the transition threshold. This is the threshold stress corresponding to the opening or closing of a family of microcracks which is at the origin of the value of the transition from one modulus to another.

One of the best parameters to characterize the evolution of damage is the modulus of compressibility. The value of the undrained modulus in the initial state is 4700 MPa, it evolves to 2200 MPa and 2000 MPa in the first and second cycle of damage. It decreases to 1300 MPa after the third cycle.

![Isotropic compression test](image)

Figure 2.9 Isotropic compression test on a sample of Bure claystone (ANDRA, 2009)

2.2 METHODS

2.2.1 Drying of specimens for MIP and SEM

For mercury intrusion porosimetry (MIP) test as well as observations by scanning electron microscopy (SEM), the samples must be completely dry. Drying technique plays an important role in the quality of the results (Guillot et al., 2002).
The often used drying techniques are:

- Air drying in an oven;
- Evaporation of water at critical point;
- Freeze-drying (lyophilization).

The first technique can lead to considerable shrinkage of the material and it often changes the pore size distribution (Diamond, 1970; Delage and Lefebvre, 1984). In the second technique, the water is removed in the conditions of the critical point, where it is transformed into vapor (red path in the fig. 2.10). It is difficult to remove water as the critical point is at $T_c = 375^\circ C$, $P = 22$ MPa. Therefore, the critical point technique consists of two stages: progressive exchange of water by ethanol then replacement of ethanol by CO$_2$. The critical point of CO$_2$ is at $T_c = 31^\circ C$, $P = 7.6$ MPa, which is much more accessible.

Among the drying methods, we chose the freeze drying method, given the drawbacks of the other techniques and the ability of this method to preserve the structure of clays (Simms and Yanful, 2004).

Freeze-drying, also known as lyophilisation, is a dehydration process typically used to preserve a perishable material or make the material more convenient for transport. In my research, it works by freezing the clay material and then reducing the surrounding pressure to allow the frozen water in the material to sublimate directly from solid phase to gas phase (Fig. 2.10). The blue line shows these steps:

- Putting the wet material in liquid nitrogen until no air comes out;
- Sublimating ice into vacuum;
- Warming the samples under vacuum to remove all traces of water vapor.

Figure 2.10 Two techniques of drying a). lyophilization (blue) b). critical point (red) (Hammad, 2010)
Experimentally, there are four stages in the complete drying process: pretreatment, freezing, primary drying, and secondary drying.

**Pretreatment**

Pretreatment includes any method of treating the materials prior to freezing. In my test, small pieces of soil samples were taken from the original materials. The dimensions of the samples are about 2 cm × 2 cm × 2 cm.

**Freezing**

This is done by placing the soil material in a freeze-drying container which is filled with liquid nitrogen. On a larger scale, freezing is usually done using a freeze-drying machine. In this step, it is important to cool the material below its triple point, the lowest temperature at which the solid and liquid phases of the material can coexist. This ensures that sublimation rather than melting will occur in the following steps. In my research, the freezing temperatures are between -50°C and -80°C. The freezing phase is the most critical in the whole freeze-drying process, because the soil sample can be spoilt if badly done.

**Primary drying**

During the primary drying phase, the pressure is lowered (to the range of a few millibars), and enough heat is supplied to the material for the water to sublimate. The amount of heat necessary can be calculated using the sublimating molecules’ latent heat. In this initial drying phase, about 95% of the water in the material is sublimated. This phase may be slow (24 hours in my research) because, if too much heat is added, the material structure could be altered.

In this phase, pressure is controlled through the application of partial vacuum. The vacuum speeds up the sublimation, making it useful as a deliberate drying process. Furthermore, a cold condenser chamber provides a surface for the water vapor to re-solidify on. This condenser plays no role in keeping the material frozen; rather, it prevents water vapor from reaching the vacuum pump, Condenser temperature is –90°C.

**Secondary drying**

The secondary drying phase aims to remove unfrozen water molecules, since the ice was removed in the primary drying phase. This part of the freeze-drying process is governed by the material adsorption isotherms. In this phase, the temperature is increased higher than in the primary drying phase, and can even be above 0 °C, to break any physico-chemical interaction that exists between the water molecules and the frozen soil samples. Usually the pressure is also lowered in this stage to encourage desorption.

At the end of the operation, the final residual water content in the product is
extremely low, below 1%.

2.2.2 Analysis of images (orientations of particles)

The approach that we propose is based on experimental observations and consists in following the microstructural evolution in the material, especially the orientation of clay particles, as a function of its hydric state. The study consists in analyzing the microstructure using photos taken by a scanning electron microscope (SEM), coupled to an adapted method for the identification of the preferential orientations of the particles with respect to the horizontal plane. SEM observations must be carried out on completely dry specimens. The cross-sections used for the SEM observations were obtained by fracturing the freeze-dried samples. The SEM pictures were taken at various points on the observation planes. Considering, for instance, the photograph in figure 2.11 (a), the method consists in randomly extracting from this photo representative surfaces, to isolate them (Fig. 2.11(b)), to identify each particle seen from the side and in front and to draw manually a segment having the same dimension and orientation as the particle edge (Fig. 2.11(c)). Image processing of the diagram thus obtained allows the orientation of all the represented particles with respect to the X axis to be calculated and then, an angular distribution diagram to be drawn as a rose diagram, giving the percentage of side particles as a function of their orientation (Fig. 2.11(d)).

A global rose diagram is then plotted as the result of the treatment of at least 3 isolated pictures randomly taken from different observation points.
2.2.2 Mercury intrusion porosimetry (MIP)

The microscopic study is complemented by mercury intrusion porosimetry (MIP) analyses, a method which allows quantifying the pore space and characterizing the void ratio.

2.2.2.1 Introduction

The pore size distribution (PSD) obtained by MIP is an essential fabric element, which is related with some soil behavior characteristics: water, air and heat conductivity properties; adsorption and desorption isotherms (capillary phenomena); and volumetric deformation (rearrangement of fabric units). MIP data are used in my research to provide information about factors influencing fabric changes (effect of mechanical stress due to sample compaction) and fabric-properties relationships (soil water retention curves related to PSD). Predictive equations for the saturated permeability based on the pore size distribution are presented in Garcia-Bengochea et al. (1979) (capillary and hydraulic-radius models) and Juang and Holtz (1986a, 1986b)

A key assumption in mercury porosimetry is the pore shape. Essentially all instruments assume a cylindrical pore geometry using the following Washburn equation (Diamond, 1970; Juang & Holtz, 1986b; Webb & Orr, 1997) where an absolute pressure \( p \) is applied to a non-wetting liquid (mercury):

\[
p = \frac{n \sigma_{Hg} \cos \theta_{nw}}{x}
\]

(Equation 2.1)

where \( \sigma_{Hg} \) is the surface tension of mercury (\( \sigma_{Hg} = 0.484 \) N/m at 25°C), \( \theta_{nw} \) the contact angle between mercury and the pore wall, and \( x \) the entrance or throat pore diameter (\( n = 4 \)) or the entrance width between parallel plates (\( n = 2 \)). The value \( n = 4 \) is often used in MIP. The contact angle, which is very sensitive to surface roughness, is usually taken between 139° and 147° for clay minerals (Diamond 1970).

2.2.2.2 Sample preparation

The very first point in a “good” analysis is having a well defined unambiguous sample. One of the key parameters here is the sample weight. Porous materials are prone to absorb water or other chemicals, which should be removed during the initial evacuation of the sample. Mercury porosimetry has no direct way of noticing if those impurities are removed and what the actual sample weight is. We can only indirectly judge from a low vacuum pressure and the absence of any possible “leak-rates” that the sample is in a clean and well defined starting condition.

MIP equipment requires dehydrated samples measuring preferably less than 1 cm\(^3\) (limited by the sample holder and the cell stem volume). Soil samples were first prepared as slurries and then submitted to different suctions (1 kPa, 10 kPa, 20 kPa, 400 kPa, 1500 kPa and 158 MPa). Subsequently these samples were cut and freeze dried to remove the pore water and then kept in a desiccators until testing. Freeze drying process involves temperature and pressure conditions to eliminate the surface tension forces caused by air-water interfaces, and thus it is assumed that no shrinkage occurs on drying which could alter soil structure. The details of the procedure of freeze-drying are summarized in section 2.2.1.

2.2.2.3 MIP equipment and testing procedures

MIP tests were carried out on a Micromeritics Autopore IV analyzer, following the recommendations of Diamond (1970) to examine the fabric and pore structure of different soil materials. This equipment has two operating units: the macroporosity
filling apparatus from approximately 11 kPa to 155 kPa measuring apparent pore diameters from 9.5 μm to 135 μm, where the air sample is initially evacuated by applying a vacuum and surrounded subsequently by mercury; and the high pressure microporosity unit, where the mercury pressure is raised continuously between 0.17 MPa to 227 MPa (apparent pore diameters from 6.6 nm to 8.8 μm). In order to avoid the perturbation of the sample during the tests, the rate of pressure increase must be low and the equilibrium times on each step must be long enough (Diamond, 1970).

2.2.2.4 Data interpretation and analysis

(1) Pore size and total pore volume \(V_{\text{tot}}\)

Intrusion pressure values are directly converted into the corresponding pore size by using the Washburn equation. However, one should be aware that mercury porosimetry does not actually measure the internal pore size, but rather determines the largest connection (throat or pore channel) from the sample surface towards that pore. Thus, mercury porosimetry results will always show smaller pore sizes compared with Scanning Electron Microscopy (SEM), which will be verified in chapter 4.

(2) Density

A simple pycnometry type calculation allows measuring the sample density. However, the precision of those measurements is rather rough (2 to 5% error) unless special care is taken with respect to temperature control.

(3) Total surface area

Total pore surface area \(S\) is calculated by Equation 2.2:

\[
s = \frac{1}{\gamma \cos \theta} \int_0^{V_{\text{tot}}} \rho dV \quad \text{(Equation 2.2)}
\]

Total pore surface area is the area above the intrusion curve, and it is thus modeless and independent of the geometrical pore shape (Rootare & Prenzlow 1967).

(4) Mean and median pore diameters

The mean pore diameter \(d_{\text{mean}}\) is calculated by Equation 2.3:

\[
d_{\text{mean}} = 4 \cdot \frac{V_{\text{tot}}}{S} \quad \text{(Equation 2.3)}
\]

based on an assumption of cylindrical shape of pores open at ends (Emmett & Dewitt 1943). Median pore diameter \(d_{\text{median}}\) is the pore diameter at which 50% of the total intruded volume of mercury is intruded into the sample (Dees & Polderman 1981). In general, mean pore diameter emphasizes the smaller pores rather than median pore diameter.
(5) Volume pore size distribution

Volume pore size distribution, $D_v(d)$, is defined as the pore volume per unit interval of pore diameter ($d$) by Equation 2.4:

$$D_v(d) = \frac{p}{d} \cdot \frac{dv}{dp}$$

(Equation 2.4)

(Ritter & Drake 1945). Volume pore size distribution is based on a model of cylindrical pores.

2.2.2.5 Advantages and limitations of mercury porosimetry

The main limitations of MIP are that: a) isolated pores enclosed by surrounding solids are not measured – this closed porosity is often not significant in soils; b) pores that are accessible only through smaller ones (constricted porosity) are not detected until the smaller pores are penetrated; c) the apparatus may not have the capacity to enter the smallest pores of the sample (non-intruded porosity); and d) the minimum practical pressure of the apparatus limits the maximum pore size to be detected (non-detected porosity). In this way, when the clay sample is intruded by mercury, the intruded void ratio estimated under the maximum applied pressure does not coincide with the estimated void ratio of the sample. Differences mainly arise from the non-intruded porosity with entrance pore sizes lower than 10 nm and the non-detectable porosity for pore sizes larger than 400 μm.

2.2.3 Imposition of suctions by “classical” methods

The five materials (kaolin P300, M35, M50, M65 and M100) were submitted to drying under several controlled conditions of suction ($s$) starting from a saturated and structurally isotropic initial state, with an initial water content $w = w_L$. In order to produce small, medium and large suctions, three different methods were applied in this research.

For the small suctions ($0.1 \text{kPa} < s < 10 \text{kPa}$), the principle is to put the specimen in contact with a column of water through a sintered glass semi-permeable filter (i.e. permeable to water but not to air). A negative pore-water pressure (with respect to the atmospheric pressure) is imposed in the column by the difference of height between the specimen and the free end of the column, some distance below. In this research, a suction of 1kPa was applied. (Fig. 2.12)
To produce suctions in the range between 100 kPa and 1500 kPa, the osmotic method was used. First, the samples are put in dialysis membranes which are tightly sealed then they are placed in polyethylene glycol 20000 (PEG). With different density of PEG, various suctions are produced. A suction of 1500 kPa was applied in our tests. (Fig. 2.13)

For higher values of suction (158 MPa), a different method is used, which makes use of saturated salt solutions. When a saturated salt is in thermodynamic equilibrium with its vapor, the relative humidity of the surrounding atmosphere remains constant at a given temperature. The relative humidity depends on the nature of the used chemical salt (Fig. 2.14).
Once the specimens have reached equilibrium under the imposed suctions at a temperature of 20°C, they are weighed in air, then in a non-wetting oil and finally dry in order to determine their external volume and, hence, their void ratio, water content and degree of saturation.

2.2.4 Dynamic vapor sorption (DVS)

Another technique was used in the same range of suction as the saturated salt solutions, using a special equipment: Dynamic vapor sorption (Fig. 2.15 (a)). Dynamic vapor sorption (DVS) is a gravimetric technique that measures how quickly and how much of a solvent is absorbed by a sample: such as a dry powder absorbing water. It does this by varying the vapor concentration surrounding the sample and measuring the change in mass which this produces. Water vapor is most commonly used, but it is also possible to use a wide range of organic solvents. DVS was originally developed to replace the time and labor intensive desiccators and saturated salt solutions to measure water vapor sorption isotherms.

2.2.4.1 The DVS Intrinsic

The DVS intrinsic used in this study is a compact vapor sorption system which rapidly measures uptake and loss of moisture in the sample by flowing a carrier gas at a specified relative humidity (RH) and temperature over a sample suspended from the weighing mechanism of the highly sensitive and stable digital microbalance, which detects the sorption/desorption of water vapor by the increase/decrease in mass of the material. The instrument is capable of accommodating a sample mass up to 4 g and with dimensions as large as 40 mm. Changes in sample mass of 0.1 μg can be
detected, which allows precise and quick measurements using a very small sample (< 100 mg, typically). Under these conditions, each measurement step takes a few hours to complete, leaving sufficient time for the sample to become equilibrated with the chamber. Temperature stability of the measurement system is essential for accurate measurements, and this is achieved using an electronic control which ensures a temperature control to ± 0.1°C. Separate temperature-controlled zones for sample and microbalance ensure stable baseline performance, while high-precision mass flow controllers mix dry air and water in the correct proportions to provide precise RH control.

2.2.4.2 Water sorption isotherms

The main application of DVS is to measure water sorption isotherms. In general, a vapor sorption isotherm shows the equilibrium amount of vapor sorbed as a function of steady state relative vapor pressure at a constant temperature. For water sorption isotherms, relative water vapor pressure is more commonly expressed as relative humidity. In our experiments this is accomplished by exposing a sample to a series of step changes in relative humidity and monitoring the mass change as a function of time (Fig. 2.15 (b)). The sample mass must be allowed to reach gravimetric equilibrium at each step change before progressing to the next humidity level. Then, the equilibrium mass values at each relative humidity step are used to generate the isotherm. Isotherms are typically divided into two components: sorption for increasing humidity steps and desorption for decreasing humidity steps.

![DVS intrinsic](image1)  ![Typical result of DVS](image2)

Figure 2.15 Dispositif of dynamic vapor sorption (DVS)
2.2.5 Free desiccation test

In free desiccation test, appearance and propagation of cracks during drying are observed. We try to measure the maximum number of parameters. The most important are: global deformation, water content variations. Tests were carried out on the five clayey materials (kaolin P300, M35, M50, M65 and M100). In order to analyze the effect of different supports, three different supports, either smooth, rough or intermediate, were used.

2.2.5.1 Fabrication of samples

The tests are performed in an air-conditioned room with a temperature of 20°C. The relatively humidity is about $40 \pm 5\%$.

![Figure 2.16 Dimension of sample in free desiccation tests:](image)

(a) Placement of boundaries to fabricate the model; (b) Smooth support with rules; (c) Schema of model of sample; (d) Kaolin P300 on smooth support ($t = 23$ h)
In the initial state, a saturated sample of soil (with an initial water content equal to the corresponding liquid limit of the material) is prepared and then is sealed for an additional 24 hours to avoid the presence of air bubbles. Then the slurry of clay is spread on the rectangular support whose dimensions are: length 30 mm × width 20 mm × thickness 4 mm (Fig. 2.16). Its surface is made flat and finally, the boundaries (red pieces on figure 2.16(c)) are taken away in order to avoid end effects.

Homogeneity of water content of clayey materials is measured in free desiccation test. Moreover, the model is placed on a balance which is connected to a computer and the weight of the sample is continuously recorded. Global deformations are also measured in free desiccation test by means of the rules around the boundaries of the sample.

The results of these two tests are presented in chapter 3.

2.2.5.2 Roughness of the three supports

Smooth support is made of perspex. Intermediate support and rough support are made by dry sanding papers with different roughnesses.

(1) Definition of roughness

Roughness is a measure of the texture of a surface. It is quantified by the vertical deviations of a real surface from its ideal form. If these deviations are large, the surface is rough; if they are small, the surface is smooth. Rough surface usually wear more quickly and have higher friction coefficients than smooth surface. On the other hand, roughness may promote adhesion.

A roughness value can either be calculated on a profile (line) or on a surface (area). The profile roughness parameter (Ra, Rp) are more common. The area roughness parameters (Sa, Sp) give more significant values. Each of the roughness parameters is calculated using a formula for describing the surface.

(2) Measurements of roughness of the three supports

The roughnesses of these three materials were measured by a Microscope LEICA DCM3D in LEM3 laboratory. The results are shown in table 2.4. This software combines confocal imaging and interferometry. The microscope gives a three-dimensional reconstruction of the surface which is studied with a magnification of ×100.

Table 2.4 The roughness parameters Sa for these three supports

<table>
<thead>
<tr>
<th></th>
<th>Smooth support</th>
<th>Intermediate support</th>
<th>Rough support</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sa</td>
<td>8.73 nm</td>
<td>37.828 μm</td>
<td>90.359 μm</td>
</tr>
</tbody>
</table>

81
(i) Smooth support

The interested area of smooth support was chosen in order to measure the Ra and Sa (Fig. 2.17 (a)). The result can be plotted in three-dimensions (Fig. 2.17 (b)). Different colors on the surface represent different elevations of the points on the support. We can choose a profile across the sample with a black line and then the roughness on this line can be calculated automatically (Fig. 2.17 (c)). For the smooth support, the parameter Sa is 8.73 nm.

(a) Smooth support made of perspex (b) 3D surface of smooth support

(c) Average thickness on the chosen profile

Figure 2.17 Experimental process and results with “D profilometer Dual core” on smooth support
(ii) Intermediate support

The same process is applied to the intermediate support (Fig. 2.18 (a)). An area of interest is chosen and then the average thicknesses of the surface are calculated (Figs. 2.18 (b) (c) and (d)). For this support, the roughness parameter $S_a$ is $37.828 \mu m$.

Figure 2.18 Experimental process and results with “D profilometer Dual core” on intermediate support
(iii) Rough support

With the same method, the roughness of the rough support is calculated (Fig. 2.19). The value of $S_a$ is 90.359 $\mu$m which is larger than for the two other supports.

(b) Rough support made by drying sand paper

(b) 3D surface of rough support  (c) 2D surface of the rough support

(d) Average thickness on the chosen profile

Figure 2.19 Experimental process and results with “D profilometer Dual core” on intermediate support
2.2.6 Digital correlation of images

The objective of digital images correlation is to provide a better understanding of the mechanism of appearance and evolution of the cracks related to desiccation in the five materials (kaolin P300, M35, M50, M65 and M100). The approach combines a classical study of cracks (by following the morphology variation of cracks during the free desiccation) and the determination of the local deformations and displacements during drying. For this purpose, photos of the soil surface are taken at regular intervals during free desiccation tests and analyzed by correlation of digital images, in order to identify at an early stage the conditions leading eventually to the formation of cracks. The experimental device is presented in figure 2.20.

There are various systems which can perform digital images correlation, for example Correli\textsuperscript{Q4} which was developed by Hild and Roux (2008), or Vic-2D and Vic-3D industrial softwares which are presented in detail. Both Correli\textsuperscript{Q4} and Vic-2D systems were tested in the framework of our study, aiming to compare the obtained results and then to select the most suitable for our clayey materials.
2.2.6.1 Vic-2D and Vic-3D

(1) 2D digital images correlation

VIC-2D is a system that uses the digital image correlation technique to make strain measurements. This system is able to provide two-dimensional strain maps of any entire planar specimen. The equipment consists of computer software, lighting device and a digital camera (Figure 2.21) with appropriate lens and resolution.

The digital image correlation consists in capturing a series of pictures of a specimen during the deformation process with a digital camera. With computer software the images are analyzed to create a contour map of strains. The specimen needs to be prepared with a random dot pattern (speckled pattern) to permit that the software be able to calculate the displacements of the points. During the tests, while the specimen is subjected to external loads, the camera takes one picture before and others after deformation, and the software analyzes the difference between the pixels of the different images and correlates them to derive the displacement field and then the strain map.

Figure 2.21 The digital camera used with VIC-2D

The image is an array of integers (here pixel 100 = white, pixel 0 = black).
The surface of the object moves 1 pixel up, 1 pixel to the right:

If we define a “small image” of 5×5 pixels in the reference image before moving it, how will we find the new location after the move?

The displacement of the points is then calculated with reference to the center of the selected area.

(2) 3D digital images correlation

Preparation of the specimen is begun by preparing the region of interest on the sample with a speckle pattern. And then, two cameras are set up on the specimen. To begin, the prepared specimen is set in its testing location, with special care being taken of the camera orientation and placement. The tested specimen for this example is kaolin P300 on the smooth support (Fig. 2.22). A balance is placed under the sample to measure its weight.
Figure 2.22 Photos of VIC-3D and the soil sample

Much like human vision, two imaging sensors provide enough information to perceive the environment in three-dimensions (Fig. 2.23).

Figure 2.23 The digital cameras used with VIC-3D

Recovering the three-dimensional structure of the environment using two imaging sensors is called stereo-triangulation.

Figure 2.24 Schema of stereo-triangulation  Figure 2.25 Calibration of the system
Stereo-triangulation requires computing the intersection of two optical rays (Fig. 2.24). It is feasible only if these rays are formulated in a common coordinate system. With this device, it is necessary to model and calibrate the stereo-rig (Fig. 2.25). The calibration procedure calculates variables related to the camera geometry and imaging; it is a very important step in the use of VIC-3D.

The measurement images are saved automatically. The image below illustrates the user interface (Fig. 2.26).

![Figure 2.26 Menu of VIC-3D](image)

With Vic-3D, the outputs are:

- **U [mm]** - displacement along the X-axis, from the reference image. For the reference image, displacement values will always be 0.
- **V [mm]** - displacement along the Y-axis.
- **Z [mm]** - displacement along the Z-axis.

Strain variables are also generated in post-processing:

- **exx [%]** - strain in the X-direction. Positive numbers indicate tension; negative numbers indicate compression.
- **eyy [%]** – strain in the Y-direction.
exy [%] – shear strain.

For VIC-2D, the results include U, V, $e_{xx}$, $e_{yy}$ and $e_{xy}$.

2.2.6.2 CorreliQ4

The technique developed in this software is based upon a multi-scale approach to determine “finite element” displacement fields by digital image correlation. The displacement field is first estimated on a coarse resolution image and progressively finer details are introduced in the analysis as the displacements are more and more securely and accurately determined. Such a scheme has been developed to increase the robustness, accuracy and reliability of the image matching algorithm. CorreliQ4 is implemented in MATLAB™. An example of output of CorreliQ4 is presented in figure 2.27 for kaolin P300 during desiccation.

![Image of CorreliQ4 results](image)

Figure 2.27 Visualization of transversal strains $\varepsilon_{11}$

The five outputs of CorreliQ4 are:

Displacement 1 (pixel) - displacement along the Y-axis, from the reference image. For the reference image, displacement values will always be 0.

Displacement 2 (pixel) - displacement along the X-axis.

Strain 11 [1] - strain in the Y-direction. Positive numbers indicate tension; negative numbers indicate compression;

Strain 22 [1] – strain in the X-direction;

The limitation of Correli\textsuperscript{Q4} is that when the displacements are too large, especially after the appearance of the cracks, the calculations are not accurate. In the following section 2.2.6.4, the calculations of Correli\textsuperscript{Q4} will be compared with those of Vic-2D.

2.2.6.3 Calculation of principal deformations (2D)

For each time considered in the analysis of the behavior of the material, there are 5 different maps (2 displacement and 3 strain maps) in the above results. It is not very direct and practical to observe and analyze the deformation with all those maps. Therefore, we derived from the strain maps a principal deformation map (Fig. 2.28).

![Figure 2.28 Circle of Mohr](image)

Figure 2.28 Circle of Mohr

With the results of the Vic-2D, \( \varepsilon_{11}, \varepsilon_{12} \) and \( \varepsilon_{22} \) are known. The major and minor principal stresses \( \varepsilon_1, \varepsilon_2 \), and the angle of the major principal stress with respect to the X axis, \( \theta \), can be calculated with the following equations (Equations 2.5-2.7):

\[
\varepsilon_1 = \frac{\varepsilon_{11} + \varepsilon_{22}}{2} + \sqrt{\left(\frac{\varepsilon_{11} - \varepsilon_{22}}{2}\right)^2 + \varepsilon_{12}^2} \quad \text{(Equation 2.5)}
\]

\[
\varepsilon_2 = \frac{\varepsilon_{11} + \varepsilon_{22}}{2} - \sqrt{\left(\frac{\varepsilon_{11} - \varepsilon_{22}}{2}\right)^2 + \varepsilon_{12}^2} \quad \text{(Equation 2.6)}
\]

\[
\theta = \frac{1}{2} \arctg \frac{2\varepsilon_{12}}{\varepsilon_{11} - \varepsilon_{22}} \quad \text{(Equation 2.7)}
\]

The data of \( \varepsilon_{11}, \varepsilon_{12} \) and \( \varepsilon_{22} \) are matrices with the same dimensions (for example, 358 values in the transversal direction and 230 values in the axial direction for kaolin P300, t = 1h). Because \( \varepsilon_1, \varepsilon_2, \) and \( \theta \) correspond to each value of \( \varepsilon_{11}, \varepsilon_{12} \) and \( \varepsilon_{22}, \varepsilon_1, \varepsilon_2 \) or \( \theta \) are also matrices with the same dimension (358×230).
With the values of $\varepsilon_1$, $\varepsilon_2$ and $\theta$, tensors of local principal deformations are calculated by a program based on MatLab (Fig. 2.29).

![Figure 2.29 Schema of the principal deformation](image)

In order to minimize the number of tensors and make the schema of the principal deformations clearer, average calculations are done. For kaolin P300 with the smooth support, there are $36 \times 24$ tensors in the schema. The red color means that the values of principal strains are positive (extensions) while the blue color means they are negative (compressions).

2.2.6.4 Two examples of comparison of the results of Correl\textsuperscript{Q4} and Vic-2D

(1) *Simulation test with rubber without an angle on the support*

In order to imitate natural desiccation process and verify the validity of the results of Correl\textsuperscript{Q4} and Vic-2D, tests with rubber were carried out (Fig. 2.30). The left boundary of the rubber model is fixed on the support and the right boundary is mobile. The initial dimension of the rubber is 300 mm (length) $\times$ 300 mm (width). The deformed image has nearly the same width and a smaller length which imitates the shrinkage of soil. The maximum displacement of the boundary is about 5 mm.
Calculations between reference image and deformed image are carried out with Correli$^Q4$ and Vic-2D, respectively. As stated before, with Correli$^Q4$ and VIC-2D, we can obtain the components of the displacement in the transversal (V) and longitudinal (U) directions and, from that, derive the 3 components of the strain tensor: longitudinal $\varepsilon_{11}$, transversal $\varepsilon_{22}$ and shear $\varepsilon_{12}$. In this example, one would expect a longitudinal displacement becoming more and more negative from left to right, a transversal displacement corresponding to the contraction of the model and quasi-homogeneous strains. The results are shown in figure 2.31.

![Figure 2.30 Simulation test with rubber](image)

(a) Longitudinal displacement (Correli$^Q4$)  (f) Longitudinal displacement (Vic-2D)

Figure 2.31 Comparison of the results of Correli$^Q4$ and Vic-2D
(b) Transversal displacement (Correli\textsuperscript{Q4})

(c) Longitudinal strain (Correli\textsuperscript{Q4})

(d) Transversal strain (Correli\textsuperscript{Q4})

(g) Transversal displacement (Vic-2D)

(h) Longitudinal strain (Vic-2D)

(i) Transversal strain (Vic-2D)
As seen in Figure 2.31 (f), at the left boundary of the sample, the longitudinal displacement $U$ is -0.55 mm. $U$ increases progressively from left to right side. At the right boundary of the sample, maximum $U$ is -7.6 mm. Figure 2.31 (g) shows transversal displacement $V$. At the upper and lower side of the rubber, $V$ is 1.81 mm and -0.32 mm, respectively. This is due to the contraction of the model. The results of longitudinal strains $\varepsilon_{11}$, transversal strains $\varepsilon_{22}$ and shear strains $\varepsilon_{12}$ are presented in Figures 2.31 (h) (i) and (j). In some areas of the left and right boundaries of the model, $\varepsilon_{11}$ is with extreme values. This is caused by the boundary effect of the model. Inside the model, $\varepsilon_{11}$ is quasi-homogeneous which is between -1.25% and -3.17%. This means that nearly all the areas are in compression in the rubber during shrinkage. Transversal strains $\varepsilon_{22}$ are relatively smaller than $\varepsilon_{11}$. Shear strains $\varepsilon_{12}$ are nearly 0 in the inner. The results correspond well with our expectations before the test. It is concluded that the results deduced by Vic-2D are more reliable to interpret free desiccation tests.

In another aspect, the results with CorrqiQ4 do not appear to be very accurate because of the large deformations (Figs. 2.31 (a) to (e)) which are difficult to take into account with this software. There are some areas in the surface where the software cannot calculate. At the left side where the displacements are larger, the calculations are not accurate. The results with Vic-2D are more reasonable.

(2) Simulation test with rubber with an elevation of the support

In free desiccation tests, there is an “uplift” phenomenon in some materials on some supports, especially with mixtures on intermediate and rough supports. In this case, the following problem we tried to solve is whether the uplift will influence calculation results?
In order to verify the validity of the results when uplift happens, tests were still carried out with rubber. At the same time, the right side of the support is elevated in order to get an angle which imitates the “uplift” phenomenon. In this test, the angle equals to 2°.

The results with Vic-2D are more reasonable as analyzed in the previous sections (Figs. 2.32 (a) to (e)). For longitudinal displacement U, they are all negative, which means the direction of U is towards left. At the right side of the boundary, U equals to -9.5 mm and at the left boundary, maximum U is -3.5 mm. Transversal displacement V are smaller compared with U. These results correspond with the process of the simulation of rubber.

For longitudinal strains $\varepsilon_{11}$, in this example, the maximum and minimum values are 0.54% and -4.14%, respectively. This means that nearly all the areas are in compression (negative) in the rubber during shrinkage. The maximum and minimum transversal strains are 2.9% and -0.12%, which are smaller than $\varepsilon_{11}$.

It is concluded that the results with Vic-2D are more reasonable and more accurate. All the digital images correlation analyses in my research were carried out with Vic-2D.

![Figure 2.32 Comparison of the results of Correli and Vic-2D](image)

(a) Longitudinal displacement (Vic-2D)  (b) Transversal displacement (Vic-2D)

Figure 2.32 Comparison of the results of Correli and Vic-2D
2.2.7 Analysis of the cracks

The software Image J can perform image processing on each picture during a test (see eg Fig. 2.33). It allows in particular the differentiation of cracks by a color code, which makes it possible to determine morphological parameters: the average width and length of cracks, the total area of the cracks. It is noted that the ratio of cracks is defined as the ratio between total surfaces of cracks and total surface of the soil sample.
2.2.7.1 Treatment of the images with software Image-J

Using the software Image-J, a delimitation of the studied area of all the processed photos is carried out, which permits to always have the same initial surface, then the binarization of the image is performed (Fig. 2.33). This operation consists in turning a color photo into black and white. Good contrast between the cracks and uncracked areas is necessary for automatic processing. However, it is preferable to divide this large area into smaller areas in order to increase the accuracy of the determined parameters. This work is all the more difficult as the contrast between the cracks and uncracked area is low. But thanks to the huge capacity of photos, we have reliable results and it is not necessary to use manual methods to redraw the network of cracks using Photoshop software.

2.2.7.2 Analysis of parameters and results with the use of Image-J

A program is written in the background of Image-J. The three following parameters are studied: 1. The total length of cracks (l); 2. The average width of all the cracks (d); 3. The cumulative surfaces of cracks (s).

Running the program under the background of Image-J, the numbers, the surfaces (s) and the perimeters of the cracks (L) are calculated automatically. The length of the crack is calculated as the half-perimeter of the crack (l = L/2). The average width of a crack is the ratio between its surface and length (d = S/l).

The following stages show the different steps in the process of Image-J:

Fig. 2.33 (a): Cut a small area (for example, with the dimension of length 300 mm × width 200 mm) to eliminate the boundaries.

Fig. 2.33 (b): Change the colors of background and cracks and then correct lighting; Make the thresholding in two colors: black and white.

Fig. 2.33 (c) (d): Perimeter of every discontinue crack is measured automatically.

Fig. 2.33 (e): The values of perimeter of every crack and number of cracks are obtained.
Figure 2.33 Process of analysis of the parameters of cracks with Image-J
2.2.8 Traction tests

The appearance and formation of cracks is strongly related to the local tensile strength of the soil. More generally, it is important to know the response of the materials on some stress paths leading to the formation of cracks, traction being only one of the simplest of them. In practice, the few measures which have been carried out are essentially measures of tension or bending.

Avila (2004) presented in his thesis an example of experimental device for the direct measurement of tensile strength of clays at different scales. The specimens have a double triangular shape (Fig. 2.34). The mold is split at the narrowed portion where the displacement can be measured. In the research of Avila, the tests are done with either controlled load or controlled deformation.

Figure 2.34 Device to measure the tensile strength of soils (Avila 2004)

2.2.8.1 Experimental device developed in my thesis

The experimental device developed in my thesis has a double triangular shape, just like Avila’s, except that it is built in plastic. The mold is also split in the narrowed portion where the displacement is measured by a sensor which is connected to a computer. The right-hand side triangular sample is fixed to the support of the device, while the left-hand side sample can move on a roller with reduced friction. The dimensions of the sample in the narrow center of the sample have a length × width× height equal to 40 mm×20 mm×40 mm. The maximum displacement for the traction test is 40 mm (Fig. 2.35). The traction is achieved by a weight of sand which is
progressively increased by adding sand and measured by a force transducer during the test. The equations are the following, assuming that the strains and stresses are homogeneous in the central part of the sample (Fig. 2.35):

\[ \sigma = \frac{F}{S_0} \]  
\[ \varepsilon = \frac{\Delta l}{l_0} \]

(Equation 2.8)  
(Equation 2.9)

where: \( \sigma \) is traction;
\( F \) is the load measured during the tests;
\( S_0 \) is the surface of section of the traction sample (20 mm×40 mm);
\( \varepsilon \) is the deformation of the sample;
\( \Delta l \) is the displacement during the traction test which can be measured automatically by the sensor;
\( l_0 \) is the original length of the traction sample (20 mm).

Previously to the tests, the friction of the rollers was measured in order to correct the applied forces. Traction tests with three different materials (kaolin P300, M35 and montmorillonite) were carried out with this device. The materials have different initial water contents and obviously their tensile strength is different. In figure 2.36, a test with montmorillonite is shown. For the different materials, the stress-strain relations are analyzed at different water contents.
Figure 2.36. Experimental device for traction tests: (a) before traction; (b) after traction; (c) the sample for traction.
CHAPTER 3 MACROSCOPIC RESULTS

This chapter is devoted to the presentation of 5 subjects which play a major part in the formation of cracks:

- the determination of the water retention characteristics which permit to relate the changes in water content, volume and degree of saturation to the variations of suction in the soil on drying and wetting paths,

- the changes in the local and global water content of the soils for a flat rectangular sample submitted to drying by evaporation,

- the local displacements and strains, derived from digital correlation of images, for the flat rectangular sample,

- the characteristics of the cracks and their evolution with time,

- and the tensile strength of the different mixtures of clay obtained by direct traction tests on specimens of the soils.

All the experiments performed on claystones at macro and micro scales will be presented in chapter 5.

3.1 DRYING-WETTING PATHS OF THE MATERIALS

The understanding of water retention features is an essential aspect for the behavior of saturated and unsaturated soils. It is known that the capillary properties of a soil are related to the pore volume; that is, the volume available for the fluid phases. The volume of voids in a soil depends on the grain size distribution and on the state of compaction of the medium. The process of drying or wetting is regarded as an additional source of deformation, indicating that the density of the soil can be strongly responsive to suction variations (Romero 1999; Fleureau et al. 2002; Olchitsky 2002).

The formulation of a general purpose water retention model should therefore account for the evolution of density or void ratio along drying and wetting paths. In most of the published articles (Gallipoli et al. 2003; Mbonimpa et al. 2006; Nuth and Laloui 2008; Masin 2010), the degree of saturation is linked to the void ratio through simple relationships that proved to be effective in the range of suction where capillarity predominates (low to moderate suctions). The following approach is used in this thesis:

Water retention characteristics were derived both from the classical methods and
from Digital Vapor Sorption (DVS) tests performed on the different clay mixtures.

3.1.1 DVS tests

Dynamic Vapor Sorption (DVS) tests are performed using an automated device which plots the sorption/desorption curves by measuring the change in mass of the sample subjected to controlled conditions of relative humidity and temperature.

3.1.1.1 Material preparation

The same material preparation procedure was followed for most of the tested materials except the M35 mixture which was prepared as powder. The dry powder of soil is mixed with de-aired water at a gravimetric water content equal to the liquid limit. The resulting slurry is then vigorously mixed and vibrated for several minutes to remove air bubbles. Following this technique, a good homogeneity and nearly 100% saturation are initially achieved. Before use, the soil is left to settle for at least 24 hours to ensure homogenization of the water content. This preparation enables the soil to be as close as possible to a virgin mechanical and saturation state. Moreover, it prevents formation of any initial soil structure, such as particle aggregates.

3.1.1.2 Experimental process

During the experimental process of DVS, different specified relative humidities (RH) are imposed to the sample suspended to the weighing mechanism of a highly sensitive and stable digital microbalance, leading to an uptake or loss of moisture in the sample. These controlled humidities are obtained by flowing a water-carrying gas in the measurement chamber. In our experiments, during drying, the relative humidity is decreased from 95 % to 0 %, while during wetting, the relative humidity is increased progressively from 0 % to 95 %.

The DVS instrument is capable of accommodating a sample with a mass up to 4 g and with dimensions as large as 40 mm. Changes in sample mass of 0.1 μg can be measured by the microbalance in the instrument. The results for the different clay mixtures, showing the variation of the weight of the sample, as well as those of the imposed and measured relative humidities, versus time, are shown in figure 3.1.
Figure 3.1 Weight of the samples and relative humidity versus time for the 5 materials

(a) kaolin P300

(b) M35 mixture
(c) M50 mixture

(d) M65 mixture

Figure 3.1 Weight of the samples and relative humidity versus time for the 5 materials
3.1.1.3 Drying/wetting paths of the tested materials

The drying and wetting paths of kaolin P300, M35, M50, M65 mixtures and montmorillonite M100 are shown in figure 3.2 which presents the water content versus suction curve. With the DVS method, the range of suctions is much higher than in the case of the usual measurements methods, where it is between 0.1 kPa and 700 MPa, much larger than the shrinkage limit suction. In the large suctions domain (10000 kPa to 1000000 kPa), these results can be compared with those of the classical tests using saturated salt solutions. In both cases, the five materials were initially prepared as slurries with water content \( w_i = w_L \).

Wetting path presents several domains which correspond to those of the drying paths. The effect of mineralogy on the behavior of the materials is very significant.

Under the high suction, the water contents of the soil samples are very small. When \( w \) reaches the maximum suction (1000 MPa), the water content is equal to 0%. Generally, the water content during drying is higher than during wetting under the same suction.

For kaolin P300, the water contents are smaller than those for the other materials under the same suction. During the drying process, under the suction of 7000 kPa, the water content is 3%. The difference between the water content during drying and
wetting is very small compared with the other materials so that the drying/wetting path appears just one line in figure 3.2.

For the M35 mixture, as indicated above, unlike the other materials, it was prepared as powder. During the wetting/drying circle, the range of water contents is much larger than that of kaolin P300. During the drying process, for the low suction (7000 kPa), the water content is 10% which is the same as during wetting. For the high suction, the water content is 0%.

For the M50 mixture, the range of water content is between 15% and 18% during drying and wetting, which is not different from the M65 mixture. For M65, the water content equals 17.5% and 21% during drying and wetting.

For M100, the range of water content is much larger than for the other materials, it reaches 27% and 30% during drying and wetting.

The drying-wetting paths are nearly reversible in the case of kaolin P300 and M35. For M50, M65 and M100, under the same suction, water content during drying is much higher than during wetting. In figure 3.3, the DVS results of these 5 materials are connected with the point of original water content \( w_L \). It is clear that the results of DVS are observed in higher suctions domain.

![Figure 3.2 Drying/wetting paths of kaolin P300, M35, M50, M65 and M100 derived from DVS tests in the \([s, w]\) coordinate system](image)

Figure 3.2 Drying/wetting paths of kaolin P300, M35, M50, M65 and M100 derived from DVS tests in the \([s, w]\) coordinate system
3.1.1.4 Equilibrium times and permeability

At the beginning of the DVS test, the original state of the soil samples is slurry with a water content equal to the liquid limit, except for M35 (soil powder). For kaolin P300, M35, M50, M65 and M100, the liquid limit increases with the montmorillonite percentage. The time for which the weight of the samples stabilizes also increases. The samples have more or less the same shape and the same weight, so that the stabilization time is representative of the permeability of the soil.

Weight of the samples versus time for these 5 materials is analyzed during the drying/wetting process. For a given relative humidity, in the beginning of drying or wetting, the weight of the samples increases or decreases with time, more or less linearly versus the logarithm of time. This phase can be called “primary drying or wetting phase” Then, the weight stabilizes. In fact, during that phase (“secondary phase of drying or wetting”, the weight of the sample continues to increase or decrease, but much more slowly than during the first phase. This phenomenon is illustrated in figure 3.4 for the relative humidity of 40%.

For kaolin P300, the times for which the weight of the sample stabilizes during wetting and drying are not so different, 0.18 h and 0.3 h respectively. In the case of M35, the stabilization times are between 0.41 h and 0.43 h, a little larger than those of

Figure 3.3 Drying/wetting paths of kaolin P300, M35, M50, M65 and M100 derived from DVS tests connected with the initial water content (w_L)
kaolin P300. For M50, during wetting and drying, the stabilization times are 0.53 h and 0.8 h respectively. The stabilization times of M65 during drying and wetting are the same, i.e. 0.95 h. For M100, just as for M65, the stabilization times are also the same, 1 h, during drying and wetting.

Figure 3.4 Weight of the samples versus time for the 5 materials (DVS)
At the end of the primary phase, the stabilization time is plotted versus the percentage of montmorillonite (Fig. 3.5). For the same material, the stabilization times during drying and wetting are quite similar. With the increase in the percentage of montmorillonite, the stabilization time increases, but it seems to reach a maximum for montmorillonite contents larger than 60%.
3.1.2 Drying paths of the materials using the classical methods

The drying paths of the slurries of kaolin P300, M35, M50, M65 mixtures and M100 were also determined by means of the classical methods (tensiometric plates, osmosis, salt solutions) For each measurement point, four samples were used to obtain the state parameters. Drying paths present several domains (Fleureau et al. 1993; Péron et al. 2006): (i) a first domain in which the soil remains saturated up to the air entry suction ($s_{DL}$). In this domain, the material exhibits the major part of its deformation and, in the [$w$ - $e$] plane, the curve follows the saturation line: $e = \gamma_s/\gamma_w w$; (ii) a second domain where desaturation happens but remains limited. Usually, the degree of saturation is larger than 80% during this phase whereas deformation of the soil goes on following the same trend as before; (iii) a third phase during which the deformation becomes smaller, especially if the soil is not very plastic, but where the degree of saturation decreases very quickly down to 0.

3.1.2.1 $w$ – $e$ curve

The relation between water content, degree of saturation and void ratio is the following one:

$$e = \frac{G_s}{S_v} \cdot w$$  \hspace{1cm} (Equation 3.1)

The grain densities ($G_s$) of kaolin P300, M35, M50, M65 and M100 are 2.65, 2.67, 2.69, 2.70 and 2.73 respectively (Figure 3.6).
For kaolin P300, the shrinkage limit water content ($w_{SL}$), defined by the intersection between the saturation line and the horizontal line for $w = 0\%$, is 16\%. For the M35 mixture, the range of void ratios is much larger than that of kaolin P300. For the lowest suction, the void ratio is 2.05 under the suction of 1 kPa, compared to 1.05 in the case of kaolin P300. The shrinkage limit water content is 21\%. The range of void ratios of the M50 mixture is much larger than that of M35, between 0.45 and 2.77. The shrinkage limit water content (24\%) is larger than that of kaolin P300 and M35. For the M65 mixture, the range of void ratios is much larger than those of kaolin P300, M35 and M50. For the lowest suction, the void ratio is 3.03, compared to 1.05 in the case of kaolinite. When suction increases, the void ratio decreases until it reaches 0.33 for the suction of $10^6$ kPa. The shrinkage limit water content of M65 mixture is 30\%. For the M100, the range of void ratios is much larger than those of the 4 other materials. For the lowest suction, the void ratio is 5.14. When suction increases, the void ratio decreases until it reaches 0.36 for the suction of $10^6$ kPa. The shrinkage limit water content of M100 is 41\%.

Corresponding to each value of the shrinkage limit water content ($w_{SL}$), there is a shrinkage limit void ratio $e_{SL}$ which increases with the montmorillonite percentage (Figure 3.7). For kaolin P300, $e_{SL}$ is 0.3, while for M35, it reaches 0.5. The difference of $e_{SL}$ between M35 and M50 is not large, for M50, $e_{SL}$ equals 0.6. For M65 and M100, the void ratios corresponding to the shrinkage limit water content are 0.8 and 1.1, respectively.

![Figure 3.6 Water content versus global void ratio](image.png)
As shown in figure 3.8, just like $e_{SL}$, the shrinkage limit water content also increases with the percentage of montmorillonite. The effect of montmorillonite on the behavior of the materials is very significant.

It is possible to use the correlations established by Biarez and Favre (1977) for oedometric tests between the Atterberg limits and the void ratios to define the drying path of a slurry:

$$e = e_L = G_s w_L \text{ when } \sigma' = 7 \text{ kPa } (G = \gamma_s / \gamma_w)$$  \hspace{1cm} (Equation 3.2)

$$e = e_p = G_s w_p \text{ when } \sigma'_v = 1000 \text{ kPa}$$  \hspace{1cm} (Equation 3.3)

Similarly, we can link the maximum level of shrinkage to the liquid limit using the correlations of Kheirbek-Saoud et al. (2007) based on experimental results, with the following parameters:

$$\delta e = e_L - e_{SL} \text{ and } e_v = \delta e / (1 + e_L)$$  \hspace{1cm} (Equation 3.4)

The correlation is shown in figure 3.9. It was found that the correlation between $w_L$ and the first parameter is good ($R^2=0.96$), although the number of points is reduced. It is deduced that the parameters are well defined and intrinsic characteristics of real soil behavior. All this allows us to offer a simple model of the drying curve of a clay from the mere knowledge of the liquid limit, which represent the limit behavior of these materials in a relatively realistic way.

With the data of these 5 materials, the correlation between the liquid limit and $\delta e$ was modified. As seen in figure 3.9, the data correspond well with the correlation of Kheirbek-Saoud et al. (2007).
3.1.2.2 $s - e$ curve

In the thesis of Hammad (2010), oedometric tests were done on the same materials as in my research. The soil samples were pre-consolidated in a consolidometer under an effective up to $\sigma'_v = 100$ kPa. The compression indexes (Cc) were calculated. For the kaolin P300, M35, M65 and M100, the Cc are 0.241, 0.65, 1.11 and 1.32, respectively.

Using the same method, compression indexes are derived from my data. For the kaolin P300, M35, M50, M65 and M100, the Cc are 0.246, 0.605, 0.759, 0.952 and 1.394 (Fig. 3.10). The results are similar to those of Hammad (2010). Cc increases with the percentage of montmorillonite. For kaolin P300, the compression index has the smallest value whereas, for M100, the parameter is the largest among all the materials. The effect of montmorillonite is significant in the behavior of the materials.

![Figure 3.9 Correlation between the liquid limit and the maximum change in void ratio during drying (with my data)](image.png)
Figure 3.10 Suction versus void ratio for the 5 materials
The evolution of the compression coefficient versus the percentage of montmorillonite, plotted in figure 3.11, has an almost linear form.

Figure 3.11 Evolution of Cc and Cs versus percentage of montmorillonite; comparison with the results of Hammad (2010)

For these 5 materials, with the increase of suction, the void ratio progressively decreases (Fig. 3.12). For kaolin P300, when the suction exceeds 1500 kPa, the specimens become unsaturated. The total volumetric strain between 1 kPa and 158 MPa reaches 35%. For the M65 mixture, the air entry value is close to $10^4$ kPa. For M100, the total volumetric strain between 1 kPa and 158 MPa reaches 65%.

These values can be compared with those derived from MIP as we will see in Chapter 4.
3.1.2.3 $s – w$ curve

For the 5 materials, with the increase of suction, water content progressively decreases (Fig. 3.13). The global measurement concentrates in the domain of 1 kPa and 1500 kPa, while the DVS is more precise for suctions larger than 7000 kPa. Globally, the two sets of results correspond well.

The shrinkage limit water content can also be expressed in terms of suction, the shrinkage limit suction, which is a very important parameter of the behavior of unsaturated soils as it corresponds to a drastic change in their properties. The “shrinkage limit suctions” for the 5 studied materials are 1.3 MPa, 1.9 MPa, 2.3 MPa, 4.1 MPa and 5 MPa, respectively (Fig. 3.14).

With the increase in the percentage of montmorillonite, the shrinkage limit suction increases too (Fig. 3.15). The shrinkage limit suctions of M65 and M100 are much larger than those of the 3 other materials. The water content of montmorillonite is variable and the material increases greatly in volume when it absorbs water. Because of the expansive characteristics of montmorillonite, the shrinkage limit suction increases greatly in M65 and M100, which both contain more montmorillonite than the other soils.

![Figure 3.14 Water content versus suction](image-url)
Figure 3.13 Water content versus suction relationship including the data of DVS
3.1.2.4 $s - Sr$ curve

Desaturation suction (air entry pressure) is deduced from the relation between suction and degree of saturation (Fig. 3.16).

At the beginning of drying path, for these 5 materials, the degree of saturation remains equal to 1. With the increase of suction, the degree of saturation decreases, the soil samples become unsaturated. Desaturation suction increases with the percentage of montmorillonite but tends towards a plateau when this percentage reaches 65% (Fig. 3.17).

For these 5 materials, the desaturation suction is 1.2 MPa, 39 MPa, 77 MPa, 110 MPa and 113 MPa, respectively.

The relationship between the diameter of the curvature of the meniscus spherical water – air in the tube and the difference between the air and water pressure is given by the law of Laplace which simplifies in the assumption of cylindrical pores and takes the expression of the law of Laplace:

$$u_a - u_w = \frac{2\sigma_s \cos \theta}{r}$$  \hspace{1cm} (Equation 3.5)

where: $u_a$ and $u_w$ is the pore air pressure and pore water pressure, respectively;

$\sigma_s$ is the tension in the water – air surface;

$\theta$ is the contact angle between the meniscus and the solid;

$r$ is the diameter of the pores.

For water, $\sigma_s = 72.75 \times 10^{-3}$ N/m and $\cos \theta = 1.$
In this test, it is assumed that $u_a - u_w = s$ (desaturation suction). It is induced that $r = \frac{4\sigma_s}{s}$.

The desaturation suction may be representative of the predominant pore diameter in a material \cite{Delage and Cui, 2000}. For kaolin P300, M35 and M65, with suctions of 1.2 kPa, 39 MPa and 100 MPa, the diameters of the pores derived from Laplace equation are equal to 0.25, 0.01, 0.003 μm respectively. The main diameters deduced from the suction-controlled tests will be compared with those derived from the mercury intrusion porosimetry tests in Chapter 4.

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Figure 3.16 Degree of saturation versus suction

Figure 3.17 Air entry suction versus percentage of montmorillonite
3.1.2.5 Comparisons of the results of kaolin P300 and montmorillonite with those of the literature

In the thesis of Taibi (1993) and Soemitro (1993), drying/wetting paths were also determined on kaolin P300 and montmorillonite. My results are compared with those of Taibi and Soemitro. The initial state of the samples (slurries) is the same in all the tests.

In the $w - e$ plane, the shrinkage water contents of kaolin P300 are quite similar, about 23% (Fig. 3.18). At the end of the drying path, the void ratio is slightly larger than that of Taibi. Concerning the $s - e$ curve, at the beginning of the drying path, the void ratio ($e = 0.96$) is a little smaller than that of Taibi ($e = 1.2$). The shrinkage limit suction is nearly the same, about 1300 kPa. At the end of the drying path, the void ratio is a little higher than that of Taibi. Regarding the $s - w$ curve, the initial water content in my test is smaller, about 37% while at the end of the drying path, it turns to be larger. The air entry pressure in my test is similar to that of Taibi, which is about 1900 kPa.

Generally, in the case of kaolin P300, the differences between my results and those of Taibi are not obvious, except under high suctions.

![Figure 3.18 Drying path of kaolin P300](image-url)
For the results of montmorillonite, at the beginning of drying path, the void ratio in my test ($e = 6$) is a little smaller than that of Soemitro ($e = 7.3$), just like in the test with kaolin P300 (Fig. 3.19). For the $s – w$ curve, at the end of the drying path, the water content and saturation degree are larger than those of Soemitro.

Just as in the case of kaolin P300, differences focus on the results under high suctions. It is assumed that there are mistakes during the measurement of void ratios.

Figure 3.18 Drying path of kaolin P300

Figure 3.19 Drying path of montmorillonite
EXPERIMENTS OF FREE DESICCATION

As stated in Chapter 2, free desiccation tests of the different materials were carried out on different supports. The initial water contents of kaolin P300, M35, M50, M65 mixtures and montmorillonite M100 were equal to \( w_L \). The results of free desiccation tests with kaolin P300 and M50 are presented in this section. These tests were devoted to the determination of the average water contents, local water contents and deformation evolution. The average gravimetric water contents change with time. They are analyzed in two different aspects: homogeneity and global value.

3.2.1 Homogeneity of water content in free desiccation test (kaolin P300)

The tests are carried out with kaolin P300 starting from a saturated and structurally isotropic initial state. In order to saturate the samples, de-aired water is mixed with the soil powder to make slurries with an initial water content \( w = w_L \) (40\%). Then the samples are carefully sealed in a plastic bag for about 24 hours before imposing on the surface of the plastic support. The dimension of the samples is: 300 mm × 200 mm × 4 mm (length × width × thickness).

As shown in figures 3.20 and 3.21, the local water contents are measured using the following experimental procedure: the samples are prepared by making the surface flat with a model. Then local small samples are cut in different parts of the surface and the water content of each sample is measured by drying in the oven. The dimension of the small samples is about 40 mm × 40 mm. There are about 5 measurements in the length and 4 measurements in the width at the same time.
The evolution of the gravimetric water content distribution along the first row plotted at different drying times is shown in figure 3.22. As the soil sample dries, water content heterogeneity increases for the first 20 hours, before the difference between the center and the extremities reaches 1%, which amounts to 20% of the initial value. Drying tends to progress from the extremities towards the center. After 20 hours, water content becomes nearly uniform at the level of 23%. No appreciable water content difference was revealed between the upper and the lower part of the drying sample. An additional measurement was also performed on the second row of the soil sample. It was used to make comparisons of water content distribution. Results show that the water content difference between the center and the extremities still exists, and is more pronounced (3%).

For the measurements of the first row of the soil sample, when the soil dries, the heterogeneity of water content increases in the first 4 hours. The maximum difference between the center and the ends reaches 1.46% (considering all the measurements).
According to figure 3.23, for the measurements of the second row, when the soil dries, the heterogeneity of the water content increases in the first 4 hours, the maximum difference between the center and the ends reaches 2.93%. Generally, after 4 hours, the water content is almost uniform.

The same results are interpreted in another way with contour maps. The water content decreases with time. It is obvious that the water content in the center of the sample is larger than that of the ends (Fig. 3.24).

Maximum w= 37.404%, minimum w = 35.282%
(a) t = 1h

Maximum w= 36.609%, minimum w = 35.017%
(b) t = 2h

Figure 3.24 Global water content in 2D
As shown in figure 3.24 (a), when t = 1h, the maximum water content is 37.4%, in the center of the soil sample. In the extremities, the minimum water content is 35.3%. Water content decreases progressively from the center to the extremities. At t = 2h (Fig. 3.24 (b)), the water content globally decreases with a larger value in the center. The maximum water content is 36.6%. Just like in figure 3.24 (a), the minimum water content is observed in the extremities.

3.2.2 Comparisons of deformations in free desiccation tests and suction-controlled tests

3.2.2.1 Kaolin P300

(1) Evaluations of global deformations in free desiccation test on smooth support

Measurements of global deformations of kaolin P300 with an initial water content of 40% were carried out.

The global deformations are calculated in the 3 different directions: longitudinal, transversal and vertical. The schema of the soil sample and the method to calculate the deformations are shown in figure 3.25 and the following equations were used:

- Longitudinal deformation \( e_l = \frac{v_1 + v_2}{200} \)
- Transversal deformation \( e_t = \frac{h_1 + h_2}{300} \)
- Vertical deformation \( e_v = \frac{a}{4} \)

Where: \( v_1 \) and \( v_2 \) are variations of the width (in mm);
\( h_1 \) and \( h_2 \) are variations of the length (in mm);
\( a \) is the variation of height (in mm).

During the tests, at a water content of about 23% and above, the soil sample experienced a slight upward uplift at its extremities. At the very end of the desiccation process, the four extremities had risen about 1 mm. For all the free desiccation tests, after nearly 6 hours of test, the first crack appeared (Fig. 3.26 (a)). At the end of the test, the cracks were stable (Fig. 3.26 (b)).
Figure 3.25 Schema of the soil sample and calculations of global deformation

(a) (b)

Figure 3.26 Cracks of the soil sample at different time (a) t = 6h (b) t = 23h

Figure 3.27 shows the results of test 1 (measurement of deformations in 3 directions of the soil sample). Longitudinal, transversal and vertical deformations are plotted versus water content. The central part of the sample shrinks slightly less than its extremities because of the heterogeneity of water content, which was already pointed out (the central part remains wetter).

From the time–deformation curve (Fig. 3.27 (a)), the deformations in the 3 directions increase with time. The relation between deformation and water content is the same as that with time. The comparison between transversal, longitudinal and vertical deformations versus water content shows that the sample shrinks a little more in the vertical directions than in the other two directions. There are frictions on the surface of the support while in the vertical directions, shrinkage is freer. At the beginning of the drying path, for times smaller than 7 h, the deformations in these 3 directions are not obvious. Then the deformations in the longitudinal and transversal
directions are larger than the vertical deformation. After about 19 h, the vertical
deformations become larger than the two other deformations, reaching 1.9% at the
end of the test. Generally, one can consider that the deformation of the samples is
isotropic because the differences between these 3 directions are not large.

(2) Comparison of free desiccation tests with drying tests under controlled
suction

The evolution of the volumetric deformation versus the global water content
derived from the free desiccation tests is plotted in figure 3.28. Comparisons are done
between two free desiccation tests on the whole soil sample and the small sample used
for drying tests under controlled suction conditions. The volumetric deformations of
small samples measured with the method of oil in suction-controlled tests are deduced
from the following equation:

\[ \varepsilon_v = \frac{e_0 - e}{1 + e_0} \]
where: \( e_0 \) is initial void ratio of the sample; 
\( e \) is the void ratio of the sample;

We can observe that the volumetric deformations for the two free desiccation tests are similar. In a first phase, when the water content decreases from 40 to 30%, the volumetric deformation of the samples remains very small. Then, it increases quickly from \( w = 28\% \). The volumetric deformation reaches 7\% for these two tests. The volumetric deformation deduced from the tests on small samples in the suction-controlled tests reaches 15\%, which is more than those of the other samples.

Comparisons of both curves allows for addressing an aspect of relevance. In suction-controlled tests, the characteristics of the small samples are determined at various equilibrium situations, once suction, deformation, water content and degree of saturation are supposed to be homogeneous throughout the sample. The similar trends of curves highlight the fact that the transient nature of the discussed desiccation tests is not marked. The observed differences between both curves may be rather related to the uplift of the ends of the samples where the deformations were measured. Anyway, there is a difference between the measurements with the two different methods (local tests with suction-controlled and free desiccation tests). But the tendency of the curve deduced from the suction-controlled tests is similar with that of the free desiccation measurements.

In the process of measuring the volume of the small samples with oil, there may be cracks appearing in the surface or center of the sample. Then the values of void ratio are larger than those of the free desiccation tests.

Figure 3.28 Comparisons of volumetric deformation in free desiccation tests and suction-controlled test versus water content
3.2.2.2 M35

(1) Evaluation of deformations on different supports

Measurements of the global deformations of M35 with an initial water content of 82 % were carried out on both smooth support and rough support. During the tests, at a water content of about 70% and above, the soil sample also experienced an uplift at its extremities which was much larger than in kaolin P300. On the smooth support, the uplift was about 2 mm while on the rough support, the value was about 5 mm (Fig. 3.29). At the very end of the desiccation process, the four extremities had risen of about 1 mm. For all the free desiccation tests, after nearly 10 hours of test, the first crack appeared (Fig. 3.30 (a)). At the end of the test, after about 40 h, the cracks were stable (Fig. 3.30 (b)). The deformations are calculated in the 3 different directions: longitudinal, transversal and vertical deformation in the same way as for kaolin P300.

Figure 3.29 Uplift of M35 on the smooth support and rough support

Figure 3.30 Cracks of the soil sample at different times (a) t = 10 h; (b) t = 40 h

Figure 3.31 shows the results of the deformation of M35 on the smooth support (measurements in the 3 directions of the soil samples). Longitudinal, transversal and vertical deformations are plotted versus water content. The central part of the sample
shrank slightly less than its extremities because of the heterogeneity of water content.

From the time – deformation curve (Fig. 3.31 (a)), the deformations in the 3 directions increase with time. At the end of the test, vertical deformation is about 39% while transversal and longitudinal deformations are nearly the same, about 8%. The comparison between transversal, longitudinal and vertical deformation versus water content shows that the sample shrank much more in the vertical direction than in the two other directions. As explained in the test with kaolin P300, there are less friction and limitations in the vertical direction than in the two other directions. At the beginning of the drying path, after less than 23 h, the deformations in these 3 directions are very small. Then the deformations in the vertical direction become much larger than in the longitudinal and transversal directions.

![Figure 3.31 Evolution of deformations in the 3 directions of the soil sample placed on the smooth support](a)

![Figure 3.31 Evolution of deformations in the 3 directions of the soil sample placed on the smooth support](b)

Figure 3.31 Evolution of deformations in the 3 directions of the soil sample placed on the smooth support

![Figure 3.31 Evolution of deformations in the 3 directions of the soil sample placed on the smooth support](c)

![Figure 3.31 Evolution of deformations in the 3 directions of the soil sample placed on the smooth support](d)
The evolution of the deformations of M35 on the rough support versus water content and time is plotted in figure 3.32. The central part of the sample shrank slightly less than its extremities because water content decreases more slowly in the center.

From the time – deformation curve (Fig. 3.32 (a)), the deformations in the 3 directions increase with time. At the end of the test, the vertical deformation is about 25% while the transversal and longitudinal deformations are nearly the same, about 3%. The comparisons between transversal, longitudinal and vertical deformations versus water content shows that the sample shrank much more in the vertical directions than the other two directions. At the beginning of the drying path, for less than 20 h, the deformations in these 3 directions are very small. Then the deformation in the vertical directions is much larger than the longitudinal and transversal deformations.

Figure 3.32 Evolution of the deformations in the 3 directions of the soil sample placed on the rough support
(2) *Comparison of free desiccation tests with suction-controlled tests*

The evolution of volumetric deformations versus water content is plotted in figure 3.33. Comparisons are done between two free desiccation tests (on smooth and rough supports) and the global suction-controlled test.

As seen in figure 3.33, during free desiccation tests, the volumetric deformation on the smooth support is larger than that on the rough support. At the end of the tests, the volumetric deformation on the smooth support reaches about 50%, while for the rough support, the value is 30%. The volumetric deformation deduced from the global test is nearly the same as that with the smooth support, i.e. approximately 52%. It increases with the decrease of water content and increases quickly from \( w = 70\% \).

For \( w \) smaller than 70% and especially below 50%, the free desiccation tests curve corresponding to the rough support tends to separate from that of the suction-controlled test, the difference becomes larger for dryer states.

Comparison of both curves permits to address an aspect of relevance. In global tests, the characteristics are determined at various equilibrium situations, when suction, deformation, water content and degree of saturation are homogeneous throughout the sample. For free desiccation tests on smooth support, the sample is isotropic. The observed differences between smooth support and rough support may rather be related to the uplift of the extremities of the samples where the deformations were measured.

![Graph showing volumetric deformations versus water content](image)

*Figure 3.33* Comparison of volumetric deformations versus water content on smooth support, rough support and drying path
(3) Comparison of kaolin P300 and M35

As seen in figure 3.34 (a), the volumetric deformations of M35 are much larger than those of kaolin P300.

The observed differences between kaolin P300 and M35 are related to the mineralogy of montmorillonite. The swelling character of montmorillonite influences the volumetric deformations of these two materials. Another reason is the uplift of the extremities of the samples where the deformations are measured.

![Figure 3.34](image)

Figure 3.34 Comparison of volumetric deformation versus time and water content (kaolin P300 and M35)

### 3.3 Digital Image Correlation

Another approach in this thesis combines a classical study of cracks and the determination of the local deformations and displacements at different times during drying. For this purpose, photos of the soil surface are taken at regular intervals and analyzed by correlation of digital images using the software Vic-2D and 3D. This method also permits to identify earlier the zones of the sample where cracks will appear.

The Vic-2D software uses the digital image correlation (DIC) technique to derive strain measurements [Limess, 2008]. This system is able to provide two-dimensional strain maps of an entire planar specimen surface. The equipment consists of computer software and a digital camera with appropriate lens and resolution. The digital camera records the pictures during the desiccation process and the software analyzes the images and calculates longitudinal and transversal displacements, from which it derives the longitudinal, transversal and shear strains. The Vic-3D uses two digital
cameras which allows it to analyze the displacements and strains in three dimensions.

The Vic-2D and Vic-3D softwares are able to provide displacement and strain fields to show the behavior of the materials during the deformation process. The system is capable to detect the areas with higher strains inside the area of interest. The Vic-2D and Vic-3D have difficulties to provide displacement and strain maps after the specimens show cracks. The maps obtained from the specimen images that show cracks are not adequate to determine the strain values in some points in the area of interest.

The analytical process used to perform the images correlation in each case (with different materials on different supports), is the following:

(1) Observations of the images at the end of the test in order to locate the precise positions of the cracks; Several cracks of different types are chosen and analyzed in detail.

(2) Observations of the images starting from the beginning of the test to show the evolution of the displacements and strains of the soil leading to the formation of cracks; The observations are centered on the places where the cracks mentioned in step (1) will appear.

(3) Precise analysis of the displacements and strains on chosen sections which cross the maximum number of cracks and in the vicinity of the cracks. The chosen sections are significant for the analysis because of the cracks they pass. This is named “Global analysis” in the following text. The analysis in the neighborhood of the cracks is named “Local analysis”.

As indicated before, all the displacement and deformation maps shown in this thesis are taken before the appearance of cracks.

### 3.3.1 Influence of time for kaolin P300 on smooth support

Tests with kaolin P300 on three different supports were performed. The initial water content is 40 %, which is equal to \( w_L \). The evolution of water content on these three supports is shown in figure 3.35. Water content in the test with the rough support changes slightly more quickly than that on the other two supports, but the difference of the rates is not large. At the end of the tests (\( t = 27 \) h), water contents with these three supports are the same and are constant, at about 2%. The roughness of the supports does not influence the evolution of water content in the case of kaolin P300. We will first present the results on the smooth support and then, the results on the intermediate and rough supports.
3.3.1.1 At the end of the desiccation (t = 19h)

At the end of desiccation, the cracks form a kind of network. Taheri (2005) did research on thermal fatigue cracking in which contraction is due to temperature decrease. The form of the crack network of soil which is caused by suction in my research is quite similar with the thermal fatigue cracking.

At t = 19 h, the two displacements (U and V) and three strains ($\varepsilon_{xx}$, $\varepsilon_{yy}$ and $\varepsilon_{xy}$) are constant (Figure 3.37). This is the end of the desiccation for kaolin P300 on the smooth support. The characteristics of the cracks are determined and stable.

As shown in figure 3.37, the white arrows represent the directions of the local displacements in the sample. Theoretically, during free drying, the displacements on the boundaries should be larger than those in the center of the sample (as shown in the illustration of figure 3.36) and the displacements should be oriented towards the center. If no other mechanism is involved in the process but homogeneous drying, i.e. if the heterogeneity of the sample and the effect of friction on the support can be neglected for instance, then the volumetric variations should result in a general centripete displacement of the material.

![Schematic diagram of displacement in the sample](image-url)

Figure 3.36 Schematic diagram of displacement in the sample
Figure 3.37 (a) shows the longitudinal displacements. The maximum displacement towards the right side is represented by the red color and the violet color represents the maximum displacement towards the left side. And the colors between them represent the displacements between the minimum and the maximum values. The maximum longitudinal displacement occurs on the right side boundary, and is oriented towards the left side, with a value of 2.28 mm. On the left boundary of soil samples, the displacements are also large. The maximum value is 2.12 mm, towards the right side. Transversal displacements are shown in figure 3.37 (b). The maximum displacements happen in the lower side of the sample and towards the center, with a value of 2.42 mm. In the upper side of the sample there is a large displacement towards the center, with a value of 1.28 mm.

The three strains are also analyzed. As stated in chapter 2, positive strains are related to extension, while negative strains are related to compressions. This corresponds to positive forces which are tractions and negative forces, compressions.

One can note that transversal strains ($\varepsilon_{yy}$) are all negative, meaning that they are oriented towards the upper side of the soil sample. The maximum value is 5.05%, mainly in the upper boundary and lower boundary of the soil sample. On the other side, the minimum value is 0.05%, all near the boundaries of the cracks (Figure 3.37 (c)). The maximum positive shear strain ($\varepsilon_{xy}$) is 1.46% and the maximum negative shear strain is 1.4%. The negative shear strains are mainly located near the boundaries of the cracks (Figure 3.37 (d)). The maximum longitudinal strains ($\varepsilon_{xx}$) are mainly in the left end of the sample. Their value is 6.45%, with an orientation towards the center of the sample (Figure 3.37 (e)). The maximum positive strain $\varepsilon_{xx}$ is 0.4%, mostly near the boundaries of the cracks. There are also large negative $\varepsilon_{xx}$ on the other side of the cracks (Figure 3.38). Except on the two sides of the cracks and near the boundaries of the sample, $\varepsilon_{xx}$ in most part of the sample is between 0 and 6.45%.

Three representative zones with cracks which correspond to 3 possible formation mechanisms named I, II and III, respectively, are analyzed. The strains on the left side of zone I are nearly 0% while, on the right side, the strains are larger. It is possible that on the left side of the crack, the soil is pasted to the support. The appearance of a crack in zone I is caused by extensions. The crack in zone II is the result of the propagation of another crack; The formation of the crack in zone III is related to the crack in the upper boundary of the sample. On the boundaries, drying of clay is quicker than inside the material (Figure 3.37 (e)).

In zone IV, shown in figure 3.37 (e), the phenomenon of stress concentration is observed. The soil sample is the strongest when the force is evenly distributed over its area, so a reduction in area, e.g. caused by a crack, results in a localized increase in stress. The soil material can fail, via a propagating crack, when a concentrated stress
exceeds the material theoretical cohesive strength. At the two ends of the crack in zone IV (crack IV), the strains are extensions with a value of about 0.4%, while in the vicinity of crack IV, the strains are evenly compressions. The same stress concentration phenomenon can also be observed in the other cracks. The strains in the ends of the cracks are also extensions (with the red color).

The bifurcation of a crack in zone V can be observed in figures 3.37 (d) and (e). Crack 2 in zone II appears from the center of the sample and propagates in the southwestern direction. But it does not evolve with the same direction in zone V. The direction of this crack turns to the right and forms an angle of 90° with the previous path. This phenomenon can be explained by the following schema: when a crack is caused by traction, the propagation direction follows the direction of strains. If there are shear strains in the vicinity of the crack, then its direction will change.

As seen in figure 3.37 (e), transversal strains $\varepsilon_{xx}$ in zone V are about 0.4%. The direction of crack 2 in this zone should be southwestern which is also the previous propagation direction. At the bifurcation point of crack 2 in this area, $\varepsilon_{xy}$ increases and reaches about 0.9%, which influences the direction of crack 2.

The direction of the displacement near the crack in zone I is towards the right which can be verified by the white vectors (Figure 3.38 (a)). On the left side of the crack, the length of the vectors is smaller than that on the right side, which means that the displacement on the right side is larger than on the left side. $\varepsilon_{xx}$ on the left is about 0.4%, its direction is also towards the right. On the right side, the value is about 4.3% and the direction is towards the left. Because of the difference in the displacements and strains, there are extensions in the soil. Then the cracks appear. Crack in zone II corresponds to the same conditions (Figure 3.38 (b)). The displacements on the right side are larger than those on the left side. And the strains on the right side are about 4% with a left direction; they are much larger than those on the left side. Crack in zone III is also of the same type. Maximum and minimum strains are 0.4% and 4%, respectively, with opposite directions (Figure 3.38 (c)).
(a) Longitudinal displacement of kaolin P300, $t = 19\ h$

(b) Transversal displacement of kaolin P300, $t = 19\ h$

(c) Transversal strains ($\varepsilon_{yy}$), $t = 19h$

(d) Shear strains ($\varepsilon_{xy}$), $t = 19h$

Figure 3.37 Displacement and strain maps of kaolin P300 on smooth support
Figure 3.37 Displacement and strain maps of kaolin P300 on smooth support

Figure 3.38 Cracks of kaolin P300 on smooth support (t = 19 h)
3.3.1.2 Evolutions of displacements and strains of kaolin on smooth support at different times (before cracking)

The two principal strain maps are drawn at different times (t = 3 h and 6 h) in the case of the kaolin P300. Figure 3.39 (a) shows the principal strain vectors at t = 3 h. At t = 3 h and 6 h, we follow the same three zones as those analyzed in the above section 3.3.1.1. For zone I, \( \varepsilon_{xx} \) is between 0.54\% and -0.72\%. The principal strains in zone I are very large and their values are positive (the color is red), which means that there are extensions in this zone. \( \varepsilon_{xx} \) in zone II is between 0.36\% and -0.72\%. The principal strains are small.

When t = 6 h, \( \varepsilon_{xx} \) and the principal strains increase while the angle of the principal strains does not change much. As seen in figure 3.39 (b), the principal strains are still large on the boundaries and, at the same time, they are larger in the other parts of the samples. It is quite obvious that the principal strains in zone I are larger than those at 3 h which indicates the crack appearance. For zone II, it is the same situation.

The first crack appears at t = 7 h. When t = 8 h, more cracks develop (Fig. 3.39 (c)). Local displacements become important in the sample. Near zone I, \( \varepsilon_{xx} \) is between 0.9\% and -2.38\%. Near zone II, \( \varepsilon_{xx} \) is between 0.9\% and -1.39\%.

There are more small cracks at t = 9 h. Displacements remain larger on the boundaries of sample and \( \varepsilon_{xx} \) evidently increase, with values between 1.15\% and -5.35\%. \( \varepsilon_{xx} \) on the left side of the crack which appears in zone I is about 1.15\% and on the right side, the value is about -2.95\%. The differences are larger than those at 8 h. \( \varepsilon_{xx} \) on the left and right sides of the crack in zone II are -2.95\% and -1.28\%, respectively. As shown in figure 3.39 (d), in the neighboring zones a, b and d, there is already the appearance of separated cracks. And in these three zones and zone d, the strains are larger than in the other areas nearby. Maximum \( \varepsilon_{xx} \) is about 1.15\%. During desiccation, there are possibilities that crack in zone a connects with the others to form a network of cracks.

At t = 10 h (Fig. 3.39 (e)), the small cracks connect together and they go through the samples providing a network of cracks like thermal fatigue cracking. Crack in zone a connects with crack in zones b and d rather than with crack in zone c. \( \varepsilon_{xx} \) in zone c are very small, and decrease significantly. This is because the cracks in zones a, b and d formed first and this resulted in stress relaxation in zone c. Therefore there is no possibility of crack in zone c although the strains in this area were initially large. After the connection and evolution of cracks in these areas, energy is released and strains decrease. Many of the cracks are transversal. In other tests with kaolin P300 on the smooth support, longitudinal cracks can also be observed. For the geometry of the sample, the directions of the cracks do not appear to follow any definite pattern.
Figure 3.39 Maps of principal strains and $\varepsilon_{xx}$ at different times
Figure 3.39 Maps of principal strains and $\varepsilon_{xx}$ at different times
3.3.1.3 Evolutions of displacements and strains of kaolin on smooth support on chosen sections

(1) Global results (for the whole section)

In order to better understand the characteristics of the observed displacements and strains especially in the crack area and in the other parts of the sample, two sections are chosen, based on the fact that they pass through several cracks. The evolution of the displacements and strains distributions along the longitudinal and transversal sections, plotted at different times, is shown in figure 3.40. The transversal displacement $V$ and $\varepsilon_{yy}$ are analyzed in a transversal section while the longitudinal displacement $U$, $\varepsilon_{xx}$ and $\varepsilon_{xy}$ are plotted along a longitudinal section (figure 3.37 (e)).

As the soil sample dries, displacements and strains increase. The first crack appears at around 7 h. During the first 9 hours, the displacements in the center of the sample are nearly homogeneous. They evolve progressively until $t = 9$ h; the displacements are a little larger on the boundaries of the sample. Progressively the displacements
near the five cracks become also a little larger than in the other parts (figure 3.37 (e)). Before 7 hours of the test, there is no visible crack. In the early 9 hours of desiccation, differences between positive and negative displacements are very small, less than 1.0 mm. And the soil sample shrinks very slowly so that the displacements are very small. The maximum U is less than 0.5 mm. On the left side, the U are positive, which means that the direction of U is towards the right side. At the right end of the section, the direction of U is negative and towards the left. According to the desiccation images, between 7 and 9 hours, cracks develop very slowly whereas, between 9 h and 11 h, more cracks develop and the displacements increase significantly, especially near the cracks. When t = 11.33 h, the maximum U reaches about 2 mm at the right end of the chosen longitudinal section. Near crack 2, 4 and 5, U turn to be negative and the direction changes. The directions on the two sides of the cracks are opposite. When t = 12 h, the sample reaches its shrinkage limit. In the more active desiccation period (between 7 h and 12 h), there are more cracks developing and the sample shrinks evidently. The volume of the samples decreases, which causes the increase of displacements.

After that, cracks do not evolve very much and U does not change significantly. At 33 and 42 hours, the maximum U are the same, about 2.3 mm, near crack 5. The differences among the values of U when t = 11.33, 33 and 42 hours are very small (Figure 3.40 (a))

For transversal displacements V (Figure 3.40 (b)), it is the same tendency as for longitudinal displacements U. In the first 9 hours, V increase progressively but slowly. V are positive and the maximum V which reaches about 0.6 mm occurs on the lower side of the transversal section. After 9 hours, V increase dramatically, especially on the lower side of the sample. Round the cracks, V turn to be negative, which means that the directions of displacements on the two sides of cracks are opposite. This verifies that the appearance of cracks is mainly caused by tractions. After 11.33 hours, V do not change much. The differences between the maximum and minimum V for 11.33, 33 and 42 hours are quite similar. At the end of desiccation, the maximum V still lies at the lower end of the transversal section. Cracks in the transversal section correspond well with the displacement schema shown in figure 3.40 (b).

Regarding the three strain components, generally \( \varepsilon_{xx} \) and \( \varepsilon_{xy} \) are much larger than \( \varepsilon_{xy} \), which means that the principal directions of strains are mainly oriented in the longitudinal and transversal directions. In the early 9 hours, strains progressively increase. Especially at the positions of cracks, strains develop quicker than in the other parts (Fig. 3.40 (c) (d)). At the ends of the sample, \( \varepsilon_{xx} \) and \( \varepsilon_{xy} \) are the largest and they are compressive. On the contrary, near the cracks, \( \varepsilon_{xx} \) and \( \varepsilon_{xy} \) are
extensions. Around the cracks, there are also compressive $\varepsilon_{xx}$ and $\varepsilon_{xy}$. The compressions are relatively small compared to the extensions. After 9 hours, $\varepsilon_{xx}$ and $\varepsilon_{xy}$ increase dramatically. There are less extensions in $\varepsilon_{xx}$ except near crack 3. In the other parts of the chosen sections, all are compressions. For $\varepsilon_{xy}$, the evolution is similar. They increase more quickly after 9 hours and, at the end of desiccation, they are negative. The presence of crack 1’, 2’ and 3’ agrees well with the distributions of transversal strains shown in figure 3.40 (d). During this period, the soil sample dries more quickly and shrinks more strongly so that compressions evolve significantly. The boundaries of the sample experience a slight uplift. This might explain why strains at boundaries are larger than in the other parts.

Compared with $\varepsilon_{xx}$ and $\varepsilon_{yy}$, the difference between the maximum and minimum values of $\varepsilon_{xy}$ is much smaller. The maximum value is 3 % (figure 3.40 (e)).

(a) Distribution of longitudinal displacements (U)

Figure 3.40 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in the chosen sections during desiccation (kaolin P300, smooth support)
Figure 3.40 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in the chosen sections during desiccation (kaolin P300, smooth support)
Figure 3.40 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in the chosen sections during desiccation (kaolin P300, smooth support)
(2) Local results (in the vicinity of cracks)

Two different types of cracks, in zone I (Crack I) and in zone III (crack III) are analyzed more precisely in figure 3.41. Crack I represents the inner type of crack and crack III is related to the boundary of the sample. For crack I, $\varepsilon_{xx}$ and $\varepsilon_{yy}$ progressively increase. Near the position of the cracks, $\varepsilon_{xx}$ correspond to extensions while, on the two sides of crack I, the strains are compressions. Before 10 hours, extension increases near the cracks and compressions also increase at the boundaries of the cracks. After 10 hours, the results in the crack I area (Fig. 41(a)) show that the extension seen before disappears, demonstrating that the crack has already developed. Then the strains turn to be compressions near the positions of the cracks and at the boundary of the cracks, compressions still increase. Shear strains control the distortions of the clay aggregates, but they are relatively smaller than $\varepsilon_{xx}$ and $\varepsilon_{yy}$. Maximum $\varepsilon_{xy}$ is 1.2%, while the maximum $\varepsilon_{xx}$ and $\varepsilon_{yy}$ are 5% and 2% respectively.

Crack III is near the boundary of the sample, and the compressions are very large in these areas. $\varepsilon_{xx}$ and $\varepsilon_{yy}$ in crack III are mainly compressions at the beginning of desiccation. They increase progressively. The difference between the strains in the cracks and on the boundaries of the cracks increases with time (Fig. 3.41 (d) (e)).

It is obvious that the deformations in zone III caused by the boundary cracks are mainly compressions while in the center of the sample, there are compressions and extensions at the same time. At the end of desiccation, the extensions turn to be compressions because of the shrinkage of soil sample.

(a) Longitudinal strains of zone I ($\varepsilon_{xx}$)  
(b) Longitudinal strains of zone III ($\varepsilon_{xx}$)

Figure 3.41 $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in zone I and III
Compared with the global measurements (i.e. the mean values measured in the whole specimen in the free desiccation tests) of longitudinal and transversal strains (Fig. 3.42), the results measured by Vic-2D are a little larger. In the early 7 hours when the displacements and strains are very small, the evolution of strains is nearly the same. On the contrary, during the period when more cracks appear, the strains measured by Vic-2D are much larger than the global measurements. At the end of desiccation, the longitudinal and transversal strains measured globally are nearly the same, reaching 1.8%. This value is not so different from the measurement by Vic-2D. Because the global measurements in free desiccation test are realized by hand with a rule the results are less precise than the measurements by Vic-2D.
3.3.2 Influence of different supports for kaolin P300

In order to analyze the influence of roughness and friction on the development of strains and the formation of cracks, another two tests were carried out on different supports, one intermediate and one rough. As already mentioned in chapter 2, the average roughness of the intermediate support and rough supports is 37.828 μm and 90.359 μm, respectively.

3.3.2.1 Evaluation of displacements and strains for kaolin P300 on intermediate support

(1) Displacements and strains at different times

(i) t = 9 h

Contrary to what is observed on the smooth support, there are already many cracks on the intermediate support when t = 9 h (Fig. 3.43). As already stated in section 3.3.2, the general tendency of shrinkage direction is towards the center of the sample from the ends. The maximum longitudinal displacement (U), on the right boundary of the sample, is 1.18 mm with the direction towards the left. The maximum U, towards the right, is 0.42 mm and it mainly occurs on the left boundary of the sample. It is also significant that these positive and negative displacements U with the different directions are frequently on the opposite sides of the cracks (Fig. 3.43 (a)). It is also significant that these positive and negative displacements U with the different directions are frequently on the opposite sides of the cracks (Fig. 3.43 (a)). This phenomenon is one reason of the cracks. The maximum transversal displacement (V) towards the upper side of the sample is 0.94 mm, located mainly in the lower end of
the sample while the maximum V on the lower side of the sample is 0.57 mm. Just like the distribution of U, these two displacements happen on the opposite sides of the crack (Fig. 3.43 (b)).

As seen in figure 3.43 (c), the maximum $\varepsilon_{yy}$ is 2.1%, mostly round the sides of the cracks. For $\varepsilon_{xy}$ (Fig. 3.43 (d)), the maximum positive value is 1.82% and the maximum negative value is 1.72%. They are also mainly near the boundaries of the cracks. For $\varepsilon_{xx}$, the conclusion is the same. The maximum positive $\varepsilon_{xx}$ is near the boundary of the cracks, with a value of about 1.4% (Fig. 3.43 (e)). For example on the left side of crack I, the minimum negative $\varepsilon_{xx}$ is 4.6% while on the right side, $\varepsilon_{xx}$ is about 1.4%. These two $\varepsilon_{xx}$ with different directions happen near the crack. This is one reason of the crack. The same phenomenon can be observed in crack of zone II. In some positions, there is no deformation on the boundaries of the considered area whereas, inside the area, the deformations are large (3.43 (e)).

(a) Transversal displacements of kaolin P300, t = 9 h  
(b) Longitudinal displacements of kaolin P300, t = 9 h

(c) Transversal strains ($\varepsilon_{yy}$), t = 9 h  
(d) Shear strains ($\varepsilon_{xy}$), t = 9 h

Figure 3.43 Results of kaolin P300 on the intermediate support
(e) Transversal strains ($\varepsilon_{xx}$), $t = 9$ h

Figure 3.43 Results of kaolin P300 on the intermediate support

(ii) $t = 1$ h and 6 h

Superposition maps of principal strains and $\varepsilon_{xx}$ are analyzed at $t = 1$ h and 6 h. At the beginning of desiccation, the maximum $\varepsilon_{xx}$ is on the boundary of the sample. Throughout the surface of the sample, principal strains are very small. In the area of crack I, principal strains are less than 0, which means there are compressions. For crack II, the principal strains $\varepsilon_1$ are less than 0 and $\varepsilon_2$ are more than 0 (Fig. 3.44 (a)). At $t = 6$ h, the maximum and minimum $\varepsilon_{xx}$ increase. The angles of the principal strains do not change much. The values of the principal strains increase obviously (Fig. 3.44 (b)). The difference between the maximum and minimum $\varepsilon_{xx}$ near the boundaries of the cracks also increases.

On the intermediate support, the frictions are the same throughout the whole sample. The formation of the cracks is more progressive; while on the smooth support, there are more points which are pasted on the support; and the evolution of cracks is quicker.
Figure 3.44 Results of kaolin P300 on intermediate support

(a) Longitudinal strains ($\varepsilon_{xx}$), $t = 1$ h

(b) Longitudinal strains ($\varepsilon_{xx}$), $t = 6$ h
(2) Evaluations of displacements and strains of kaolin P300 on chosen sections

(i) Global results

The first crack appears after 4 h of desiccation. Most cracks appear at about 9 hours (Fig. 3.43).

During the first 4 hours of desiccation, the displacement $U$ increases progressively and homogeneously. At the boundaries of the sample, $U$ is a little larger than at the other points in the longitudinal section. After 4 hours, on the two sides of the cracks, $U$ increases largely. For example, at 7 h, $U$ around crack 4 is nearly 0.4 mm, much larger than the other parts. Between 7 h and 10 h, there are more cracks appearing which correspond with the variation of the difference between the maximum and minimum $U$ ($V$). The displacement $U$ around the cracks is much larger than in the other parts too, especially the displacement in crack 4. At the end of desiccation, the maximum $U$ is 1.4 mm, located at crack 4. Comparing with the desiccation on the smooth support, the evolution of the displacements and deformations is quicker. On the smooth support, after about 9 hours the maximum displacement is still less than 0.5 mm. (Fig. 3.45 (a))

For displacement $V$, it is the same tendency as for displacement $U$. Near the cracks, $V$ is larger than elsewhere. The maximum $V$ is at the boundary of crack 4 and reaches 2.5 mm. The variations of $V$ round the cracks agree well with the position of the cracks. (Figure 3.45 (b))

The difference between the maximum and minimum $\varepsilon_{xx}$ is the same as that for $\varepsilon_{yy}$. $\varepsilon_{xy}$ is smaller than the two other strains. The strains develop progressively. In the early 7 hours, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ increase slowly while after that, they increase strongly. At $t = 9.6$ h, $\varepsilon_{xx}$ and $\varepsilon_{yy}$ reach their maximum value, which are about 9% and 7% respectively. Just like the evolution of the strains on the smooth support, in the early times of desiccation, there are extensions and compressions at the same time, especially around the cracks, where the extensions are obvious. As the cracks develop, extensions and compressions increase at the same time. The variations of compression are larger than those of extension (Fig. 3.45 (c), (d) and (e)).

The global evolution of water content on these two supports is nearly the same. The evolution of the cracks on the intermediate support is faster than on the smooth support. This depends on the roughness of the supports. On rougher support, friction is larger. During desiccation the samples shrink towards the center. The shrinkage movement of soil is more difficult on rougher support in longitudinal and transversal directions while in vertical directions, the shrinkage movement is easier.
Figure 3.45 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distribution in chosen sections during desiccation (kaolin P300, intermediate support)
Figure 3.45 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distribution in chosen sections during desiccation (kaolin P300, intermediate support)
Figure 3.45 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distribution in chosen sections during desiccation (kaolin P300, intermediate support)

(ii) Local results

Precise analysis of cracks in zone I (crack I) and zone II (crack II) are shown in figure 3.46. Crack I is near the boundary. In the early 4 hours, there are compressions in the positions of the cracks, then, with the development of desiccation, compressions increase. At $t = 7$ h, the extensions in cracks and the compressions round the cracks increase at the same time. At the end of desiccation, $\varepsilon_{xx}$ and $\varepsilon_{yy}$ reach their maximum value. They are 4.5% and 3.1%, respectively. It is obvious that at the positions of the cracks, there are extensions.

For crack II, it is the same condition as for crack I. Around crack II, it is mainly compressions.
3.3.2.2 Evaluations of the displacements and strains on the rough support

Evolution of displacements and strains on the rough support, which has an average roughness of 90.359 μm, is analyzed. The process of cracking is more rapid on rough
support than on the other two supports, and the first crack appears at about 2.67 h, which is earlier than in the tests on the other supports. As stated at the beginning of this section, because of the limitations of Vic-2D, it is not possible to analyze the images after about 10 h in the case of the rough support because of the appearance of cracks.

(1) Displacements and strains of kaolin at different times

(i) At the end of desiccation

Figure 3.47 shows the image at the end of desiccation at 28 h. The deformations of the sample on the boundaries and on the two sides of the cracks are important.

![Figure 3.47 Image at the end of desiccation (t = 28 h)](image)

(ii) t = 10.67 h

Theoretically, the directions of displacement are towards the center of the sample from the boundaries (Figure 3.48 (a)). On the left boundary of crack in zone II (crack II) (Figure 3.49), the displacement is nearly 0 mm, this may be due to the fact that the soil sample is pasted on the support. On the left side of crack in zone I (crack I), the direction of displacement is towards the right, which is the same as that on the right side. It appears that the displacement on the right side is much larger. Crack I corresponds to extensions related to the differences between the displacements on both sides. On the left side of crack I, some displacements are directed towards the lower side of the sample, which is also different from the direction on the other side of the crack. For crack II, the same phenomenon is observed. On the left side of crack II, the displacement is about 0 mm, and towards the left side whereas, on the right side of
the crack II, the displacement is towards the right. These two cracks are due to the extensions in the soil sample.

The maximum negative $\varepsilon_{xx}$ is 11.6% at the right end. At the left end $\varepsilon_{xx}$ is smaller, about 6.5%. Near the boundaries of the cracks, $\varepsilon_{xx}$ is positive, the maximum value is about 5.7%.

(iii) $t = 1 \text{ h}$

Like for $t = 10.67 \text{ h}$, the maximum negative $\varepsilon_{xx}$ is observed on the left and right boundaries of the sample. The principal strains near crack I are positive, which means that there are extensions in these areas. $\varepsilon_{xx}$ is about 0.6% in crack I. In crack II, the principal strains are small compared with crack I. There are extensions and compressions at the same time in crack I. $\varepsilon_{xx}$ is about 0.03% in crack II, which is less than that of crack I. This can be verified with the principal strains.

(iv) $t = 5 \text{ h}$

After 4 hours, positive and negative $\varepsilon_{xx}$ are larger compared with those at $t = 1 \text{ h}$. At the boundaries, the maximum $\varepsilon_{xx}$ is 5.25%. In crack I, $\varepsilon_{xx}$ is about 1.21%. The principal strains are larger than before and the angles of principal strains do not change much in crack I. The maximum $\varepsilon_{xx}$ in crack II is about 0.2%. The principal strains are much larger than those at $t = 1 \text{ h}$.

(v) $t = 6 \text{ h}$

When $t = 6 \text{ h}$, more cracks appear and the displacements are much larger than before. The directions of displacements are obvious with the white vectors. On the four boundaries of the sample, the directions of the displacements are towards the center of sample while, in the other areas, the directions are transversal and towards the right. At the right ends of the sample, there are compressions. $\varepsilon_{xx}$ in crack I and crack II are larger than before.

With the appearance of cracks and the tractions throughout the soil sample, the displacements increase dramatically.
Figure 3.48 Results of kaolin P300 on the rough support

(a) Longitudinal strains ($\varepsilon_{xx}$) and principal strains, $t = 10.67$ h

(b) Longitudinal strains ($\varepsilon_{xx}$) and principal strains, $t = 1$ h
Figure 3.48 Results of kaolin P300 on the rough support

(c) Longitudinal strains ($\varepsilon_{xx}$), $t = 5$ h

(d) Longitudinal strains ($\varepsilon_{xx}$), $t = 6$ h

Figure 3.48 Results of kaolin P300 on the rough support
(2) Evaluations of displacements and strains of kaolin on representative sections

(i) Global results

In the early times of desiccation, the differences between the maximum and minimum displacements $U$ ($V$) on the rough support evolve more rapidly in comparison with those tests on the smooth and intermediate supports. The difference between the maximum and minimum $U$ is 4 mm while for $V$, the value is about 3 mm at the end of desiccation. After 7 hours, $U$ increase strongly and at 8 h, the differences between maximum and minimum $U$ around the cracks are larger than in the other parts of the sample. For displacements $V$, they increase progressively and the variations are very large around the cracks (Figure 3.50 (a) and (b)).

In the same way as for the other supports, around the cracks, the strains are extensions and they increase progressively. The maximum values correspond to the cracks observed in figure 3.48 (a).
Figure 3.50 U, V, $\varepsilon_{\text{xx}}$, $\varepsilon_{\text{xy}}$ and $\varepsilon_{\text{yy}}$ distribution in chosen sections during desiccation.
(d) Distribution of transversal strains ($\varepsilon_{yy}$)

(e) Distribution of shear strains ($\varepsilon_{xy}$)

Figure 3.50 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distribution in chosen sections during desiccation (kaolin P300, intermediate support)

(ii) Local results

Crack I is in the center of the sample. At the beginning of desiccation, before 7 hours, the extensions in the cracks decrease and the compressions near the boundaries of the cracks increase. At $t = 8$ h, the extensions and compressions increase strongly at the same time. As described in chapter 1, there are three modes of cracking: opening mode, sliding mode and tearing mode. It is obvious that the formation of crack I
corresponds to an opening mode caused by the tractions.

Crack III is near the boundary of the sample. As seen in figure 3.48 (a), its formation corresponds to tearing mode. Unlike crack I, at the beginning of desiccation, extensions and compressions increase at the same time while after 7 hours, extensions decrease and compressions increase.

Figure 3.51 $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in zone I and III
(3) Comparisons of the results of kaolin P300 on the three supports

Comparison of the differences between the maximum and minimum displacements following the direction U (V) on the three different supports is shown in figure 3.52. The evolution of displacements is nearly the same for the same material and the same support. For the smooth support, the evolution of the displacements is slower and cracks appear in a very short time (between 8 h and 10 h). The development of the displacements is quicker on the rough support and the values are larger than for the two other supports at the same time. This influence of the supports roughness corroborates quite well the evolution of water content observed previously in section 3.31. The samples paste themselves more easily to the smooth support. In that case, when the tractions reach a threshold value, more cracks appear in a very short time. On the contrary, for the two other supports, the evolution of cracks is more progressive. For the rougher support, the displacements are larger than for the two other supports at the same time (Fig. 3.53).

![Graph showing comparisons of displacements](image)

Figure 3.52 Comparison of the differences between minimum and maximum displacements on the three supports

Bending (also known as flexure) characterizes the behavior of a slender structural element subjected to an external load applied perpendicularly to a longitudinal axis of the element. In the case of soil, when the support is rougher, it is harder for the soil to move in the transversal and longitudinal directions of the plane because of the large friction on the support. The easier direction for the soil to move is the vertical direction, which results in the deformation pattern shown in figure 3.53.
3.3.3 Comparison between the free desiccation behaviors of the different materials on smooth support

In addition to the research on the effect of the supports, the digital image correlation was also used to analyze the free desiccation behavior of the four other mixtures (M35, M50, M65 and M100). Because of the swelling characteristics of the montmorillonite fraction contained in these four mixtures, the results are different. All the tests are carried out on the smooth support because swelling accentuates the bending phenomena illustrated in figure 3.53, making the measurements difficult or even impossible.

3.3.3.1 M35

Tests with M35 on 3 different supports were performed. The initial water content is 82%, which is equal to $w_L$. The evolution of water content on these 3 supports is shown in figure 3.54. With the rough support, the water content changes more rapidly than on the two other supports. At the end of the tests ($t = 40$ h), water content with these 3 supports is constant, with a value of about 2% for all supports. The roughness
of the supports does not influence much the evolution of water content of M35. In the same way as for kaolin P300, we will first present the results on the smooth support and then, those on the intermediate and rough supports.

![Figure 3.54 Evolution of water content of M35 mixture on the 3 different supports](image)

**I. Displacements and strains on the smooth support at different times**

(i) At the end of desiccation (t = 37.33 h)

As analyzed before, the displacements on the boundaries are much larger than in the other areas, for example, the center of the sample and all the displacements should be directed towards a point in the center of the sample (Figure 3.55 (e)). The displacements of M35 on the smooth support are very large compared with those of kaolin P300. One reason is the shrinkage of the material and another reason is the bending phenomenon as mentioned before.

On the left side of crack I, $\varepsilon_{xx}$ is -0.95%. Near the spots where there are small negative $\varepsilon_{xx}$, there are zones with larger negative $\varepsilon_{xx}$ equals to -9.35%. Because of the large difference of strains in this area, there are tractions. The direction of the displacement confirms the results. On the boundaries of crack II, the average $\varepsilon_{xx}$ is about 6.2%.

(ii) t = 1.33 h

At the beginning of desiccation, the maximum negative $\varepsilon_{xx}$ occur in the left and right ends of the sample. As shown in figure 3.56 (a), the principal strains on the boundaries are larger than elsewhere and they are mainly compressions. On the contrary, in the other areas, the principal strains are all extensions. The values of the principal strains near cracks I and II are nearly the same.

(iii) t = 9.33 h

When $t = 9.33$ h, $\varepsilon_{xx}$ increases obviously. The maximum negative $\varepsilon_{xx}$ still are observed at the four ends of the sample. In the center of the sample, the principal strains are also extensions. The principal strains increase and the angles change too.
Figure 3.55 Results of M35 on the smooth support

(a) Longitudinal displacement of M35, $t = 37.33\, \text{h}$

(b) Transversal displacement of M35, $t = 37.33\, \text{h}$

(c) Transveral strains ($\varepsilon_{yy}$), $t = 37.33\, \text{h}$

(d) Shear strains ($\varepsilon_{xy}$), $t = 37.33\, \text{h}$

(e) Longitudinal strains ($\varepsilon_{xx}$), $t = 37.33\, \text{h}$
Figure 3.56 Results of M35 on smooth support at different times

(a) Longitudinal strains ($\varepsilon_{xx}$), $t = 1.33$ h

(b) Longitudinal strains ($\varepsilon_{xx}$), $t = 9.33$ h
As shown in figure 3.55, there is no uplift phenomenon until the end of desiccation. The quantitative analysis with software VIC-2D remains valid with M35 on smooth support.

(2) Evaluations of displacements and strains of M35 on chosen sections (smooth support)

(i) Global results

During the desiccation of M35 on smooth support, the first crack appears at about 8 h. The longitudinal and transversal displacements (U and V) are very small in the first 16 hours, less than 1 mm. Then U and V progressively increase. Deformations end at about 38 h.

At the beginning of desiccation, at the left side of the chosen longitudinal section, U is negative and oriented towards the left side. On the right side of the section, U is towards the right side. During desiccation, on the left side of the sample, U turns to be positive, which means that the direction of U changes. At the other end of the section, the direction of U changes too. At the end of drying, the differences of U around the cracks are large. For displacement V, it is the same condition.

Compared with the desiccation of kaolin P300 on smooth support, the process of desiccation is less rapid, which is related to the lower permeability of the mixture. And at the end of desiccation, the maximum U and V are much larger for kaolin P300.

Like in the case of the desiccation of kaolin P300, the maximum $\varepsilon_{xx}$ and $\varepsilon_{yy}$ are much larger than $\varepsilon_{xy}$. The maximum difference between $\varepsilon_{xx}$ is 14% at 34.33 h. At 37.33 h, the difference between maximum and minimum $\varepsilon_{xx}$ decreases down to 8% at the end of desiccation. For $\varepsilon_{yy}$ the evolution is similar. At the beginning of desiccation, $\varepsilon_{yy}$ increases progressively; after 34.33 h, it decreases a little. Generally the difference between maximum and minimum $\varepsilon_{yy}$ is larger than that of $\varepsilon_{xx}$. The evolution of the difference between maximum and minimum $\varepsilon_{xy}$ is the same as that of $\varepsilon_{xy}$.
Figure 3.57 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in the chosen section during desiccation (M35, smooth support)
Figure 3.57 U, V, ε_{xx}, ε_{xy} and ε_{yy} distributions in the chosen section during desiccation (M35, smooth support)
(e) Distribution of shear strains ($\varepsilon_{xy}$)

Figure 3.57 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in the chosen section during desiccation (M35, smooth support)

(ii) Local results

Crack in zone I (crack I) is near the boundary while crack in zone II (crack II) is in the center of the sample. As shown in figure 3.58 (a) and (b), in the early times of desiccation, there are very small extensions $\varepsilon_{xx}$ in the two cracks. During desiccation, for zone I, $\varepsilon_{xx}$ are extensions and near the boundary of crack, they are compressions. For zone II, $\varepsilon_{xx}$ are mainly compressions and they increase progressively. On the boundary of the sample, compressions at the end of desiccation are larger than in the center. The tendency of $\varepsilon_{yy}$ for these two cracks is the same: $\varepsilon_{yy}$ increase during desiccation while at the end of tests, they decrease.

For zone I, shear strains $\varepsilon_{xy}$ on the left and right sides of the sample are opposite. On the contrary, for zone II, they are all positive. These two cracks are the results of two different failure mechanisms: Crack I on the boundary corresponds to tearing mode whereas crack II corresponds to traction mode. This is the cause of the differences between the $\varepsilon_{xy}$ of crack I and crack II (Figure 3.58 (c) and (f)).
(a) Longitudinal strains in zone I ($\varepsilon_{xx}$)  
(b) Transversal strains in zone I ($\varepsilon_{yy}$)  
(c) Shear strains in zone I ($\varepsilon_{xy}$)  
(d) Longitudinal strains in zone II ($\varepsilon_{xx}$)  
(e) Transversal strains in zone II ($\varepsilon_{yy}$)  
(f) Shear strains in zone II ($\varepsilon_{xy}$)

Figure 3.58 $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in zone I and II

(3) Evaluation of displacements and strains of M35 on intermediate and rough supports

Tests with the intermediate and rough supports are done with M35. As shown in figure 3.59, compared with kaolin P300, there are not so many cracks. For M35 on the
rough support, there is even no crack. But the displacements are very large compared with those of kaolin P300. The displacements on the boundaries of the sample are directed towards the center. The conditions are similar on the rough support (Figure 3.59 (b)). As mentioned before, the large displacements are caused by the important shrinkage of the materials due to the presence of montmorillonite. During drying, montmorillonites shrink considerably due to the removal of water located in the interlayer spaces.

For these two cases, $\varepsilon_{xx}$ are all negative throughout the soil sample. At the boundaries, the values are larger while in the center where the displacements are small, $\varepsilon_{xx}$ are very small too. With the intermediate support, the maximum $\varepsilon_{xx}$ is 12.45%, which is smaller than the value measured with the rough support. The roughness of support affects the displacements and strains.

(a) Maps of $\varepsilon_{xx}$ on intermediate support $t = 26.67$ h

(b) Maps of $\varepsilon_{xx}$ on rough support $t = 33.33$ h

Figure 3.59 Longitudinal strains ($\varepsilon_{xx}$) on intermediate and rough support
3.3.3.2 M50

Tests with M50 on the three different supports were carried out. The initial water content is 100 %, which is equal to $w_L$. The evolution of water content on these 3 supports is shown in figure 3.60. Contrary to kaolin P300 and M35, the evolution of the water content on the rough support is more rapid than that on the two other supports.

![Figure 3.60](image)

Figure 3.60 Evolution of water content of M50 on 3 different supports

(1) Displacements and strains of M50 at different times (smooth support)

(i) $t = 26.33$ h

Near the end of desiccation ($t = 26.33$ h), the software Vic-2D cannot analyze the whole area of the images because the deformations and displacements are very large and exceed the limits of the software. We still can see that in the center of the soil sample, displacements are nearly 0 and the displacements on the boundary are very large and directed towards the center. (Figure 3.61 (a) and (b))

(ii) $t = 17.33$ h

When $t = 17.33$ h, there are a lot of small cracks. At the end of desiccation, these small cracks connect together and combine to form a large crack which crosses the sample. At the boundary of small cracks, $\varepsilon_{xx}$ are mainly compressions and change to small extensions in the positions which are more distant from the cracks. In the boundary of the soil sample, $\varepsilon_{xx}$ are compressions and very large. In most parts of the sample, especially in the center, $\varepsilon_{xx}$ are very small extensions. The maximum value is 0.7% compared to the extensions of -9.3 %, which is very small. It is the same condition for $\varepsilon_{yy}$. Near the boundary of the soil sample and the small cracks, $\varepsilon_{yy}$ are compressions while in the other parts of the sample, they are small extensions,
the maximum value being 0.6% (Figure 3.61 (c) and (d)). Positive and negative $\varepsilon_{xy}$ exist at the same time near the sides of the cracks. Compared with the value of $\varepsilon_{xx}$ and $\varepsilon_{yy}$, $\varepsilon_{xy}$ are very small, and therefore the principal directions of strains are not far from longitudinal and transversal. The maximum and minimum values are 1.74% and -1.48%, respectively. (Figure 3.61 (e)).

(iii) $t = 1 \, h$

In the early times of desiccation, U and V are very small. The maximum U is 0.226 mm and the maximum V is 0.168 mm which are observed on the boundaries of the sample. $\varepsilon_{xx}$ and $\varepsilon_{yy}$ are all extensions throughout the surface (Figure 3.62 (a)).

(iv) $t = 3 \, h$

At $t = 3 \, h$, there appear some cracks. As seen in figure 3.62 (b), near the boundary, the principal strains are large. In zone I and zone II, the principal strains are extensions. The values of strains are very large compared with those at $t = 1 \, h$. 

![Figure 3.61 Results of M50 on smooth support](image)
(e) Longitudinal strains ($\varepsilon_{xx}$), $t = 17.33h$

Figure 3.61 Results of M50 on smooth support

(a) Longitudinal strains ($\varepsilon_{xx}$), $t = 1h$

Figure 3.62 Results of M50 on smooth support at different times
Just like the analysis of M35 on smooth support, the digital image correlation with M50 on smooth support is at 17.33 h. There is no uplift phenomenon during this period. The quantitative analysis is valid.

(2) Evaluation of displacements and strains of M50 on chosen sections (smooth support)

(i) Global results

U and V vary with time during desiccation. Near the cracks, variations of displacements are obvious. The dimension of crack 2 is larger than the other two cracks and U near crack 2 is more important than near crack 1 and crack 3. For V, the variations agree well with the positions of the two cracks. (Figure 3.63 (a) and (b))

Just like in other materials, at the beginning of desiccation, $\varepsilon_{xx}$ and $\varepsilon_{yy}$ are mainly extensions. During desiccation, at the positions of cracks, extensions increase while, at the other points of the chosen sections, they turn to be compressions and increase with the development of cracks. At the boundary of the sample, compressions
are much larger than in the other parts. $\varepsilon_{xy}$ is smaller compared with the two other strains (Figs. 3.63 (c) (d) and (e)).

Figure 3.63 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions during desiccation (M50, smooth support)
Figure 3.63 U, V, \( \varepsilon_{xx} \), \( \varepsilon_{xy} \) and \( \varepsilon_{yy} \) distributions during desiccation (M50, smooth support)
(e) Distribution of shear strains ($\varepsilon_{xy}$)

Figure 3.63 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions during desiccation (M50, smooth support)

(ii) Local results

In the early times of desiccation, $\varepsilon_{xx}$ and $\varepsilon_{yy}$ are very small, they are all less than 1% when $t = 3$ h. During desiccation, at the positions of the cracks, $\varepsilon_{xx}$ are extensions and progressively increase. By the side of the cracks, $\varepsilon_{xx}$ are compressions and increase too (Fig. 3.64 (a)). Concerning $\varepsilon_{yy}$, they are extensions and increase with the development of cracks while, at the end of the tests, they turn to be compressions (Fig. 3.64 (b)). The formation of the cracks changes the strain field.

(a) Longitudinal strains in zone I ($\varepsilon_{xx}$)

(b) Transversal strains in zone I ($\varepsilon_{yy}$)

Figure 3.64 $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in zone I
(3) Evaluation of displacements and strains of M50 on intermediate and rough supports

Compared with the results of kaolin P300, there are fewer cracks in the case of M50 (Figure 3.65). The shrinkage phenomenon is quite obvious in M50 on intermediate and rough supports. The maximum U and V are very large at the end of desiccation, larger than those on the smooth support. Obviously, the large displacements are caused by the shrinkage of the sample (Figure 3.65 (a)). In some part of the support, there is an uplift phenomenon which is responsible for the numerous cracks on the rough support.

Just like in the case of M35, $\varepsilon_{xx}$ are all negative throughout the soil sample. In the center of sample, $\varepsilon_{xx}$ are very small compressions.

(a) Map of $\varepsilon_{xx}$ of M50 on intermediate support (t = 22 h)
3.3.3.3 M65

For the M65 mixture and montmorillonite M100, the contents of montmorillonite are larger than those of the other three samples. The volume of montmorillonite changes greatly when it absorbs or loses water. The numerical interpretation must be considered very cautiously with these two materials. The uplift phenomenon on intermediate and rough supports for M35 and M50 has already been observed in previous sections and figure 3.66. For these 2 materials (M65 and M100), the global analysis will be only qualitative but the local analysis is still valid as it concerns zones in which the uplift is not very important. The results which are not presented in the text because they may be affected by the vertical deformation of the sample will be given in the annex.
(a) Typical bending phenomenon seen from the vertical directions on intermediate support and rough support respectively with M35

(b) Bending phenomenon seen from the longitudinal directions on intermediate support and rough support respectively with M35

Figure 3.66 Uplift phenomenon on the different supports with M35

Tests with M65 of the 3 different supports were performed. The initial water content is 118%, which is equal to $w_L$. The evolution of water content on these three supports is shown in figure 3.67. The change in water content is less rapid with the intermediate support than that with the two other supports. At the end of the tests ($t = 80\text{ h}$), water contents on the 3 supports are constant, at about 2%.
Figure 3.67 Evolution of water content of M65 on the three different supports

(1) *Displacements and strains of M65 at different times (smooth support)*

The shrinkage of M65 on the smooth support at the end of desiccation is very large. On the boundary of crack III, the directions of the displacements are not the same. On the left side of crack III, the directions are towards the right while, on the other side, the directions are towards the bottom of the figure. It can therefore be assumed that crack III results from a tearing mode (Fig. 3.68 (e)).

Compared with kaolin P300, M35 and M50, the strains in M65 are very large. This is partly due to the presence of montmorillonite which results in large strains in the soil during drying, as shown for instance by Soemitro (1993) and confirmed in our tests (section 4.2), but also to the uplift phenomenon which tends to exaggerate the strains derived from 2D digital image correlation analysis.

In the early stages of desiccation, near crack I and crack II, there are extensions which are small (Figure 3.69 (a)). With the evolution of cracks, tractions increase in these areas.
(a) Longitudinal displacements of M65, $t = 77$ h

(b) Transversal displacements of M65, $t = 77$ h

(c) Transversal strains ($\varepsilon_{yy}$), $t = 77$ h

(d) Shear strains ($\varepsilon_{xy}$), $t = 77$ h

(e) Longitudinal strains ($\varepsilon_{xx}$), $t = 77$ h

Figure 3.68 Results of M65 with smooth support
Figure 3.69 Results of M65 with smooth support at different times

(a) Map of longitudinal strains ($\varepsilon_{xx}$), $t = 1$ h

(b) Map of longitudinal strains ($\varepsilon_{xx}$), $t = 8$ h
(c) Map of longitudinal strains ($\varepsilon_{xx}$), $t = 13$ h

Figure 3.69 Results of M65 with smooth support at different times

Before 13 hours, there is no uplift phenomenon in the analysis with M65 on smooth support, whereas at the end of desiccation, at $t = 77$ h, the uplift is quite obvious at the boundaries of the sample. The longitudinal section chosen in the center of the sample is not affected by the uplift phenomenon. The transversal section which passes through several cracks is influenced by the uplift. But the analysis section we chose between crack 1’ and 2’ is not affected by the uplift (Fig. 3.68 (e)). Under these conditions, the analysis with M65 on the smooth support is valid.

(2) Evaluation of displacements and strains of M65 on chosen sections (smooth support)

Quantitative analysis on the longitudinal section is valid between 0 mm and 300 mm. For the transversal section, the zone was limited in order to avoid the uplift phenomenon. The valid zone is between 30 mm and 140 mm where the uplift is not important.

(i) Global results

On the left side of the chosen sections the directions of $U$ are towards the right while on the other side, it is the opposite. In the center of the section, $U$ and $V$ are nearly 0 mm. In the early stages of desiccation, the displacements increase very
slowly. After about 65 hours, the displacements increase strongly (Figure 3.70 (a) and (b)).

Strains increase with desiccation. In the early times of desiccation, there are small extensions in the positions of the cracks. Then extensions turn to be compressions and increase strongly at the end of desiccation.

Figure 3.70 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions during desiccation (M65, smooth support)
Figure 3.70 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions during desiccation (M65, smooth support)
(e) Distribution of shear strains ($\varepsilon_{xy}$)

Figure 3.70 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions during desiccation (M65, smooth support)

(ii) Local results

Zone I (crack I) is located in the center of the sample and zone II (crack II) is near the boundary of the sample. Zone I is not affected by the uplift but zone II is much affected by uplift. In that case, analysis in zone II is limited to qualitative. In zone I, at the beginning of desiccation, there are extensions and compressions at the same time. For zone II, it is mainly compressions. With the development of desiccation, $\varepsilon_{xx}$ in zone I increase and then decrease. At the end of desiccation, they turn to be compressions. At about 69 h, $\varepsilon_{xx}$ in zone II increase and turn to be extensions. It is concluded that crack II is caused by tensions. At the end of desiccation, it turns to be compressions. $\varepsilon_{xy}$ for zone I is larger than that of zone II. For the crack near the boundary, the effect of shear strains is not significant.
As stated in previous sections, it is assumed that crack in zone III (crack III) results from a tearing mode (Fig. 3.68 (e)). In the early 53 hours, shear strains $\epsilon_{xy}$ are nearly 0 in zone III, whereas for zone I and zone II, shear strains progressively change. At $t =$
65 h, $\varepsilon_{xy}$ increase significantly and the maximum value reaches 3%. At the end of desiccation, at $t = 77$ h, in the section between 83 cm and 95 cm, $\varepsilon_{xy}$ remains stable with the value of 4%, which means that, in a large area of zone III, $\varepsilon_{xy}$ is homogeneous and it can be concluded that this is one of the reasons of crack appearance. And the strain direction in the clay aggregates in zone III is obviously changed because the value of $\varepsilon_{xy}$ varies from positive to negative (Fig. 3.72).

![Image](image_url)

Figure 3.72 $\varepsilon_{xy}$ distribution in zone III

(3) Evaluations of displacements and strains of M65 on intermediate and rough supports

Shrinkage is very important in the case of M65. The uplift of the clay model in its ends and near the cracks is also very large and leads to consider the results very cautiously as the displacements and strains derived from digital image correlation are probably very much influenced by this phenomenon. As seen in figure 3.73 (a), crack I on the intermediate support results from a tearing mechanism. The directions of the displacements on the two sides of the cracks are different. For crack I on the rough support, the cracking mode is also tearing. The deformations on the right side of the crack are obviously larger than on the other side.

The maximum U and V for M65 on the intermediate support are very large, larger than those measured in the case of the smooth support. In the same way as for M35 and M50, the nature of the support has a great influence on the displacements and
deformations of materials.

(a) Map of $\varepsilon_{xx}$ of M65 on intermediate support (t = 69 h)

(b) Photo of M65 on rough support (t = 69 h)

Figure 3.73 Longitudinal strains ($\varepsilon_{xx}$) on intermediate and rough support
3.3.3.4 M100

Tests were also carried out with pure montmorillonite M100 on the 3 different supports. The initial water content is 170 %, which is equal to \( w_L \). The evolution of water content on these 3 supports is shown in figure 3.74. The water content with the smooth support changes more rapidly than those on the two other supports. At the end of the tests, water contents on these 3 supports are constant, with the same value as for the other materials, approximately 2 %.

It may be concluded that the support has little effect on the evolution of water content. For kaolin P300 and M35, water content changes faster on the rough support, while for M50 and M65, evolution of water content is more rapid on the intermediate support. With M100, water content changes more quickly on the smooth support. The temperature and relative humidity are important factors during the tests, which can influence the evolution of water content results.

![Figure 3.74 Evolution of water content of M100 on the 3 different supports](image)

(1) **Displacements and strains of M100 at different times (smooth support)**

(i) \( t = 36 \) h

The process of desiccation of M100 is slower than that of the other materials because the initial water content is larger (180 %) and the permeability of the soil is lower. At \( t = 36 \) h, the maximum U and V are smaller than those of the other materials too. As shown in figure 3.75 (a) and (b), at the boundary of cracks, the directions of the displacements are opposite. The maximum and minimum displacements are situated on the two sides of the cracks.

The uplift phenomenon is very important at that time, so that displacement and
strain values derived from DIC must be considered only as indicative. There are more cracks on the boundaries of the sample than in the inner areas. Maximum $\varepsilon_{xx}$ and $\varepsilon_{yy}$ are compressions which are observed at the boundaries of the sample. Around the cracks, there are small extensions.

(ii) $t = 1\ h, 10\ h$ and $14\ h$

Until $t = 14\ h$, the uplift phenomenon at the ends of the specimen does not appear to be very large and the quantitative interpretation of the DIC results can be considered as valid, especially at some distance from the ends.

At the beginning of desiccation, when $t = 1\ h$, the displacements and strains are very small. The maximum strains are located at the boundaries of the sample and they do not exceed 0.5%. As shown in figure 3.76 (a), near the positions of crack I and crack II, there are already extensions in these areas.

Compared with the other materials, the desiccation process of M100 is very slow. At 10 hours, the maximum $U$ and $V$ are 0.21 mm and 0.11 mm. They are much smaller than those of the other materials at the same time. Maximum $\varepsilon_{xx}$, $\varepsilon_{yy}$ and $\varepsilon_{xy}$ are 2.52%, 2.58% and 0.41%, respectively. At the positions of cracks, the principal strains increase, especially the extensions (Figure 3.76 (b)).

When $t = 14\ h$, the maximum $U$ and $V$ are 0.28 mm and 0.16 mm, which is nearly the same as that of 10 h. The maximum $\varepsilon_{xx}$ is 3.42% on the boundary of the sample (Figure 3.76 (c)). Maximum $\varepsilon_{yy}$ and $\varepsilon_{xy}$ are 3.46% and 0.48%, respectively. $\varepsilon_{xy}$ is relatively small compared to the two other strains.

(a) Longitudinal displacements of M100, $t = 36\ h$  (b) Transversal displacements of M100, $t = 36\ h$

Figure 3.75 Results of M100 on smooth support
Figure 3.75 Results of M100 on smooth support

(c) Transversal strains ($\varepsilon_{yy}$), $t = 36$ h

(d) Shear strains ($\varepsilon_{xy}$), $t = 36$ h

(e) Longitudinal strains ($\varepsilon_{xx}$), $t = 36$ h
(a) Map of longitudinal strains ($\varepsilon_{xx}$), $t = 1$ h

(b) Map of longitudinal strains ($\varepsilon_{xx}$), $t = 10$ h

Figure 3.76 Results of M100 on smooth support at different times
As stated before, in the case of M100 on the smooth support, during the first 36 hours of desiccation, the effect of uplift is not very important. The quantitative analysis with VIC-2D is still valid.

(2) Evaluation of displacements and strains of M100 on chosen sections (smooth support)

(i) Global results

As for the other materials, at the beginning of desiccation, the longitudinal displacement $U$ is very small, less than 0.1 mm. During the first 25 hours, the longitudinal displacements progressively increase. The maximum $U$ is located at the right end of the sample, equal to 0.6 mm. In the other parts of the chosen section, longitudinal displacements also increase, but the rate of increase is much smaller than that on the right end of the sample. After $t = 30$ h, longitudinal displacements noticeably increase, especially around the cracks (Figure 3.77 (a)). This result corresponds well with the images of the evolutions of cracks. After 30 hours of
desiccation, there are more cracks and the length and width of cracks also increase. At \( t = 36 \) h, the maximum U is about 0.8 mm, on the left side of crack 1. For transversal displacement V, it is the same condition. At about 36 hours, the maximum V is 1.25 mm, on the left side of crack 1’ (Figure 3.77 (b)).

At the beginning of desiccation, the strains are very small. For \( \varepsilon_{xx} \) and \( \varepsilon_{yy} \), at the two ends of the chosen section, strains are larger than in the other parts. The strains are extensions at the positions of the cracks, while in the other parts of the section, they are compressions (Figure 3.77 (c) and (d)). With the development of desiccation, before 30 hours, for \( \varepsilon_{xx} \) at the positions of cracks, extensions and compressions increase at the same time. After 30 hours, on the left side of the chosen section, near crack 2 and crack 3, the extensions of \( \varepsilon_{xx} \) change to be compressions (Figure 3.77 (c)). Except at crack 1 and crack 2, \( \varepsilon_{yy} \) are obviously extensions. With the evolution of cracks, \( \varepsilon_{yy} \) are mainly compressions and the maximum value is 8% at the right ends of the chosen section (Figure 3.77 (d)). Generally \( \varepsilon_{xy} \) are much smaller than the two other strains. The maximum value is 1.1% at crack 1 at 36 h (Figure 3.77 (e)).

(a) Distribution of longitudinal displacements (U)

Figure 3.77 U, V, \( \varepsilon_{xx} \), \( \varepsilon_{xy} \) and \( \varepsilon_{yy} \) distributions during desiccation (M100, smooth support)
Figure 3.77 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions during desiccation (M100, smooth support)
(d) Distribution of transversal strains ($\varepsilon_{yy}$)

(e) Distribution of shear strains ($\varepsilon_{xy}$)

Figure 3.77 U, V, $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions during desiccation (M100, smooth support)

(ii) Local results

As shown in figure 3.75 (e), crack in zone I (crack I) is in the center of the soil sample and crack in zone II (crack II) is a boundary crack.
At the beginning of desiccation, at the position of crack I, there are extensional $\varepsilon_{xx}$. On the left and right side of the crack, they are compressions. With the development of cracks, $\varepsilon_{xx}$ progressively increases (Figure 3.78 (a)). While for crack II at the boundary, it is the opposite. In the early hours, at the position of the cracks, there are compressions $\varepsilon_{xx}$ and on the two sides of the cracks, $\varepsilon_{xx}$ are extensions. After 30 hours, at the positions of the cracks, $\varepsilon_{xx}$ turn to be extensions (Figure 3.78 (d)).

For the two cracks, $\varepsilon_{yy}$ are generally extensions and they increase with the evolution of desiccation. The maximum value of $\varepsilon_{yy}$ are 0.45% and 0.26% respectively (Figure 3.78 (b) and (e)).

$\varepsilon_{xy}$ in these two cracks increase with the development of desiccation. The maximum values are 0.8% and 0.3%, respectively, which is smaller than $\varepsilon_{xx}$ and larger than $\varepsilon_{yy}$ (Figure 3.78 (c) and (f)).
(c) Shear strains in zone I ($\varepsilon_{xy}$)

Figure 3.78 $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{yy}$ distributions in zone I and II

(3) Evaluation of displacements and strains of M100 on intermediate and rough supports

Compared with the other materials, the forms of the cracks of M100 are more orderless and the distances between the cracks are also larger. For M100 on the intermediate support, the deformations are very large and the evolution of cracks begins at the corner of the soil sample (Figures 3.79 (a) and (b)). At the beginning of desiccation with M100 on the rough support, the cracks are longitudinal because the rough support in the test is not flat like the other supports, but tend to bend. When this happens, it causes the appearance of the longitudinal cracks. At the boundaries of cracks, the directions of displacements are opposite which cause the cracks (Figure 3.79 (c)).

(a) t = 23 h
(b) t = 60 h

Figure 3.79 Transversal strains ($\varepsilon_{xx}$) on (a)(b) intermediate and (c)(d) rough support and the images of cracks at the end of desiccation
3.3.4 Comparison of longitudinal and transversal displacements of different materials (on smooth support)

As analyzed in section 3.32 and 3.33, the longitudinal (U) and transversal displacement (V) at different times are known. In the following analysis, the difference between the maximum displacements (positive U or V) and minimum displacements (negative U or V) are calculated on the same chosen longitudinal line for five materials. The difference between the maximum and minimum displacements versus time represents the evolution of cracks.

In figure 3.80, it is shown that the evolutions of the cracks of kaolin P300 is quicker than for the other materials. Generally the evolution of the differences between the maximum and minimum displacements (U and V) do not change much. The difference between the maximum and minimum U is a litter larger than that of V. In the early 9 hours, the difference between the maximum and minimum U (V) is very small, nearly 0 mm. After 9 h and 12 h, the differences between the maximum and minimum U (V) increase very quickly, to reach 5 mm and 4.5 mm respectively. In this period, a large number of cracks appears which can be verified by the images of the evolution of cracks. After that, the difference between maximum and minimum U (V) is stabilized.

Just as for kaolin P300, for M35 and M50, at the end of desiccation, the difference between the maximum and minimum U is a little larger than that of V. For these two materials, in the early 17 hours, the difference between the maximum and minimum U (V) is very small. After 17 hours, the evolution of M50 is a little quicker than that of M35. At the end of the tests, the maximum difference between the maximum and minimum U of M35 and M50 are 13 mm and 10 mm, respectively.
The free desiccation tests on M100 end after 36 hours. In this period, the evolution is exactly the same as that of M65. For M65, because of the initial water content which is larger than those of kaolin P300, M35 and M50, and because of the lower permeability of this material compared to the others, the evolutions are slower. For M65, the difference between the maximum and minimum $U$ ($V$) is 20 mm at the end of the test, which is larger for the other materials.

![Figure 3.80 Evolution of the displacements of the different materials on smooth support](image)

3.3.5 Example of result obtained with VIC-3D on kaolin P300 on the smooth support

Although the results obtained by digital image correlation in 3D should be taken very carefully, as the device was not fully calibrated before the tests and because of a lack of experience in its use, the interest of the following paragraph is to show the feasibility of this method of analysis and its interest, especially in the case of the highly swelling soils (M65, M100…) which have been shown to experience large vertical displacements during drying on intermediate and rough supports.

Although the results obtained on the digital image correlation in 3D should be taken very carefully, the interest is that they can show the feasibility of this method of analysis.

Analysis of the cracks with the software VIC-3D was performed on kaolin P300 on the smooth support. The interpretation of these tests is more complicated than with
During correlation and post-processing, Vic-3D presents a wide range of output data available for 3D and contour plotting, extraction, and export. Except longitudinal displacement (U), transversal displacement (V), longitudinal strain ($\varepsilon_{xx}$), transversal strain ($\varepsilon_{yy}$) and shear strain ($\varepsilon_{xy}$), there is another important output:

$$Z \text{ [mm]} - \text{metric displacement along the Z-axis.}$$

It is noted that the Z-scale will usually be exaggerated. It is larger than the other two scales (X and Y); the actual limits are displayed on the axis (in the following example with kaolin P300, -0.1 to 0.14 mm).

3.3.5.1 Results of Vic-3D for kaolin P300 on smooth support

As indicated before, the following results must be taken as an example and considered as indicative. It was observed in the previous paragraphs that the vertical displacements were very small in the case of kaolin P300 on the smooth support and that the material did not experience visible uplift on its boundaries. The main objective of the use of VIC-3D in that case is to provide a finer analysis of the movements of the surface mainly due to the heterogeneity (of water content, thickness, etc.) of the soil layer. Figure 3.81 presents the displacements and strains at $t = 28$ h. Like in the results in Vic-2D, the displacements at the boundaries of the sample are larger than in the other parts (figure 3.81(a) and (b)). As stated in section 3.3, the white vectors in the Vic-2D maps indicate the direction of total displacements (U and V). The black vectors in the following results also represent the total displacements of the sample (U, V and Z). The length of the vectors indicates the size of displacement. At the end of desiccation, there are various cracks. Near the main crack in the center of sample, the direction of the displacements is upwards. Closer to the crack, the total displacements are larger. At the boundaries of the sample, the displacements are also very large while the directions are downwards. This can be verified by the displacement results. Figures 3.81(b) and (d) show that at the boundaries of the cracks, the transversal and vertical displacements are very large. In the case of Z, they are the largest around the boundaries of the main crack, but they remain negligible - smaller than 0.01 mm. Around the boundaries of cracks, $\varepsilon_{xx}$ is positive and there are extensions in these areas (Figure 3.81 (d)). This verifies that the cracks are caused by tractions.
3.3.5.2 Analysis of vertical displacements with Vic-3D

Another important parameter – the vertical displacements - can be analyzed with Vic-3D. This characteristic is another advantage of Vic-3D. At the beginning of desiccation, the surface of the soil sample is not absolutely flat (4 mm). The thickness in the center of the sample is larger than in the other parts. The left and right
boundaries are also higher as indicated in figure 3.82 (a) when t = 0 h. When t = 10 h, all the vertical displacements decrease while the height in the center of the sample is still larger. The directions of Z are downwards because of the shrinkage of the sample. When t = 14 h, there are various cracks. On the left boundary and near the cracks, the directions of Z are upwards.

As we noted previously with Vic-3D, it is easy to observe the surface in 3D. The evolution of the cracks and the directions of displacements are obvious. In another aspect, the explanation and treatment of the results are more complicated than with Vic-2D.

(a) Map of vertical displacements Z (t = 0 h)

Figure 3.82 Vertical displacement of kaolin P300 at different times
Figure 3.82 Vertical displacement of kaolin P300 at different times
3.4 Analysis of the Cracks in Free Desiccation Tests

As stated in chapter 2, with the software Image-J, the total length (l), average width (d) and surface crack ratio (SCR) can be analyzed.

3.4.1 Kaolin P300 with different supports

As seen from figure 3.83, these different parameters were calculated for kaolin P300 on the three supports. The evolution of the length and ratio of cracks for the same material depends on the support. In the early stages of desiccation, the evolution of cracks is very slow. For kaolin P300 on the smooth support, before 7 h, the length of the cracks is nearly 0. Between 7 h and 12 h, their length increases more quickly. After 12 h, the desiccation is nearly stable and the length of the cracks is constant. For the rough support, the length of the cracks increases more quickly between 7 h and 10 h while, at the end of desiccation, the length of the cracks decreases a little. This may be because some of the cracks are closed at the end of desiccation. For the intermediate support, the total length is larger than for the other two supports, and reaches about 250 mm at the end of desiccation. As in the case of the smooth support, at the beginning of desiccation, the length of crack is very small. Between 9 h and 20 h, it increases dramatically (Figure 3.83 (a)).

After the first crack, the average width increases dramatically. Generally, the maximum width of the cracks on these three supports is the same. For the smooth support, the maximum width is about 0.055 mm. For the intermediate and rough supports, the maximum width is 0.65 mm at the end of desiccation (Figure 3.83 (b)).

For these three supports, the change in the ratio of cracks is the same as that of the length. The ratio of cracks on smooth and rough supports is nearly the same, 0.012%, while for the intermediate support, the value is about 0.025 % (Figure 3.83 (c)).

![Graph](image-url)  
(a) Total length of cracks versus time
It can be concluded that the roughness of the support influences the crack appearance and their evolution during desiccation. The smooth support is made of plastic on which clay tends to adhere so that the evolution of cracks is a little difficult. On the other hand, for the rough support, the friction between the soil sample and the surface of the support is higher. The development of cracks is not easy either on this support. For these two supports, the length and ratio of cracks are smaller than those on the intermediate support.

Other tests with more materials are probably necessary to confirm these conclusions.
3.4.2 Comparison between the cracks in the different materials on the smooth support

The parameters of the cracks in the tests with the three other mixtures M35, M50 and M65 on the smooth support were also analyzed.

The total length of the cracks in kaolin P300, M35 and M50 at the end of desiccation is nearly the same. The value is about 120 mm which may be related to the dimension of the model rather than the material or support. For kaolin P300, the evolution of desiccation is more rapid than for the other 3 materials. The cracks generally appear between 7 h and 12 h. After about 12 h, the length of cracks is constant. For M35, M50 and M65, the process of desiccation is quicker than for montmorillonite. During the first 18 hours of desiccation, the length of the cracks increases slowly in M35. Between 18 h and 40 h, the length of cracks increases dramatically. For M50, the evolution is the same. After 10 h, the length of cracks increases dramatically and reaches about 125 mm. Before 16 hours, it evolves slowly, to less than 10 mm. The initial water content of M65 is larger than that of the other materials so that the development of cracks in M65 is less quick. In the early 20 h of desiccation, there is practically no crack. After that, the length of the cracks increases progressively until the end of desiccation. The maximum total length of crack is about 100 mm (Figure 3.84 (a)).

With the evolution of the cracks, the average width of cracks increases dramatically. For kaolin P300, the maximum width is about 0.055 mm at the end of the test. For M35, between 10 h and 20 h, the width of the cracks rises dramatically and reaches 0.07 mm. The value is smaller for M50. At 3 h, the width reaches about 0.04 mm, after that, it increases progressively to 0.06 mm. For M65, the average width of cracks is larger than for the other three materials. The process is also more progressive (Figure 3.84 (b)).

The tendencies of the ratio of cracks are the same as that of length of cracks (Figure 3.84 (c)).

To conclude, the montmorillonite fraction in the mixture plays an important part in the kinetic of the crack development before stabilization. This can be explained by the increase in the plastic properties and the decrease in permeability with the montmorillonite percentage, the crack development becoming thus slower and slower during the free desiccation process.
(a) Total length of cracks versus time

(b) Average width of cracks versus time

(c) Surface crack ratio versus time

Figure 3.84 Parameters of the cracks for kaolin P300, M35, M50 and M65
3.5 Traction tests

Traction tests were carried out on three materials: kaolin P300, M35 and montmorillonite M100. The original water content of these materials are 40%, 82% and 180%, respectively corresponding to their liquid limit \( w_L \). Then, they were let to dry during some time and their water content was homogenized in order to study the effect of initial water content on tensile behavior. The maximum displacement in traction tests is 20 mm (see chapter 2).

3.5.1 Stress-train relationships under controlled deformation conditions

3.5.1.1 Kaolin P300

As seen in figure 3.85, tensile stress increases with displacement. With the decrease of water content, the maximum tensile stress increases. For kaolin P300 with the water contents of 39%, 31% and 26%, the maximum tensile stress is 9 kPa, 11 kPa and 14 kPa respectively.

For these three different water contents, at the beginning of traction tests, the deformation remains very close to 0 when the tensile stress increases. This is the first period during traction. After this period, the deformation increases dramatically in a very short time. For kaolin P300 with the water content of 39%, the deformation increases to 3.7% when tensile stress is 3.7 kPa. When the water content is 31%, the deformation turns to 4% for a tensile stress of 3.7 kPa. With a smaller water content of 26%, deformation increases more evidently, to 9.5% when the tensile stress is 5.5 kPa. This is the second period of traction. After that, deformation increases progressively with the tensile stress until the end of the test, which is the third step of traction. At the end of traction, the sample in the center of the device is totally broken and displacement increases very quickly. The tensile stress value corresponds to the maximum value of the traction force.
Just like in the case of kaolin P300, the maximum tensile stress increases with the decrease of water content. For M35 with the highest water contents of 90% and 85%, there are also three different traction periods (Fig. 3.86). With the water content of 90%, the deformation increases dramatically to 15% when the tensile traction is 3.1 kPa. With the water content of 85%, the deformation turns to 7.7% and the tensile stress is 3.3 kPa. With the other smaller water contents, there is no phenomenon of dramatically increasing deformation. The maximum tensile stresses are 10.3 kPa, 9.3 kPa and 8.7 kPa, respectively, when the water content is 53%, 56% and 59%. And the deformation increases very little when the tensile stress increases strongly. The deformations are very large. The rupture of the sample occurs very slowly with a strongly increasing tensile stress. When the water content is higher, the tensile stress is much smaller. With water contents higher than 70%, the maximum tensile stresses are less than 6 kPa. And the cracks develop very quickly in the sample.
3.5.1.3 Montmorillonite

Maximum tensile stress also increases with the decrease of water content. With the smallest water content of 81%, the maximum tensile stress is 12 kPa, while for the maximum water content of 176%, the maximum tensile stress is just 3.2 kPa. For the sample with water contents of 176% and 153%, the first period of traction can be observed. When the water content is less than 100%, the process of rupture of the samples is very progressive and the tensile stress increases during a very long time. For larger water contents, it is the opposite (Fig. 3.87).
3.5.2 Comparison of the results

3.5.2.1 Relationship between tensile strength and water content

As seen in figure 3.88, at the same water content, the maximum tensile stress of montmorillonite is larger than that of M35. For kaolin P300, the maximum tensile stress is the smallest among these three materials. With the increase in the percentage of montmorillonite, the maximum tensile stress increases too.

The liquidity index ($I_L$) is used for scaling the natural water content of a soil sample with respect to the liquid and plastic limits. The effects of the water content on the strength of saturated remolded soils can be quantified by using the liquidity index. When the $I_L$ is 1, the remolded soil is at the liquid limit and it has a very small undrained shear strength (for instance, about 2 kPa). At the same liquidity index, the maximum tensile stress of kaolin P300 is larger than those of M35 and montmorillonite.

![Figure 3.88 (a) Maximum tensile stress versus water content](image)

Figure 3.88 (a) Maximum tensile stress versus water content
3.5.2.2 Relationship between tensile strength and suction

Water content is related to both maximum tensile stress and suction. In Taibi (1994)'s model, for instance, the tensile strength of a soil is equal to the capillary stress, a non-linear function of suction which depends on a characteristic dimension of the material. The relationship between suction and maximum tensile stress is plotted in figure 3.89, using the relationship between water content and suction derived from suction-controlled drying tests. For the 3 studied materials, the maximum tensile stress increases in a non-linear way with suction. When plotted versus the logarithm of suction, the relationship appears to be linear, with nearly superimposed lines for kaolin P300 and M35 mixture and a line located slightly below for montmorillonite M100. The effect of suction on tensile strength therefore seems qualitatively similar to what is observed in the case of unconfined compression strength, for instance. The reason is the fact that the tensile strength is related to the capillary stress, which is a non-linear function of suction, as shown by many authors (e.g. Taibi et al. 2008).
Figure 3.89 Relationship between maximum tensile stress and suction

(a)

(b)

Figure 3.89 Relationship between maximum tensile stress and suction
3.6 Conclusions

In this chapter, water retention characteristics, experiments of free desiccation interpreted through digital image correlations, characteristics of the cracks and traction tests on different mixtures have been studied. It is important that the macroscopic results in this chapter can be related with the microscopic research in chapter 4.

The following conclusions can be derived from the macroscopic results of the tests:

1. Two methods (DVS and classical method) are used in order to analyze the drying/wetting path of the materials. On drying paths, starting from a saturated slurry prepared at \( w = w_L \), all the soils remain saturated or quasi-saturated up to a large suction exceeding 1 MPa.

Under such conditions, results in the \([w - e]\) plane show that the shrinkage limit void ratio \( e_{SL} \) for kaolin P300, M35, M50, M65 and M100 are 0.3, 0.5, 0.6, 0.8 and 1.1, respectively. In the \([\log(s) - e]\) plane, the compression indices (Cc) for these 5 materials are 0.241, 0.65, 1.11 and 1.32, respectively, similar to the values obtained by Hammad (2010) in oedometric tests. The shrinkage limit suctions deduced from the \([\log(s) - w]\) plane, range from 1.3 to 5 MPa, corresponding to shrinkage limit water contents between 16 and 41%. Desaturation suction (air entry pressure) deduced from the relation between suction and degree of saturation are comprised between 1.2 and 113 MPa. The effect of the montmorillonite content on all these features is very important.

2. A first series of free desiccation tests on kaolin P300 and M50 was devoted to the determination of the average water contents, local water contents and deformation evolution.

The average water contents decrease with time. And in the center of the sample, local water content is a little larger than on the boundaries. The maximum difference is 1.46%. The comparison between transversal, longitudinal and vertical deformations versus water content shows that the sample shrinks a little more in the vertical direction than in the two other directions because of the friction on the surface of the support.

3. Photos of the soil surface in the second series of free desiccation tests were taken at regular intervals and analyzed by correlation of digital images using the softwares Vic-2D and 3D. Five materials and three different supports were studied in these tests.

Two-dimensional strains and displacement maps are obtained with Vic-2D. The zones of the sample where cracks appear are identified. And the evolution of strains and displacements before the appearance of cracks is also analyzed. In kaolin P300,
the cracks evolve more quickly than in the other materials according to the displacement and strain maps. Two modes of cracks are detected during the tests: traction mode and tearing mode.

Analysis of the cracks with the software VIC-3D was also performed on kaolin P300 on the smooth support. The evolution of vertical displacements, in addition of the displacements in the plane, was analyzed. Near the boundaries of the cracks, the direction of displacements can be observed.

(4) With the software Image-J, the total length (l), average width (d) and ratio of cracks (r) (the ratio between the surface of cracks and the total surface of the sample) were analyzed. It is concluded that the roughness of the support influences the crack appearance and their evolution during desiccation.

(5) Traction tests were carried out on three materials: kaolin P300, M35 and montmorillonite. For a given material, tensile strength increases with the decrease of water content. With the increase in the percentage of montmorillonite, the maximum tensile stress decreases at the same liquid index.
CHAPTER 4 MICROSCOPIC RESULTS

The microscopic approach that we propose in this thesis consists in following the microstructural evolution of the material, especially the orientation of the clay particles and the pore diameter distribution, as a function of its hydric state on a drying path.

At the macroscopic scale, the study is based on controlled-suction techniques and, at the microscopic level, on scanning electron microscopy coupled to an adapted method for the identification of the preferential orientations of the particles and mercury intrusion porosimetry, a method which allows to quantify the pore space and void ratio.

4.1 CORE-SHELL STRUCTURE OF THE SPECIMENS AFTER DRYING

After the specimens have been freeze-dried, a special phenomenon is observed (Fig. 4.1). The specimens are found to have core-shell structures. Core-shell structures have received considerable attention because it has been demonstrated that the shell can significantly alter the core properties in biology and other domains.

The same phenomenon was also observed by Yoshinaka and Kazama (1973). It appears that this structure corresponds to the turbostratic structure defined by Aylmore and Quirk (1960) and the curved trajectories of Sloane and Kell (1966). The form of blocks may be deformed and elongated, to various degrees, by compaction or imposed suctions.

This phenomenon is found in M35, M50, M65 and M100 materials under different suctions, but is not visible in kaolin P300, which suggests that the phenomenon may be due to the presence of montmorillonite. Indeed, in kaolinite, piled-up particles are linked to each other by strong hydrogen and van der Waals bonds and the resulting crystallites are stiff plates with a lateral extension of a few hundreds of nm and a thickness of a few dozens of nm (van Damme, 2001). In the case of montmorillonite, the layers are much larger and hydrated cations can enter between the layers which are less strongly bonded together. Thus, the particles of montmorillonite are much more flexible than those of kaolin P300 (refer to Fig. 2.1) and, under suction, they are easier to bend and undergo larger deformations than those of kaolinite.

Under increasing suctions, the volume of the specimens becomes smaller and smaller. As the specimens were initially prepared as slurries, they must have a rather isotropic initial microstructure if the effect of gravity is neglected. We will show in the following section that suction results in globally isotropic strains. This may be the
cause of the layers in the form of circles found in the specimens. Hammad (2010)
showed that, in this type of material, micro-cracks form themselves at the ultimate
state of triaxial paths. Those micro-cracks, in form of groups of particles arranged
face to face along a privileged oriented plane, mainly occurred in the regions of
montmorillonite fraction. The shape obtained on figure 4.1 may be the result of the
friction between the particles, producing tractions which lead to the formation of
cracks (Figs. 4.1(a), (g) and (j)). For larger suctions, the cracks between the layers are
not obvious (Figs. 4.1(b), (c), (e), (f), (h), (i), (k) and (l)). It can be noted that there is
also friction between the bottom of the specimens and the supports on which they are
placed in the different devices used to impose suctions. So it is observed that the
core-shell structures are not that obvious in the part of the specimens which is nearer
to the supports.
Figure 4.1 Core-shell structures of M35, M50, M65 and M100 under different suctions
Figure 4.1 Core-shell structures of M35, M50, M65 and M100 under different suctions
4.2 RESULTS OF MERCURY INTRUSION POROSIMETRY TESTS

4.2.1 Kaolin P300

The evolution of the differential and cumulative mercury intrusion volumes versus pore diameter for the different suctions, in the case of the kaolin P300, is shown in figure 4.2. When the suction of the specimen is equal to 1 kPa and 10 kPa, the most frequent diameter is the same (0.227 μm). The cumulative volumes of mercury under the suctions of 1 kPa and 10 kPa are also very close, which means that the structure of kaolin P300 under these two small suctions does not change much. For the suctions of 100 kPa and 1500 kPa, the most frequent diameters are quite similar (0.2836 μm). Then they decrease progressively to 0.15 μm for the suction of 158 MPa. Under the suction of 1 kPa, a second family of pores appears with a diameter smaller than 0.15 μm. Figure 4.2(b) shows the cumulative volume of mercury introduced into the sample. The volume of mercury gradually decreases according to the increase in suction due to the decrease in porosity. The very similar values of the predominant pore diameters for the suctions of 1 kPa, 100 kPa and 1500 kPa mean that, as long as the soil remains saturated or nearly saturated, suction does not modify significantly the predominant diameter of the pores. Under the suction of 1 kPa, the large difference observed in the cumulative volumes at the end of the tests is due to the second family of pores smaller than 0.15 μm. Under the suction of 10 kPa, the condition is exactly the same as under 1 kPa.

With the equation given by the law of Laplace: \( u_a - u_w = \frac{2\sigma_{cw}\cos\theta}{r} \), the diameter of the pores \( d = 2r \) corresponding to the air-entry pressure in the suction-controlled
tests can be calculated in the $[s - Sr]$ plane. The diameter of the pores is 0.25 μm.

For kaolin P300 under the suction of 1500 kPa with the MIP method, the main diameter is 0.284 μm which is very near to the results derived from suction-controlled tests.

4.2.2 M35

The evolution of the differential and cumulative pore volumes of the M35 mixture is shown in figure 4.3. Under the suctions of 1 kPa and 20 kPa, the predominant pore diameter is the same, equal to 1.05 μm. When the suction of the specimen increases to 100 kPa and 1500 kPa, the most frequent diameter is comprised between 0.43 and 0.18 μm. It decreases to 0.15 μm when the suction reaches 158 MPa.

Figure 4.3 (b) shows the cumulative volume of mercury introduced into the sample. The volume of mercury gradually decreases according to the increase in suction due to the decrease in porosity. For M35 under 1 kPa, on the left side of the curve, the curve is completely flat which means that there are no pores smaller than 6 nm. Under the suction of 1500 kPa and 158 MPa, the cumulative volumes at the end of the tests are similar. For M35 under 20 kPa, 100 kPa, 1500 kPa and 158 MPa suctions, the curves present slopes which means that there are smaller pores which are not measured.

There is an increase in the cumulative volume for the large diameters for M35 under 1 kPa, 20 kPa and 1500 kPa compared with the other two suctions. This could be due to the presence of cracks in the samples which can be observed on the SEM photos in the following sections.
4.2.3 M65

The evolution of the differential and cumulative pore volumes of the M65 mixture is shown in figure 4.4. Under the suction of 1 kPa, the predominant pore diameter is 0.28 μm. This diameter decreases to 0.08 μm when suction reaches 158 MPa. Moreover, figure 4.4 also shows that, contrary to M35, M65 features another smaller pore family, whose diameter is around 0.13 μm under the suction of 1 kPa and between 0.01 and 0.03 μm under the suction of 158 MPa. Figure 4.4(b) shows the cumulative volume of mercury introduced into the sample. The volume gradually decreases according to the increase in suction. It can be concluded that suction affects all the sizes of pores, from the small pores to the large pores. Under larger suction, the large pores become smaller, and the originally small pores become even smaller (Fig. 4.4(a)). Under the suction of 1 kPa, the cumulative volume appears to be small compared to the others which is probably due to the preparation of the sample. Under the suction of 100 kPa, the predominant pore diameter is 0.55 μm, which is the largest among all of them under different suctions. The cumulative volume of mercury introduced into the specimen under this suction is the largest compared with that under the other suctions.

The results confirm the general conclusions of many studies that the changes occurring in small pores are related to large suctions or pressures. As loading or suction increases, the pores with smaller diameters tend to become more frequent and the cumulative intrusion volume increment becomes smaller and smaller.

4.2.4 M100

The evolution of the differential and cumulative pore volumes of the pure montmorillonite M100 is shown in figure 4.5. Under the suction of 100 kPa, the predominant pore diameter is 0.68 μm. This diameter decreases to 0.42 μm when
suction reaches 1500 kPa. Figure 4.5 (b) shows the cumulative volume of mercury introduced into the sample. The volume of mercury gradually decreases according to the increase in suction due to the decrease in porosity.

For the cumulative pore volume curve under 1500 kPa, there is a large increase in the large diameter domains. There is a possibility of the presence of cracks in the sample which will be verified in the SEM observations in the following sections.

![Figure 4.5](image1.png)

(a) (b)

Figure 4.5 Evolution of the pore space under different suctions in M100

4.2.5 Comparison between the MIP results for the different materials under the same suction

In order to compare the differences of the porosities of the different materials under the same suction, we chose 3 suctions: 100 kPa, 1500 kPa and 158 MPa.

4.2.5.1 Under the suction of 100 kPa

The evolution of the pore diameter of the four materials under the suction of 100 kPa is shown in figure 4.6. Under the same suction, the most frequent diameters of kaolin P300, M35, M65 and M100 are 0.28, 0.43, 0.55 and 0.68 μm, respectively. They increase progressively with the increase in montmorillonite content. For kaolin P300 under the suction of 100 kPa, just as under the suction of 1 kPa, as was explained in section 4.3.1, a second family of pores appears with a diameter smaller than 0.15 μm. This second family is not observed in the other materials. Figure 4.6 (b) shows the cumulative volume of mercury introduced into the sample. The volume of mercury gradually increases with the montmorillonite content, which means that the porosity of the materials also increases with the increase in the percentage of montmorillonite.

In the same way for the global void ratios (e) of the materials which are calculated
in chapter 3, the void ratios deduced from the results of porosimetry are also calculated using the following equation:

\[
e = \frac{n}{1-n} = \frac{\rho_h \cdot a}{1-\rho_h \cdot a} = \frac{\rho_s (1+w) \cdot a}{1+e' \cdot \rho_s (1+w) \cdot a} = \frac{\rho_s (1+w) \cdot a}{1+e' \cdot \rho_s (1+w) \cdot a}
\]

(Equation 4.1)

where:
- \(e\) = void ratio deduced from the results of porosimetry;
- \(n\) = porosity of the materials;
- \(\rho_h\) = wet density of the materials;
- \(a\) = cumulative volume of mercury;
- \(\rho_s\) = density of the solids;
- \(e'\) = global void ratio (derived from the measurement in oil);
- \(w\) = water content corresponding to the void ratio;

Using equation 4.1, it is concluded that the void ratios of kaolin P300, M35, M65 and M100 under 100 kPa derived from the results of porosimetry are 0.40, 0.81, 1.68 and 2.05 respectively (Table 4.1). The results confirm that the void ratio increases with the increase in the percentage of montmorillonite, which corresponds to the cumulative volume of mercury introduced into the specimens. As was explained in chapter 2, montmorillonite is much more swelling than kaolinite. This confirms that the percentage of montmorillonite affects the sizes of pores.

The porosities deduced from MIP are smaller than those derived from global measurements, as is usually observed, due to the difficulty of the mercury to enter the smallest and the closed pores (Souli, 2006).

Figure 4.6 Evolution of the pore space of the different materials under the suction of 100 kPa
Table 4.1 Void ratios deduced from the results of porosimetry under the suction of 100 kPa

<table>
<thead>
<tr>
<th></th>
<th>$\rho_s$ (g/cm$^3$)</th>
<th>w</th>
<th>a (mL/g)</th>
<th>$e'$-oil</th>
<th>e-MIP</th>
</tr>
</thead>
<tbody>
<tr>
<td>kaolin P300</td>
<td>2.65</td>
<td>0.33</td>
<td>0.291</td>
<td>0.88</td>
<td>0.40</td>
</tr>
<tr>
<td>M35</td>
<td>2.67</td>
<td>0.45</td>
<td>0.424</td>
<td>1.32</td>
<td>0.81</td>
</tr>
<tr>
<td>M65</td>
<td>2.70</td>
<td>0.79</td>
<td>0.518</td>
<td>1.91</td>
<td>1.68</td>
</tr>
<tr>
<td>M100</td>
<td>2.73</td>
<td>0.75</td>
<td>0.557</td>
<td>2.19</td>
<td>2.05</td>
</tr>
</tbody>
</table>

4.2.5.2 Under the suction of 1500 kPa

The evolution of the pore diameters of the 4 materials under the suction of 1500 kPa is shown in figure 4.7. For kaolin P300 and M35, the predominant pore diameter is comprised between 0.28 and 0.18 μm. Then it increases to 0.35 μm with the increase in the percentage of montmorillonite (M65). The predominant pore diameter is 0.33 μm for M100, which is not very different from that of M65. Figure 4.7 (b) shows the cumulative volume of mercury introduced into the sample, which gradually increases according to the increase in montmorillonite content.

As seen in the cumulative pore volumes, the porosity increases with the proportion of montmorillonite, but the curves do not present a progressive increase. The variation of porosity seems to be sudden. As stated before, the curves of M100 and M65 are very similar. The result with M35 is relatively away from these two curves. For M65, porosity for a given suction does not change significantly. In the research of Hammad (2010), oedometric tests on the different mixtures under 1000 kPa vertical stress (which is near to my tests) were also carried out. The maximum cumulative pore volumes are 0.2, 0.2 0.25 and 0.45 mL/g for kaolin P300, M35, M65 and M100, respectively. The porosity also increases with the percentage of montmorillonite.

The void ratios of kaolin P300, M35, M65 and M100 under 1500 kPa suction deduced from the results of porosimetry are 0.85, 0.58, 1.41 and 0.92, respectively (Table 4.2). For M65 and M100, a large volume of mercury is introduced into the sample for diameters between 10 and 100 μm. This may be caused by the micro-cracks in the sample. The void ratio of M100 is relatively smaller than the average value of montmorillonite under the same suction, which may be due to the preparation of the samples.
Figure 4.7 Evolution of the pore space of the different materials under the suction of 1500 kPa

Table 4.2 Results of void ratio deduced from the results of porosimetry under the suction of 1500 kPa

<table>
<thead>
<tr>
<th></th>
<th>$\rho_s$ (g/cm$^3$)</th>
<th>$w$ (mL/g)</th>
<th>$a$ (mL/g)</th>
<th>$e'$ - oil</th>
<th>$e$ - MIP</th>
</tr>
</thead>
<tbody>
<tr>
<td>kaolin P300</td>
<td>2.65</td>
<td>0.30</td>
<td>0.236</td>
<td>0.77</td>
<td>0.85</td>
</tr>
<tr>
<td>M35</td>
<td>2.67</td>
<td>0.42</td>
<td>0.213</td>
<td>1.21</td>
<td>0.58</td>
</tr>
<tr>
<td>M65</td>
<td>2.70</td>
<td>0.44</td>
<td>0.355</td>
<td>1.36</td>
<td>1.41</td>
</tr>
<tr>
<td>M100</td>
<td>2.73</td>
<td>0.61</td>
<td>0.317</td>
<td>1.91</td>
<td>0.92</td>
</tr>
</tbody>
</table>

4.2.5.3 Under the suction of 158 MPa

The evolution of the pore diameters of kaolin P300, M35 and M65 under the suction of 158 MPa is shown in figure 4.8. The most frequent diameter for kaolin P300 and M35 is the same, equal to 0.15 $\mu$m. It decreases to 0.08 $\mu$m for M65. Using equation 4.1, it is concluded that the void ratios of kaolin P300, M35 and M65 under 158 MPa deduced from the results of porosimetry are 0.79, 0.91 and 0.44, respectively (Table 4.3). The results confirm that the void ratio increases with the percentage of montmorillonite, which corresponds to the cumulative volume of mercury introduced into the specimens. The cumulative pore volume curve of P300 and M35 are very near, while the difference between M65 and the other two curves is a little larger.
Table 4.3 Results of void ratio deduced by the results of porosimetry under the suction of 158 MPa

<table>
<thead>
<tr>
<th>Material</th>
<th>$\rho_s$ (g/cm$^3$)</th>
<th>w</th>
<th>a (mL/g)</th>
<th>$e'$ - oil</th>
<th>$e$ - MIP</th>
</tr>
</thead>
<tbody>
<tr>
<td>kaolin P300</td>
<td>2.65</td>
<td>0.04</td>
<td>0.227</td>
<td>0.42</td>
<td>0.79</td>
</tr>
<tr>
<td>M35</td>
<td>2.67</td>
<td>0.24</td>
<td>0.244</td>
<td>0.70</td>
<td>0.91</td>
</tr>
<tr>
<td>M65</td>
<td>2.70</td>
<td>0.20</td>
<td>0.15</td>
<td>0.59</td>
<td>0.44</td>
</tr>
</tbody>
</table>

4.3 Evolution of the orientation of the clay particles under different suctions

4.3.1 Analysis of scanning electron microscope (SEM) images

The void ratios of kaolin P300, M35, M65 and M100 under 1 kPa, 1500 kPa and 158 MPa, derived from the measurements in oil, are presented in table 4.4. As indicated in the previous paragraph, the void ratio decreases when the suction increases and increases with the montmorillonite content in the mixture. An anomaly is observed in the case of M65 under the highest suction, for which the void ratio is too small. This anomaly is also observed in the value derived from MIP tests and must probably be attributed to some drying of the specimen.

The numbers of particles in the observed zone for these five materials are summarized in table 4.5. The percentage of particles seen from the side is presented in figure 4.9. The percentages of particles seen from the side are generally much larger than those of the particles seen in front. The difference is more obvious in mixed materials (M35 and M65), while in kaolin P300 and M100, the difference between the percentages of these two kinds of particles is smaller.
Table 4.4 Void ratio of different materials under different suctions

<table>
<thead>
<tr>
<th>Materials</th>
<th>Suctions</th>
<th>1 kPa</th>
<th>1500 kPa</th>
<th>158 MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kaolin P300</td>
<td>0.96</td>
<td>0.86</td>
<td>0.41</td>
<td></td>
</tr>
<tr>
<td>M35</td>
<td>2.05</td>
<td>1.21</td>
<td>0.70</td>
<td></td>
</tr>
<tr>
<td>M65</td>
<td>3.04</td>
<td>1.36</td>
<td>0.59</td>
<td></td>
</tr>
<tr>
<td>M100</td>
<td>5.14</td>
<td>1.91</td>
<td>1.07</td>
<td></td>
</tr>
</tbody>
</table>

Table 4.5 Number of particles seen in front and from the side

<table>
<thead>
<tr>
<th>Suctions</th>
<th>Kaolin P300</th>
<th>M35</th>
<th>M65</th>
<th>M100</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 kPa</td>
<td>seen in front 380</td>
<td>251</td>
<td>252</td>
<td>170</td>
</tr>
<tr>
<td></td>
<td>seen from the side 242</td>
<td>465</td>
<td>202</td>
<td>191</td>
</tr>
<tr>
<td>1500 kPa</td>
<td>seen in front 345</td>
<td>160</td>
<td>192</td>
<td>113</td>
</tr>
<tr>
<td></td>
<td>seen from the side 316</td>
<td>576</td>
<td>313</td>
<td>176</td>
</tr>
<tr>
<td>158 MPa</td>
<td>seen in front 405</td>
<td>168</td>
<td>145</td>
<td>113</td>
</tr>
<tr>
<td></td>
<td>seen from the side 267</td>
<td>567</td>
<td>451</td>
<td>127</td>
</tr>
</tbody>
</table>

Figure 4.9 Percentage of particles seen from the side for different contents of montmorillonite

From the photos of scanning electron microscope (SEM) (Figs. 4.10 (a, b, c)), it is quite obvious that, for the sample of kaolin under the suction of 1 kPa, the porosity and void ratio are much larger than under larger suctions. In the aspect of structure, the clay minerals appear in the form of clusters of a few micrometers, which appear to
take the shape of the larger grains nearby in figure 4.10 (c). Many particles can be observed in front direction in the form of tortoiseshell (Figs. 4.10 (b) (c)).

With the M35 and M65 materials (Figs. 4.10 (d, e, f, g, h, i)), the phenomenon is the same as with kaolin P300. The porosity and void ratio under the smallest suction are obviously higher than those under higher suctions. No preferred orientation of particles can be observed in this case. In M65, the presence of a family of smaller pores can be observed in these photographs. This is confirmed by the results of the MIP tests in section 4.2.

SEM results for the M100 material are shown in figures 4.10 (j, k, l). For the sample under 1 kPa, it is also quite obvious that the porosity and void ratio are much larger than under higher suctions. The particles of kaolinite are much more rigid than the particles of montmorillonite. It is not easy to distinguish the form and the directions of the montmorillonite particles. Under 1 kPa, 1500 kPa and 158 MPa suctions, the number of particles is 361, 289 and 240 in the observed zone, respectively. The percentage of particles seen from the side is 52.9%, 60.9% and 52.9% for these three suctions.
Figure 4.10 SEM photos of kaolin P300, M35, M65 and M100 under different suctions

(a) P300-1 kPa
(b) P300-1500 kPa
(c) P300-158 MPa
(d) M35-1 kPa
(e) M35-1500 kPa
(f) M35-158 MPa
Figure 4.10 SEM photos of kaolin P300, M35, M65 and M100 under different suctions
Under the highest suction (158 MPa), cracks appear in the materials, as shown in figure 4.11. This corresponds to the lowest water content of all the samples under three different suctions (1 kPa, 1500 kPa and 158 MPa) where the cracks are more frequent. For kaolin P300 and M35, the cracks are open whereas, in M65, they are closed. As explained in section 4.1, in kaolinite, the crystallites are stiff plates while the particles of montmorillonite are much more flexible. Below the shrinkage limit, capillary phenomenon results in tensile forces in the materials. Tensile tests were carried out on the three materials and showed that, at the same water content, the tensile strength of kaolin P300 and M35 was much lower than that of M65, which means that failure appears first in kaolin P300 and M35 with the particles of montmorillonite filling the spaces of cracks (section 3.5). The content of montmorillonite in M65 is higher than that in M35, which means there are more montmorillonite particles filling the spaces of cracks and may explain that the cracks in M35 are open whereas those in M65 are closed. This makes the cracks appear differently in kaolin P300, M35 and M65.

Under high suction (158 MPa – the driest state) (Figs. 4.13 (b) (d) and (f)), the SEM photos show a dense clay matrix more or less unstructured, where numerous closed cracks appear to consist of clayey particles which are arranged face to face.

We expected to find cracks in M100 under 158 MPa, but the cracks are not observed in M100 under 158 MPa suction whereas they are found under 1500 MPa (Figs. 4.11 and 4.12). The cracks are open. This may be due to the fact that the particles of montmorillonite are more flexible.
Figure 4.11 Cracks in soils under 158 MPa suction and different magnifications
(a) Kaolin P300 ×1000; (b) Kaolin P300 ×3000; (c) M35 ×1000; (d) M35×3000;
(e) M65 ×1000; (f) M65×3000
4.3.2 Orientation of the particles under suction

Microscopic observations were performed after imposing suctions of 1 kPa, 1500 kPa and 158 MPa to the kaolin P300, M35, M65 and M100 materials. Figures 4.14 (a) (c) (e) and (g) present the percentage of particles as a function of their orientation represented by the angle $\theta$. $\theta$ is measured with respect to the horizontal plane, perpendicular to the loading axis. As stated in chapter 2, image processing of the diagram thus obtained allows the orientation of all the represented particles to be
calculated. Then, an angular distribution diagram can be drawn as a rose diagram, giving the percentage of particles seen from the side as a function of their orientation.

The “D” mode designates the structural isotropy of a fictional material represented in figures 4.14 (a) (c) (e) and (g) by the “D” line (where all the orientations are represented by the same percentage corresponding in our case to 5.5%). The mechanism of random orientation of the particles is indicated here by the term “depolarization” as defined by Hattab and Fleureau (2010, 2011).

The SEM photographs highlight a decrease in the pore space when suction increases, compared to the initial state (1 kPa).

4.3.2.1 Global analysis of the particles orientation under suction

Qualitative analysis of the SEM photographs of the specimens at the suction of 1 kPa, 1500 kPa and 158 MPa was performed. The global rose diagrams shown in figures 4.14 (b) (d) (f) and (h) result from several observations carried out at different points on the observed planes.

For kaolin P300, the global rose diagram derived from the analysis on the observed plane (Fig. 4.14 (b)) indicates an isotropy without a preferential orientation of the particles. One can notice a few points quite distant from the D line, corresponding to particles oriented towards 50-60° (for 1 kPa), 150-160° (for 1500 kPa) and 140-150° (for 158 MPa); their percentage, however, remains just a little higher than those corresponding to the other particles orientations.

For M35, the orientation of the particles also indicates that the sample is globally isotropic (Fig. 4.14 (c)). The global rose diagram derived from the analysis on the observed plane (Fig. 4.14 (d)) shows an isotropy without any preferential orientation of particles. A seen in the kaolin case, a few particles oriented in the 110-120° (1 kPa), 100-110° (1500 kPa) and 120-130° (158 MPa) ranges can be observed. They are a little more obvious than for the other angles.

The orientation of the particles in M65 and M100 remains globally isotropic (Figs. 4.14 (f) (d)). The SEM photographs taken on the observed plane show a structural isotropy without a preferential orientation of the particles.

The angular distribution rose diagram on the observed plane may be analyzed in terms of orientation curves (Fig. 4.14 (d)), giving the percentage $P$ of particles viewed from the side as a function of their orientation $\theta$. This kind of representation allows the curves corresponding to different suctions to be superimposed in order to compare them. From their clear wave shape, the curves obtained under the lowest suction and represented in Figs. 4.14 (b) (d) (f) and (g) clearly confirm the isotropy of structure for all the studied materials. The analysis of the SEM photographs highlights a decrease in the pore space compared to the lowest suction, but with the same random orientation of the particles for these four materials.
4.3.2.2 Comparison of the effects of suction and stress on the orientation of the particles

Partly because of the lack of experimental data, especially in clayey materials, incorporating in continuous models the local mechanisms that occur at the microscopic scale remains a complex approach. In this way, work can be found in the literature, in particular for granular material, where fabric is regarded as a tensor representing the anisotropy of the granular packing (Hattab & Fleureau, 2010). Hammad (2010) analyzed the orientation of particles for different clays on isotropic path.

In my research, it is the effect of suction on the orientation of the particles which is analyzed. My results are compared with those of Hammad (2010) on M35 and M65 on isotropic path. Two isotropic stresses (200 and 600 kPa) were applied. The results (Fig. 4.15) show that except for montmorillonite, all the other materials present a preferred orientation. The effect of initial state is concluded. In fact, in Hammad’s tests, before the isotropic tests, the specimens were prepared by uniaxial consolidation in a mold under 100 kPa vertical stress. The stresses applied during the isotropic tests were probably not sufficient to erase the memory of the soils, so that the measured orientations were probably the result of the fabric imposed by the uniaxial consolidation rather than the effect of the isotropic consolidation.
The conclusion is that, when the isotropic stress is not much higher than the vertical stress applied during uniaxial consolidation, the fabric is imposed by the initial consolidation whereas, when the isotropic stress is sufficiently increased, the fabric becomes more and more isotropic. The effect of anisotropy caused by the one-dimensional consolidation vanishes when the isotropic consolidation stress increases.

This verifies the fact that if the sample is under an isotropic stress, there is a tendency towards isotropic organization. The results obtained under isotropic suctions in my tests confirm this conclusion.

4.3.2.3 Local analysis of the orientation of the particles under suction

The analysis of the orientation of the particles was also performed at a local level, i.e. considering the area of one photograph only in order to see whether the depolarization is also active locally.

On the SEM photos of kaolin P300, M35, M65 and M100 under 1 kPa suction, (Figs. 4.16 (a) (c) (e) (f)), the pores can be seen very clearly. The number of particles for these four materials is 145, 250, 173 and 134, respectively. The preferential angles for kaolin P300 seems to appear at 10-20°, 120-130° and 140-150° (Fig. 4.16 (b)). For M35, a few particles oriented in the 100-110° and 150-160° ranges can be observed, but their percentage is just a little higher than for the other particle orientations, not very large (Fig. 4.16 (d)). For M65, the orientation of the particles remains locally isotropic. The SEM photographs taken on the observed plane show a structural
isotropy without a preferential orientation of the particles (Fig. 4.16 (f)). For M100, the analysis on the observed plane (Fig. 4.16 (h)) indicates that the sample is isotropic without a preferential orientation of the particles. A slightly larger percentage of particles oriented in the 50-60° and 150-160° ranges can be observed.

Figure 4.16 (a) (c) SEM photo of kaolin P300 and M35 under 1 kPa.
(b) (d) Orientation of the particles of kaolin P300 and M35 under 1 kPa.
Results under high suction, at a local level, are shown in figure 4.17 considering also the area of one photograph only. These analyses show that the fabric of these materials, whatever their mineralogy, is not quite isotropic. Figs. 4.17 (b) (d) (f) and (h) indicate that, for kaolin P300 under 158 MPa suction, there is a preferential orientation of the particles around (140-150°). The preferential orientation of M35 under 158 MPa is around (120-130°). The preferential orientation can also be observed in M65 under 1500 kPa suction in the range between 30 and 70°. For M100 under 158 MPa, the preferential orientation is between 30 and 40°. However, as
presented previously, when considering the average orientation on several photographs, the conclusion is that suction induces a random organization of the structure. Therefore, even if the structure of the specimens remains globally isotropic, locally, the particles may exhibit preferential orientation.

Figure 4.17 (a) (c) SEM photo of kaolin P300 and M35 under 158 MPa suction; (b) (d) Orientation of the particles of kaolin P300 and M35 under 158 MPa suction.
Figure 4.17 (e) (g) SEM photos of M65 mixture under 1500 kPa suction and M100 under 158 MPa suction; (f) (h) Orientation of the particles of M65 under 1500 kPa suction and M100 under 158 MPa suction.

4.4 CONCLUSIONS

The aim of the microscopic research was to establish a link between the properties of the four studied clayey materials, kaolin P300, M35, M65 and M100 mixtures and their microfabric. At the microscopic level, the approach consists in performing MIP tests in order to study the variation of the porosity with suction. The change in these parameters is related to the study of the orientation of the clay particles derived from a thorough analysis of SEM photographs.

The following conclusions may be derived from the results of the tests:
The core-shell structures is present in the M35 and M65 mixtures, as well as in M100 whereas not in kaolin P300, which reveals that the particles of montmorillonite play an important part in the shrinkage mechanism, due in particular to their lower stiffness. Their higher flexibility may result in a turbostratic fabric of the material under stress or suction.

The porosities deduced from MIP are smaller than those derived from global measurements, as is usually observed, due to the difficulty of the mercury to enter the smallest and the closed pores. Kaolin P300, M35 and M100 present a unimodal distribution of pores whereas M65 features the presence of a family of smaller pores in addition to the predominant pores. When suction increases, both families of pores are affected by a reduction of the pore entry diameter and volume.

Under the different conditions of suction (e.g. 1 kPa, 1500 kPa and 158 MPa), the analysis highlights the global isotropy of the microfabric, with a random orientation of the particles, while a finer analysis reveals that the fabric may present locally some anisotropy. For a slurry, the isotropy of fabric is obvious as long as the suction tensor is isotropic (i.e. for saturated or quasi-saturated soils) but the important conclusion is that modeling of the effect of suction in quasi-dry soils can also be carried out considering an isotropic “capillary” stress tensor in the definition of the effective stress.
Another important material used in this research is a clay rock which comes from the site of the French lab for the study of high activity nuclear wastes storage in highly consolidated clayey rock in Bure (Meuse - Haute-Marne). The objectives of this research were the following ones:

- To study the effect of suction on the formation of cracks
- To investigate the properties of the cracked material
- To try to relate the macroscopic changes to the changes in the microstructure and porosity.

This work was carried out in the framework of the ForPro national program sponsored by Andra and CNRS. Several researchers took part in this research:

- Pr. S. Taibi, from LOMC laboratory (University of Le Havre),
- Dr. H. Souli, from LTDS laboratory (ENISE),
- Dr. Thierry Reuschlé, from EOST (University of Strasbourg)
- Dr. M. Duc from IFSTTAR
- Pr. M. Hattab and myself, in LEM3 Laboratory (University of Lorraine)
- Pr. J.-M. Fleureau and myself, in LMSSMat laboratory (Ecole Centrale Paris)

As mentioned in Chapter 2, when we received the samples, they were conditioned under stress in a cell, as shown in figure 5.1.

It was observed that decompression of the samples could lead after some time to the formation of visible cracks. Therefore, it was decided to study separately the two phenomena:

- Decompression of clay rocks under constant water content;
- Changes in macroscopic and microscopic properties under different water contents and suctions.

In this chapter, my results are presented in the following three aspects:

- Effect of decompression on the microstructure of the clay rock, derived from
MIP measurements and SEM observations,  
- Effect of suction on the macroscopic properties of the clay rock, mainly its drying-wetting characteristics and tensile strength,  
- Effect of suction on the microstructure of the clay rock, with MIP and SEM;  

Whenever possible, my results will be compared with those of the literature or those obtained by the other members of the team in the ForPro Project.

Fig. 5.1 Compression cells used for the conservation of the clay rocks specimens of Bures laboratory
5.1 EFFECT OF DECOMPRESSION ON MACROSCOPIC AND MICROSCOPIC BEHAVIOR OF CLAY ROCKS

5.1.1 Introduction

The effect of decompression was clearly highlighted by the results of S. Taibi at Le Havre university (Taibi et al., 2011) using ultrasonic wave propagation (Pundit test) in the 3 directions of a rock block (Fig. 5.2). The technique permits to deduce elastic modulus in the three directions. Direction 1 corresponds to the vertical direction, i.e. perpendicular to the bedding of the material.

Figure 5.3 show that the modulus is smaller in direction 1 and decreases noticeably during a dozen of days after the opening of the sample (the removal of stress). In directions 2 and 3, the decrease is less important. This highlights the anisotropy of the material and an important effect of decompression in direction 1 which can be related to the separation of the different layers parallel to bedding.

The question we tried to answer in my study was the following one: can we observe at the microscopic scale the anisotropy of the elastic properties and the relaxation of the material which were evidenced at the macro scale?

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**Figure 5.2** Orientation of the sample for the measurement of elastic moduli (Taibi et al., 2011)

**Figure 5.3** Anisotropy of elastic moduli and large decrease in direction 1 linked to decompression in this direction (Taibi et al., 2011)
5.1.2 Effect of decompression on porosity and fabric

To study the effect of decompression on the microscopic properties of the clay rocks, we performed 2 series of analyses, using MIP measurements and SEM observations, in order to characterize the possible changes in the pore-size distribution (PSD) and in the organization of the particles. Both were carried out on freeze-dried samples.

In term of PSD variations, tests were performed at different times: \( t = 0 \), 3 months and 12 months after the opening and decompression of the core-samples. Predominant pore diameters are 0.032 \( \mu m \), 0.021 \( \mu m \) and 0.014 \( \mu m \), respectively (Fig. 5.4 (b)). At the same time, a small decrease in the pore volume is observed (Fig. 5.4 (a)). The changes in porosity are relatively small and they do not correspond to what is expected as the effect of decompression. In fact, decompression should result in an increase in the cumulated pore volume and predominant pore size, which is the contrary of what is observed. The conclusion is that this effect is probably negligible and that the observed changes may probably be due to some drying of the specimens in spite of their careful wrapping and conservation.

![Figure 5.4 Evolution of the pore space at different times in clay rocks](image)

The organization of Bure clay rock is very complex, as pointed out by many authors (e.g. Bauer-Plaîndoux et al., 1998; Wright, 2001; Sammartino et al., 2003; Su, 2005). To characterize the microstructure of the clay rock, observations were carried out with SEM. The anisotropic fabric of the material is clearly visible on the photos of figure 5.5. Before decompression, SEM photos show oriented particles in bands parallel to bedding which represent preferential planes for the development of cracks.
5.1.3 Analysis of the results

If we compare the SEM observations of the samples in the initial state before decompression with those done by M. Duc (IFSTTAR), using ESEM, on the same material 1 year after decompression, we do not see significant difference in the microstructural organization of the clay rock (Fig. 5.6). These observations confirm that the microfabric of the clay rock is constituted by horizontal layers parallel to bedding. They are consistent with those of Wright (2001) who concluded to the existence of a lamellar porosity parallel to stratification.

Another observation related to decompression was highlighted by M. Duc (IFSTTAR) in her study (Taibi et al., 2011). It is the fact that pyrite (iron disulfide) is often present in the decompression planes in the form of small crystals or framboïdal...
clusters (Fig. 5.7 (a)). Oxidation of pyrite by air leads to the formation of gypsum with an increase in volume (Fig. 5.7 (b)) and this mechanism could represent some kind of “chemical cracking”. However, the question about the oxidation of pyrite being the result of the pre-existence of cracks and of the possibility for the air to come in contact with pyrite cannot be excluded either. It is therefore difficult to know if pyrite is responsible for cracking or simply accompanies it. Indeed, these heterogeneities in veins parallel to bedding naturally form weak zones which are favorable to the development and propagation of cracks, reinforced during the crystallization of gypsum. M. Duc (IFSTTAR) also observed other heterogeneities, such as carbonate and aluminosilicate particles (mica hypothetically) in the form of large plates up to 100 μm with an orientation generally following the bedding. These inclusions can influence the changes of microstructure during drying or wetting, in one way or another: propagation of cracks, blocking particle reorganization, etc.

Figure 5.7 ESEM observations along a macroscopic pyrite vein on the surface of a crack after decompression: (a) Macro-heterogeneities (pyrite) present in the planes of mechanical decompression; (b) Pyrite-rich zone; (c) gypsum (Taibi et al., 2011)
On the other side, no change in mineralogy occurred after 1 year decompression (Fig. 5.8), according to the XRD measurements carried out by H. Souli (ENISE), which show that the chemical changes observed locally (e.g. the formation of gypsum) do not result in a change at a more global scale.

![XRD analysis of clay rocks 3 months and 1 year after opening of the core-samples](image)

Figure 5.8 XRD analysis of clay rocks 3 months and 1 year after opening of the core-samples (Taibi et al., 2011)

## 5.2 Effect of Drying and Wetting on Macroscopic and Microscopic Properties of Clay Rocks

To assess the effect of suction on the formation of cracks in clay rocks, the study was carried out both at the macroscopic and microscopic scales through:

- drying-wetting tests
- indirect traction tests,
- MIP
- SEM observations

Then, as previously, the results were confronted with those from the literature and the other participants in the ForPro project.
5.2.1 Drying/wetting tests

The DVS method was used to study the effect of suction on the changes in water content of the specimens. The method was presented in Chapter 2.

The results are plotted in figure 5.9 as the changes in water content versus suction. The dispersion of the points is very small due to the use of one sample for the whole test, instead of several for each suction as it is generally the case for the “classical” method, highlighting the reversibility of the path.

![Figure 5.9 Drying-wetting cycle on clay rock starting from its natural state, derived from DVS test](image)

5.2.2 Effect of suction on tensile strength

To characterize the effect of suction on the tensile strength of the clay rock, Brazilians (indirect traction) tests were carried out on disks of clay rock, 15 mm thick, cut in a vertical sample. The samples were equilibrated with saturated salt solutions for three months to allow reaching equilibrium. The following suctions were imposed by the salt solutions: 158 MPa for CaCl₂; 38 MPa for NaCl; 22 MPa for KCl; 6.9 MPa for Na₂SO₃. Two sets of tests, corresponding to two orthogonal directions of the samples (both in the horizontal plane) were performed for each value of suction, the results of which are shown in figure 5.10. They highlight an increase in tensile strength with suction. Compared with the initial state which corresponds approximately to a suction of 80 MPa, it is noted that drying (point at 158 MPa) results in an increase in resistance, whereas the presence of microcracks in the sample would deteriorate the material.
strength. As this is the contrary of what is observed, it is very likely that drying does not result in the formation of microcracks in the rock. Photos of the samples after the tests are presented in figure 5.11. The photo corresponding to a suction of 6.9 MPa shows that the sample is completely destroyed during the traction test. In that case, the application of stress reveals the likely pre-existence of cracks in the sample.

It can be concluded that tensile strength increases with suction up to $s = 158$ MPa, which shows that drying from 80 to 158 MPa did not damage the material and did not result in the formation of cracks. The failure pattern observed in figure 5.11 is fairly normal under high suction, but it is not so under low suction. Contact with water vapor for a long time (3 months) can lead to the formation of cracks on wetting.

![Figure 5.10 Tensile strength of clay rock versus suction](image)

![Figure 5.11 Photos of clay rocks after the indirect traction tests under different suctions](image)
5.2.3 Effect of suction on porosity

As shown in figure 5.12, the porosity changes little with suction. The pore size distribution is bimodal with a major dimension of about 0.02 – 0.04 μm and a second dimension at about 0.008 μm (Fig. 5.12 (b)). The predominant diameter of the pores in the initial state and under 158 MPa is the same, equal to 0.021 μm. That is to say, the driest samples (initial state and under 158 MPa) have the lowest pore volume and the smallest pores, with the curves virtually superimposed. The pore volume and the pore size increases slightly during wetting. This means that, independently from the formation of cracks, porosity of clay rocks changes little during drying or wetting.

Figure 5.12 Evolution of the pore size distribution under different suctions in clay rock
5.2.4 Effect of suction on fabric

Once in equilibrium with the imposed suctions, the samples were freeze-dried, then submitted to microstructure investigations with SEM observations.

Under high suction (158 MPa – the driest state) (Fig. 5.13 (a)), the photos show a dense clay matrix with bedding planes (visible in clay rocks observed in the initial state) more or less unstructured, numerous closed cracks appear, which consist of clayey particles which are arranged face to face (note: this leads to assume that the observed cracks are hydrous and were not pre-existing). And a dense clay matrix, more or less destructured also appears. When the suction decreases in the process of wetting (38 MPa and 22.4 MPa) (Fig. 5.13 (b) and (c)), the orientation is similar to that in the initial condition and the clayey particles seem to arrange less densely. Moreover, it is observed that some cracks are open. When the suction becomes lower and in the wettest condition (6.9 MPa) (Fig. 5.13 (d)), the organization of the particles in the general direction of the bedding of the rock becomes more apparent.

<table>
<thead>
<tr>
<th>Suction</th>
<th>Obs 1 (plane parallel to bedding)</th>
<th>Obs2 (plane perpendicular to bedding)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) 158 MPa (\text{CaCl}_2) (drying)</td>
<td><img src="image" alt="SEM photo" /></td>
<td><img src="image" alt="SEM photo" /></td>
</tr>
<tr>
<td>(b) 38 MPa (\text{NaCl}) (wetting)</td>
<td><img src="image" alt="SEM photo" /></td>
<td><img src="image" alt="SEM photo" /></td>
</tr>
</tbody>
</table>

Initial suction of samples \(\approx 80 - 100\) MPa

Figure 5.13 SEM photos of the microstructure of clay rock under different suctions (LEM3)
## 5.2.5 Analysis of the results

### 5.2.5.1 Drying-wetting path

The results obtained with the DVS method (Fig 5.9) can be compared with those derived from the classical tests performed by S. Taibi in LOMC at Le Havre university in the framework of the ForPro project.

Figure 5.14 shows the results obtained by S. Taibi (Taibi et al., 2011) on the natural material, plotted in the 5 corresponding planes: void ratio versus water content and suction, degree of saturation versus water content and suction and water content versus suction. These results permit to characterize the drying wetting paths in this material, with the great advantage of following simultaneously the change of all the state parameters together.

We can observe that, in the initial state, there is a suction of approximately 80 MPa in the natural specimen and its properties are represented by the circle. If the suction is decreased, the material follows a wetting path and, if the suction is increased, a drying path. The initial suction is very high, so that the main part of the test corresponds to wetting.

The tests were carried out on vertical and horizontal specimens. The wetting path from the initial state begins with some dispersion of the experimental points, which is partly due to microcracking occurred during wetting. Moreover, for very low values of suction (< 10 kPa), it is noted that in the \([\log s - e]\) plane, the vertical samples present
void ratios which stabilize around an average value of 0.4, whereas, for the horizontal samples, the void ratio continues to increase to a mean value around 0.55. The same phenomenon can be observed in the \([\log s - w]\) plane where water content is around 12\% for the vertical sample and reach 16\% for the horizontal sample. As the exchange of water is mostly vertical in these tests, this could indicate the possible formation of cracks preventing the passage of water, and therefore swelling, in the vertical samples.

This can be explained by the fact that for low suctions (a few cm), “macroscopic” cracks due to wetting should be more developed in the case of the vertical specimen than in that of the horizontal one. These cracks perpendicular to the wetting path create a discontinuity which can prematurely stop the upward flow and thus stabilize the water content and stop the swelling phenomenon. Indeed, the final value of void ratio in the vertical specimen is lower than the value that the sample would have reached if the infiltration was not blocked by the discontinuities. In all the cases, the maximum degree of saturation does not exceed 85\%. The conditions of the tests may be responsible for the formation of microcracks, as we will see in the following paragraph, as the samples are under no stress and as they are in contact with liquid water for a long enough time.

In the range of suctions common to DVS and classical tests (8 to 100 MPa), the water content appears slightly lower in the DVS test \((w = 2.5 \text{ to } 6\%)\) than in the classical tests \((4 \text{ to } 8\%)\) but this difference may be due to the use of different samples and the natural heterogeneity of the clay rock.
Figure 5.14 Drying/wetting paths of clay rock (Taibi et al., 2011)
Similar results (Figs. 5.15 and 5.16) were presented by several authors in various coordinate systems and confirm the previous conclusions, especially the quasi-reversibility of the drying-wetting paths.

![Figure 5.15](image1.png)  
Figure 5.15 Capillary pressure versus the saturation of a clay rock on drying path (Zhang et al., 2005)

![Figure 5.16](image2.png)  
Figure 5.16 Sorption isotherms on Callovo-Oxfordian clay rock (Boulin, 2008)

Concerning the microstructure investigation, ESEM observations were done by M. Duc (IFSTTAR) in the framework of the ForPro project on natural specimens (i.e. not freeze-dried, but observed under wet conditions) and they led to the same conclusions as ours concerning the effect of drying and wetting on the fabric of the clay rock. They highlighted in particular the fact that, if wetting the specimen by putting it in contact with water vapor generally does not result in the formation of cracks, the contact with liquid water for a few minutes leads very quickly to the disintegration of the material. Figure 5.17 shows surface cracking with opening of fissures which multiply and propagate in the 3 directions during wetting-drying cycles with liquid water. However, as indicated in § 5.2.2, these conclusions must be considered carefully as the samples used for indirect traction tests, equilibrated with water vapor during 3 months, seemed to have developed microcracks. Microcracks were also observed with SEM (§ 5.2.4).
Figure 5.17 (a) Surface of a crack by decompression parallel to bedding planes with cracks in the form of tortoiseshell; (b) (c) Multiplication and opening of cracks after condensation of water on the surface for a few minutes

5.3 CONCLUSIONS

In this chapter, the effects of decompression and suction on the formation of cracks in a clay rock were analyzed. More importantly, the relationship between macroscopic changes and the changes in the microstructure and porosity were investigated.

It is concluded that it is necessary to uncouple the effect of mechanical decompression (at constant water content) from that of drying-wetting (effect of
suction under constant stress) to better understand their respective effects. The effects clearly appear at different scales.

Effect of decompression is focused on the following two aspects:

(1) No effect on the structural anisotropy of the clay stone.

(2) Appearance of chemical cracks which may be due to (or accompanied by) oxidation of pyrite and formation of gypsum.

The effect of suction on clay rocks is as follows:

(1) The shrinkage limit corresponds to the initial state of the specimens, so that drying does not result in large changes in the microstructure of the rock nor in the formation of microcracks. On the contrary, wetting may damage the material, either after a long time of contact with water vapor or a short time in contact with liquid water.

(2) Suction little affects anisotropy during wetting (except at low suctions).

(3) Tensile strength increases when suction increases. Under low suctions (high degree of saturation), the tensile strength decay is accentuated by the possible formation of microcracks on wetting.

(4) MIP: The pore size distribution is slightly modified by suction changes, with a small increase in cumulated pore volume and predominant pore size when suction decreases.

(5) SEM: Microcracking occurs during wetting, rather than drying (more open and numerous cracks); Under 158 MPa, closed cracks formed by particles assembled face to face are observed. And a dense clay matrix, more or less destructured also appears. Then, the number of cracks progressively increases and these cracks become open. When the suction decreases to 6.9 MPa, the organization of particles in the directions of the general bedding of the rock becomes more apparent.

(6) Damages are limited during short water vapor drying – wetting cycles, but may increase for longer contact time.
6 CONCLUSIONS AND PERSPECTIVES

6.1 CONCLUSIONS

This thesis contributes to the characterization of cracks in six clayey materials: 5 mixtures of kaolin and montmorillonite, containing 0, 35, 50, 65 and 100% montmorillonite, and one natural clayey rock during desiccation, using both macroscopic and microscopic approaches.

The main goal is trying to understand how, in clayey materials submitted at macroscopic scale to a given drying path, cracks and microcracks appear, and how the phenomenon evolves. The approach that is proposed is based on experimental investigations and consists, at the microscopic scale, in following the microstructural evolution of the material by means of scanning electronic microscope observations and analysis and mercury intrusion porosimetry measurements on specimens prepared at different suctions. The aim of the microscopic research is to establish a link between the macroscopic properties of the clayey materials and their microfabric. At the macroscopic scale, the method is based on (1) measurements of water content, void ratio and degree of saturation versus suction \( s \) during drying, which allows to specify the relationship between shrinkage and desaturation and highlights the characteristic phases of behavior; (2) measurements of water contents and global deformations in free desiccation tests in order to study their homogeneity; (3) the determination of the local deformations and displacements during drying using the softwares VIC-2D and VIC-3D. (4) a classical study of the parameters of cracks. (5) traction tests in order to identify the tensile properties of the clays involved in the formation of cracks.

6.1.1 Macroscopic results on clays

6.1.1.1 Drying-wetting paths of the materials

Two methods (DVS and classical method) are used in order to analyze the drying/wetting paths of the materials. On drying paths, starting from a saturated slurry prepared at \( w = w_L \), all the soils remain saturated or quasi-saturated up to a large suction exceeding 1 MPa. Under such conditions, results in the \([w - e]\) plane show that the shrinkage limit void ratio \( e_{SL} \) for kaolin P300, M35, M50, M65 and M100 are 0.3, 0.5, 0.6, 0.8 and 1.1, respectively. In the \([\log(s) - e]\) plane, the compression indices (Cc) for these 5 materials are 0.241, 0.65, 1.11 and 1.32, respectively, similar to the values obtained by Hammad (2010) in oedometric tests. The shrinkage limit
suctions deduced from the \([\log(s) - w]\) plane, range from 1.3 to 5 MPa, corresponding to shrinkage limit water contents between 16 and 41%. Desaturation suction (air entry pressure) deduced from the relation between suction and degree of saturation are comprised between 1.2 and 113 MPa. The effect of the montmorillonite content on all these features is very important.

The results obtained on kaolin P300 and montmorillonite on drying-wetting paths were compared with those of Taibi (1993) and Soemitro (1993). In the case of kaolin P300, the shrinkage limit suctions and the air entry pressures are nearly the same. In the case of montmorillonite, at the beginning of the drying path, the void ratio in my tests \((e = 6)\) is a little smaller than that of Soemitro \((e = 7.3)\). Generally, the differences between my results and those of these authors are not obvious, except under high suctions where the difference may be due to the preparation of the samples.

6.1.1.2 Experiments of free desiccation

A first series of free desiccation tests on kaolin P300 and M50 was devoted to the determination of the average water contents, local water contents and deformations.

The average water contents decrease with time. Homogeneity of water content in free desiccation tests is analyzed by measuring the water content of small samples in different parts of the specimens. The same results are interpreted in another way with contour maps. It is verified that in the center of the sample, local water content is a little larger than on the boundaries. The maximum difference is 1.46%. The comparison between transversal, longitudinal and vertical deformations versus water content shows that the sample shrinks a little more in the vertical direction than in the two other directions because of the friction on the surface of the support. In the case of kaolin P300, the difference reaches 1.9% at the end of the test. Generally, one can consider that the deformations of the samples are isotropic because the differences between the values in the 3 directions are not large. Comparisons are done between free desiccation tests on the whole soil sample and the small sample used for drying tests under controlled suction conditions. In suction-controlled tests, the characteristics of the small samples are determined in various equilibrium situations, once suction, deformation, water content and degree of saturation are supposed to be homogeneous throughout the sample. The similar results in these two tests highlight the fact that the transient nature of the discussed desiccation tests is not marked.

6.1.1.3 Digital image correlation

Photos of the soil surface in the second series of free desiccation tests were taken at regular intervals and analyzed by correlation of digital images using the softwares Vic-2D and 3D. Five materials and three different supports were studied in these tests.
Two-dimensional strains and displacements maps are obtained with Vic-2D. There are five maps with different parameters: longitudinal displacement U, transversal displacement V, longitudinal strain $e_{xx}$, transversal strain $e_{yy}$ and shear strain $e_{xy}$. The zones of the sample where cracks appear are identified. And the evolution of strains and displacements before the appearance of cracks is also analyzed. Theoretically, during free drying, the displacements on the boundaries should be larger than those in the center of the sample and the displacements should be oriented towards the center. Maps of principal strains are analyzed with the purpose of identification of extensions and compressions.

In kaolin P300, the cracks evolve more quickly than in the other materials according to the displacements and strains maps. At the end of desiccation, the cracks form a kind of network. Bifurcation of cracks can be observed in some cases. When a crack is caused by traction, the propagation direction follows the direction of strains. If there are shear strains in the vicinity of the crack, then its direction will change. Two modes of cracks are detected during the tests: traction mode and tearing mode. Evolutions of displacements and strains of kaolin P300 on smooth, intermediate and rough supports on chosen sections are analyzed in order to better understand the characteristics of the observed displacements and strains and the mechanisms involved. It is observed that the displacements and strains increase with time. In the early time of desiccation, the displacements and strains on the boundaries are larger than those in the other parts of the model. With the development of cracks, in the vicinity of cracks, displacements and strains are relatively larger than those in the other parts. Longitudinal and transversal strains $e_{xx}$, and $e_{yy}$ change from extensions to compressions during desiccation. Shear strains $e_{xy}$ are relatively smaller than $e_{xx}$, and $e_{yy}$, which means that, in most parts of the model, the principal strains are mainly longitudinal and transversal. Influence of different supports on the development of cracks in kaolin P300 is analyzed. At the end of desiccation, maximum displacements and strains are similar on the different supports, whereas, at the same time, the displacements on the rough support are larger than those on the smoother supports.

For M35, M50, M65 mixtures and montmorillonite, there is an “uplift” phenomenon at the boundaries of the samples on the intermediate and rough supports. For this reason, the quantitative results for these four materials are only presented on the smooth support. For M65, the phenomenon of “uplift” is even observed on one side of the smooth support. For these four materials, just as for kaolin P300, displacements and strains increase during desiccation. With the evolutions of cracks, they are larger in the vicinity of cracks than in the other parts. Comparison of longitudinal and transversal displacements of the five materials on the smooth support
is presented. Generally the differences between the maximum and minimum displacements (U and V) do not change much.

Analysis of the cracks with the software VIC-3D was also performed on kaolin P300 on the smooth support. In addition to longitudinal and transversal displacements, longitudinal, transversal and shear strains, there is another important output – vertical displacements. The evolution of vertical displacements, in addition to the displacements in the plane, is analyzed. Near the boundaries of the cracks, vertical displacements can be observed.

6.1.1.4 Analysis of cracks

With the software Image-J, the total length (l), average width (d) and surface crack ratio (SCR) (the ratio between the surface of cracks and the total surface of the sample) were analyzed. For a given material and a given support, the evolution of the length of cracks is the same as that of surface crack ratio. For kaolin P300, the total length on the intermediate support is larger than that on the two other supports, and reaches about 250 mm at the end of desiccation. For the smooth support, the maximum width is about 0.055 mm. For the intermediate and rough supports, the maximum width is 0.65 mm at the end of desiccation. It is concluded that the roughness of the support influences the crack appearance and their evolution during desiccation.

Comparison between the cracks in the different materials on the smooth support is made. The parameters of the cracks in the tests with the three other mixtures M35, M50 and M65 on the smooth support were also analyzed. The total length of the cracks in kaolin P300, M35 and M50 at the end of desiccation is nearly the same. The value is about 120 mm. For M35, M50 and M65, the process of desiccation is quicker than for montomillonite. The montmorillonite fraction in the mixture plays an important part in the kinetic of the crack development before stabilization. This can be explained by the increase in the plastic properties and the decrease in permeability with the montmorillonite percentage, the crack development becoming thus slower and slower during the free desiccation process.

6.1.1.5 Traction tests

A traction device was developed in order to measure the tensile strength of the different studied materials. The maximum displacement in the traction tests is 20 mm. Traction tests were carried out on three materials: kaolin P300, M35 and montmorillonite. For a given material, tensile strength increases when water content decreases. The tensile stress value corresponds to the maximum value of the traction force. In the case of kaolin P300, with the water contents of 39%, 31% and 26%, the maximum tensile stresses are 9 kPa, 11 kPa and 14 kPa, respectively. For M35, the maximum tensile stresses are 10.3 kPa, 9.3 kPa and 8.7 kPa, respectively, when the
water content is 53%, 56% and 59%. Cracks develop very quickly in M35. In the case of M100, with the smallest water content of 81%, the maximum tensile stress is 12 kPa while, for the maximum water content of 176%, the maximum tensile stress is just 3.2 kPa. When the water content is less than 100%, the process of rupture of the samples is very progressive and the tensile stress increases during a very long time. With the increase in the percentage of montmorillonite, the maximum tensile stress decreases at the same liquid index.

Water content is related to both maximum tensile stress and suction. The effect of suction on tensile strength seems qualitatively similar to what is observed in the case of unconfined compression strength.

### 6.1.2 Microscopic results on clays

#### 6.1.2.1 Core-shell structure of the specimens after drying

After the specimens have been freeze-dried, a special phenomenon is observed. The specimens are found to have core-shell structures. The core-shell structures are present in the M35 and M65 mixtures, as well as in M100 whereas not in kaolin P300, which reveals that the particles of montmorillonite probably play an important part in this phenomenon, due in particular to their lower stiffness. Their higher flexibility may result in a turbostratic fabric of the material under stress or suction, whereas in kaolinite, piled-up particles are linked to each other by strong hydrogen and van der Waals bonds and the resulting crystallites are stiff plates.

#### 6.1.2.2 Results of mercury intrusion porosimetry tests

The mercury intrusion porosimetry tests are carried out on kaolin P300, M35, M65 and montmorillonite specimens. For these four materials, the volume of mercury gradually decreases according to the increase in suction, which results in a decrease in porosity. The porosities deduced from MIP are smaller than those derived from global measurements, as is usually observed, due to the difficulty of the mercury to enter the smallest and the closed pores. Kaolin P300, M35 and M100 present a unimodal distribution of pores whereas M65 features the presence of a family of smaller pores in addition to the predominant pores. When suction increases, both families of pores are affected by a reduction of the pore entry diameter and volume. In the case of M35 and M100, there is a large increase in the large diameter domains for the cumulative pore volume curve under 1500 kPa. There is a possibility of the presence of cracks in the sample which is verified in the SEM observations.
6.1.2.3 Evolution of the orientation of the clay particles under different suctions

Specimens of kaolin P300, M35, M65 and M100 slurries submitted to 1 kPa, 1500 kPa and 158 MPa suctions are observed with SEM. For these four materials, the void ratio decreases when suction increases, and increases with the montmorillonite content in the mixture. Under the highest suction (158 MPa), cracks appear in the materials. For kaolin P300 and M35, the cracks are open whereas, in M65, they are closed. This phenomenon is related to the difference in the flexibility of the particles. Under the different conditions of suction, the analysis highlights the global isotropy of the microfabric, with a random orientation of the particles, while a finer analysis reveals that the fabric may present locally some anisotropy. For a slurry, the isotropy of fabric is obvious as long as the suction tensor is isotropic (i.e. for saturated or quasi-saturated soils) but the important conclusion is that modeling of the effect of suction in quasi-dry soils can also be carried out considering an isotropic “capillary” stress tensor in the definition of the effective stress.

6.1.3 CLAYSTONES

The effects of decompression and suction on the formation of cracks in a clay rock were analyzed. More importantly, the relationship between macroscopic changes and the changes in the microstructure and porosity were investigated.

It is concluded that it is necessary to uncouple the effect of mechanical decompression (at constant water content) from that of drying-wetting (effect of suction under constant stress) to better understand their respective effects. The effects clearly appear at different scales.

Effect of decompression is focused on the following two aspects:

(1) No effect on the structural anisotropy of the clay stone.

(2) Appearance of chemical cracks which may be due to (or accompanied by) oxidation of pyrite and formation of gypsum.

The effect of suction on clay rocks is as follows:

(1) The shrinkage limit corresponds to the initial state of the specimens, so that drying does not result in large changes in the microstructure of the rock nor in the formation of microcracks. On the contrary, wetting may damage the material, either after a long time of contact with water vapor or a short time in contact with liquid water.

(2) Suction little affects anisotropy during wetting (except at low suctions).

(3) Tensile strength increases when suction increases. Under low suctions (high
degree of saturation), the tensile strength decay is accentuated by the possible formation of microcracks on wetting.

(4) MIP: The pore size distribution is slightly modified by suction changes, with a small increase in cumulated pore volume and predominant pore size when suction decreases.

(5) SEM: Microcracking occurs during wetting, rather than drying (more open and numerous cracks); Under 158 MPa, closed cracks formed by particles assembled face to face are observed. And a dense clay matrix, more or less destructured also appears. Then, the number of cracks progressively increases and these cracks become open. When the suction decreases to 6.9 MPa, the organization of particles in the directions of the general bedding of the rock becomes more apparent.

(6) Damages are limited during short water vapor drying – wetting cycles, but may increase for longer contact time.

6.2 PERSPECTIVES

There is a lot of room for improving present knowledge. We hope that we have brought a useful and improved method to the analysis of microcracks in clays related to desiccation. There are many aspects for future research.

(1) Tests with larger models can be considered in the future. The model in this research is with a length of 300 mm, a width of 200 mm and a height of 4 mm which is relatively small. Larger models will provide different characteristics of cracks which will be very interesting to analyze.

(2) Measurement of the properties of cracked materials can be carried out in the future. At the end of free desiccation tests, properties of the specimens can be investigated, for example, void ratios, observations with SEM, pore size distributions with MIP, etc. The results can be compared with those in slurry specimens used in the other tests.

(3) Digital image correlation in 3D is very promising but should be taken more carefully and the device should be fully calibrated before the tests. Experiments with highly swelling soils (M65, M100…) can be carried out in order to analyze large vertical displacements during drying on intermediate and rough supports. However, the interpretation of the results using this technique is very delicate and very time-consuming.

(4) Modelling of desiccation cracking in soils can be performed in the future. The modeling results of crack propagation, crack pattern etc. will be compared with those during the experiments.
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