



Article

Experimental Investigation of the Shrinkage-Limit and its Impact on the Physico-Mechanical Properties of Dried Natural Clay

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Abstract

This study offers an experimental analysis of the thermo-hydro-mechanical coupling of convective-dried natural clay. The significant structural changes that the material undergoes as an unsaturated, shrinking porous medium define its final quality. The main objective of this paper is to explain the coupling of mass, energy, and momentum transfers, by the so-called shrinkage-limit of the porous medium, which is a value to be determined experimentally based on the physical and mechanical properties of the material. Characterizing how important some characteristics, such as apparent density, volumetric shrinkage, and porosity, can vary with the moisture content. Furthermore, the Young's modulus evolution during the drying stages was used to quantify the mechanical stiffness. The exact identification of the shrinkage-limit is essential to this study, which acts as a crucial transition threshold. The findings demonstrate how the material transitions from a saturated shrinking state to an unsaturated rigid phase by comparing Atterberg limits with experimental drying data. The results highlighted a strong correlation between mechanical strength and the hygroscopic state, indicating that the shrinkage-limit, found at approximately 15% water content, is essential for forecasting structural stability and averting crack formation. The established correlations provide essential data for the development of predictive models for porous media undergoing high-stress convective evaporation.

Keywords

Shrinkage-limit, Clay drying, Density and porosity

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1. Introduction

The study of drying processes in porous media is a fundamental challenge in many scientific and industrial domains, including civil engineering, soil science, ceramics, and food technology. Drying is not only a process of moisture removal; it is a complex multi-physical phenomenon involving simultaneous heat and mass transfer, which induces significant structural modifications. Among the materials most sensitive to these variations, natural clay stands out due to its highly deformable nature and its complex response to hydric stress. Clay materials are characterized by a high affinity for water, primarily due to their large specific surface area and mineralogical composition. During convective drying, the water evaporation from the pores generates capillary pressures that exert compressive forces on the solid matrix. In natural clay, these forces lead to a significant reduction in volume, a phenomenon known as shrinkage. Unlike rigid porous media, the coupling between the hydraulic state and the mechanical structure in clay is non-linear and interdependent. This thermo-hydro-mechanical coupling is responsible for the evolution of the material's physical properties, often leading to undesirable effects such as warping, cracking, or loss of structural integrity in final products.

In literature, the convective drying control has involved the study and setting equations of the hydro-thermo-mechanical coupling during this process. It reflects, essentially, the interaction between the mechanical aspects and the drying process in these materials [1-7]. Generally, the problem in modelling drying process is to have good and accurate data of the dried product. Attention has been paid to the experimental studies and analyses for each internal/external aspect connected with this process: product shape [8], skin cracks and breaking [9,10], Atterberg limits [11], Young modulus and its variation versus time and/or water limit [5,8,12,13], shrinkage phenomenon [12-14], drying kinetics [12,15] and many other phenomena relative to the drying process and depending on different materials physico-chemical properties. Each described drying variable has been, in the majority of study cases, treated separately as it was detailed in the references cited above.

We choose in this work to be concentrated on the volume shrinkage phenomenon appearing during drying of porous media. It reflects the reaction of the completely dried body under the conditions of mass transfer essentially: when moisture flows out of the product [16]. It is known as deformations in different directions causing, in many critical cases, the cracks in the product. In the literature, many papers deal with the experimental study of cracking during drying by determining the kinetics of cracking [5], studying the dependence of the deformation phenomena on space and time [12], and illustrating the influence of the evolution of the material density on the shrinkage [12].

As we treat industrial products cases which are made of porous media is manufacturing (clays, ceramics, bricks...), the final state of the dried product is important for industrialization (minimum waste, better quality, final shape). Achieving this objective, the energy consumption ratio must also be taken into account. To do it, the identification of a so-called shrinkage limit (subject of this work) may allow managing the drying process. In fact and relatively to this, the latter takes place in two phases:

- (1) First stage: the product is subjected to hot, humid air to prevent it from shrinking abruptly on the surface and to promote the evaporation of the water.
- (2) Second stage: the dry air is always warm but dry as the product no longer takes off.

As we deal with high thermo-hydro-mechanical coupled issue, we try in this work to focus on the effect of the shrinkage on the physico-mechanical behaviour of natural clay. The purpose was to demonstrate experimentally the coupling between different phenomena occurring during drying of porous medium specifically clay materials. Concentrating on this aim and as a result, a same approximate value of water content was found and discussed as a parameter to describe the influence of the shrinkage on: physical identification of the test material (determination of Atterberg limits), deformations characterized during drying (description of volumetric shrinkage), rheological behaviour (setting of stress-strain relations) and mechanical strength (behaviour during compression tests).

The shrinkage behaviour of clay is generally divided into several stages (Figure. 1). In the beginning of drying, the volume loss is usually proportional to the amount of evaporated water. However, as drying progresses, the material can reach a critical threshold known as the shrinkage-limit. Beyond this point, the air starts to enter the pores and the material transitions from a saturated, plastic state to an unsaturated, rigid state [17,18]. Understanding this transition is crucial for industrial applications, yet the precise impact of this limit on the evolution of fundamental properties like density, porosity, and especially stiffness remains a subject of ongoing research.

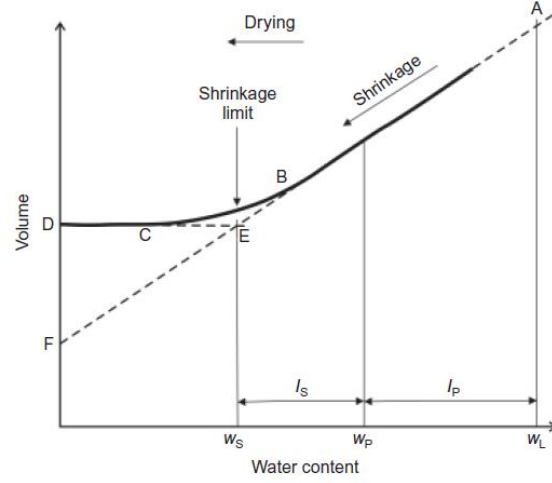


Figure 1. Schematic plot of water content against volume showing Atterberg limits: (W_s : shrinkage limit; W_p : plastic limit; and W_L : liquid limit).

Despite the extensive literature on clay material drying, there is often a gap in the integrated characterization of these properties within a single experimental framework. Most studies focus on either the drying kinetics or the mechanical strength independently. However, for precise modelling and industrial optimization, it is essential to establish a direct correlation between the macroscopic shrinkage behaviour and the evolution of mechanical parameters such as the Young's modulus. In addition, the relationship between the Atterberg limits and the dynamic behaviour of the material during convective drying needs further clarification to provide more results explanation. This study's main goal is to present a comprehensive experimental illustration of the thermo-hydro-mechanical coupling in natural clay during the convective drying process. In contrast to conventional studies that frequently separate physical and mechanical parameters; this work establishes a direct, systematic correlation between the shrinkage limit and the simultaneous evolution of density, porosity, and Young's modulus. This paper's unique contribution is the demonstration of the material's intrinsic shrinkage limit, which connects dynamic structural stiffness and traditional geotechnical indicators (Atterberg limits). This all-encompassing method provides an accurate physical signature of the material's transition, which is crucial information for creating predictive models and streamlining industrial drying procedures.

This paper aims to bridge this gap by providing an exhaustive experimental investigation of the shrinkage effect on the physico-mechanical properties of a natural clay material during a convective drying process. The primary objective was to demonstrate the coupling between the hygroscopic, thermal, and mechanical aspects. We focused on:

- (1) Determining the shrinkage limit and its physical significance.
- (2) Analysing the evolution of apparent density and porosity during the drying.
- (3) Quantifying the change in mechanical stiffness through the Young's modulus.
- (4) Correlating these transitions with the Atterberg limits to establish a comprehensive material signature.

2. Materials and Methods

The model material used in this experimental study was natural clay extracted from Tabarka region in the north-west of Tunisia. It was found formed mainly of kaolin (for more details see the work of Chemkhi et al. [19]). The experimental study used a fine-grained, unpurified clay powder that was mixed with distilled water to form a slurry with an initial water content of 36%. The performed experiments were the determination of the evolution of density, the volume shrinkage and the porosity during the convective drying as a function of water content. This study provided essential data for the demonstration of the relation between the shrinkage limit of the material and the other properties evolution.

2.1 Behaviour during Drying: Volume Shrinkage

As the volume shrinkage is a macroscopic characteristic function dependent on the quantity of water existing in the material for low and medium temperatures, the experiments were performed at a temperature equal to 40° C to an initial water content close to 36% (dry basis). The water content is defined, based on the mass of the dry sample m_s , by:

$$W = \frac{m - m_s}{m_s} \quad (1)$$

The procedure was summarized in sample volume monitoring during drying, using a calliper. After taking the necessary data for tracing curves, shrinkage was calculated from the ratio of volume compared to the initial volume of the sample (V/V_0).

In general, the shrinkage follows a decreasing trend until cancelling out. The water content corresponding to zero shrinkage, and from this value, the product does not change volume. In order to generalize the results and conclude on the relevance of the deductions made following the volume monitoring experiments during drying, we carried out the tests on samples of different shapes (parallelepiped and cylindrical).

The reproducibility criterion of volumetric shrinkage was checked, to avoid the random effects of the appropriate measurement method for the tests in question, by resuming the experiment for two samples (same preparation) with the same temperature condition and the same measuring instruments.

2.2 Physical Characterization

The Atterberg limits represent fundamental physical constants, expressed as gravimetric water content, which define the critical transitions in the consistency of fine-grained soils. These empirical thresholds predict the rheological behaviour of the clay during desiccation, specifically marking:

Liquid Limit (W_L): The boundary at which the soil transitions from a viscous liquid state to a plastic state.

Plastic Limit (W_P): The point of transition from a plastic state to a semi-solid state, marked by the beginning of crumbly behaviour and a significant reduction in deformability.

In the context of this study, these limits are essential for identifying the range of moisture content over which the material remains susceptible to significant volumetric deformation. The experimental determination of these parameters was conducted in accordance with the standard protocols (NF EN ISO 17892-12) [20]. The laboratory tests were performed using specialized equipment at the Civil Engineering Laboratory of the National Engineering School of Tunis.

2.2.1 Determination of Liquid Limit (W_L)

We used the Casagrande device (Figure 2), where the rotation of a cam causes a series of shocks. It implies the approximation of the paste mass, prepared by shuffling clay powder and adding water. A mass of the paste was removed to calculate the corresponding water content. It will be noted as the liquid limit (W_L).

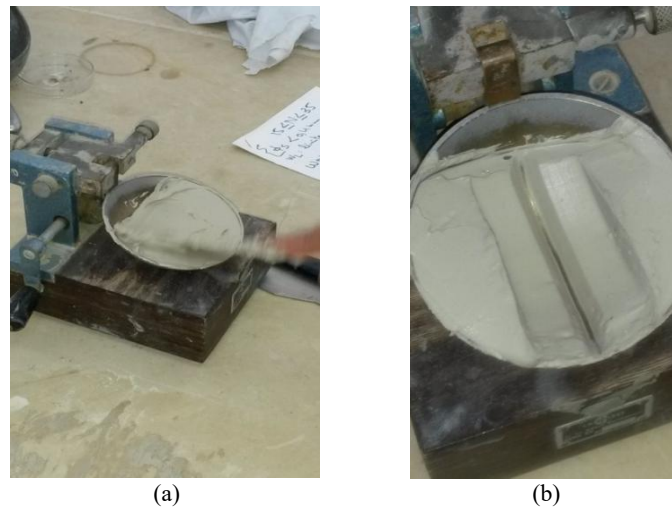


Figure 2. Test on the Casagrande apparatus. (a) Filling the cup, (b) Sharing dough in half.

2.2.2 Determination of Plasticity's Limit (W_P)

It was proceeded similarly as above to prepare homogeneous paste. Beads (rollers) were formed of length between 10 and 15 cm and close to 3 cm diameter. As soon as cracks or breaks were occurring, the limit of plasticity was calculated by weighing the roller, on a scale in order to get the wet solid content and introducing it into an oven for the water content of dry solid after 24 hours.

2.2.3 Rheological Behaviour: Interpretation of Atterberg Limits

Basing on Atterberg limits, we can discuss the rheological state of a porous medium subjected to a change of moisture content. Figure 3 illustrated the description of each state respecting intervals of water content [21]. We noted that W_S (named solid limit according to Atterberg limits) could designate the shrinkage limit whose value will be estimated in the following parts. It is also noted that the range of small deformations (elastic type) described in Figure 3 is very small compared to the other intervals.

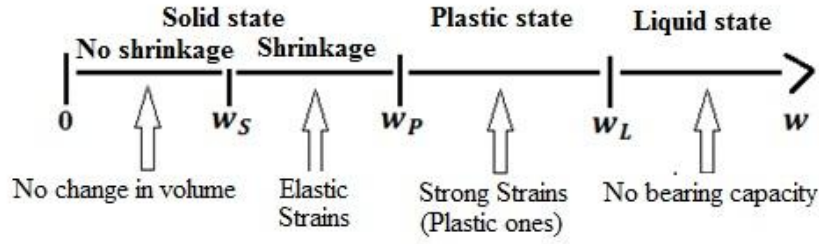


Figure 3. States of porous medium depending on Atterberg limits.

2.3 Apparent Density

The apparent density is defined basing on the mass and the volume of the product, expressed as:

$$\rho = \frac{m}{V} \quad (2)$$

Experimentally, the measurements were based on the Archimedes principle: the mass of the sample is determined in the air and then when it is emerged in a liquid [19] and the density is calculated using the equation (3) given by the constructor of the experimental device.

$$\rho = \frac{m(a) \times (\rho(f) - 0.0012)}{0.99983 \times G} + 0.0012 \quad (3)$$

Where $\rho(f)$ is the density of the fluid; $G = m(a) - m(f)$ is the difference between the two masses: $m(a)$ and $m(f)$ are the masses of the solid taken in the air and in the fluid, respectively.

Samples were prepared from a mixture of clay powder and water and formed into small spheres (5mm of radius). They were put in an oven at 40 °C and taken out at various intervals of time (5 to 10 minutes) to obtain several values of water contents ranging from 0 to 36% (on dry basis). Before proceeding, the sample was covered by a layer of para-film to prevent mass loss in contact with water. To obtain the mass of the dry product and after so the water content, the sample was placed in an oven at 105 °C during 24 hours.

2.4 Gaseous Porosity

To calculate the gas porosity, we used the presented formulation, through Eq. 4. The following properties of the medium were used: initial moisture content, initial apparent density and solid intrinsic density.

$$\phi_g = \frac{Z - (1 - \phi_0)Y}{Z} \quad (4)$$

Note that Z is the experimental shrinkage, Y is the ideal shrinkage, ϕ_0 is the initial gas porosity of the medium [22].

This initial gaseous porosity was calculated using the initial properties of the clay material using the following equation:

$$\phi_0 = 1 - \frac{\rho_0}{\rho_s} \cdot \frac{(1 + W_0 \cdot \rho_s / \rho_l)}{1 + W_0} \quad (5)$$

2.5 Mechanical Behaviour

This section was dedicated to study the profiles evolution of Young Modulus versus water content. To do it, we determined the mechanical behaviour based on compression tests results. Samples were tested using a traction-compression machine (LRX Plus) with console (Ref. No. 01/2962) shown in Figure 4. It served to determine the modulus of elasticity (Young's modulus) of the clay.

Unlike what is common to the relations linearity when determining the Young's modulus, the evolution of stress-strain depends on what is small or large deformations hypothesis [22,23]. In this work, we used what it is called a "secant" modulus (E_{sec}) defined by the slope of the line connecting the origin to the current module. The level of strains (line slope of 0 to 1% strains), considered to ensure a result on the elastic domain, was chosen by referring to the work of [24] (testing on plates of clay of the same composition as our studied clay). We tried to compare the results of the Young Modulus evolution profile with those of volume shrinkage and Atterberg limits by looking for identifying the shrinkage limit and its effect on the evolution of the rigidity of such medium.

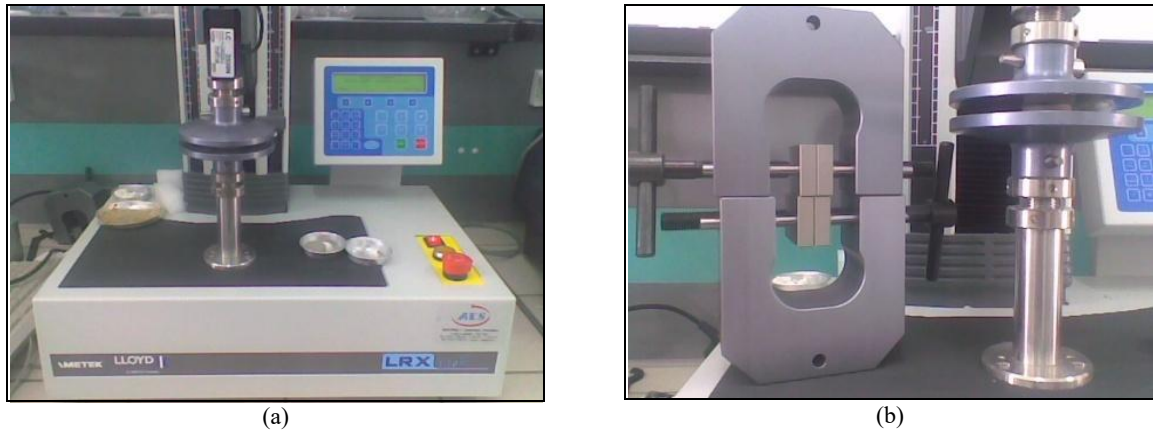


Figure 4. Machine LRX Plus for compression tests. (a) Monitoring console for compression, (b) Tests accessories.

This section details the experimental protocol established to quantify the evolution of the material's stiffness, expressed through the Young's Modulus as a function of its decreasing water content during the drying process. In geotechnical and materials science, the mechanical response of clay is intrinsically linked to its hydric state, necessitating a precise tracking of its elastic properties.

2.5.1 Sample Preparation and Experimental Setup

The experimental study used a fine-grained, unpurified clay powder. The powder was saturated with distilled water to form a slurry with a water content of 36%. This mixture undergoes intensive mechanical stirring to achieve a high degree of homogenization, eliminating air pockets and ensuring a uniform distribution of clay particles. The resulting paste was molded into calibrated cylindrical specimens with a diameter of 30 mm and a height of 10 mm. Mechanical testing was performed using the traction-compression machine equipped with a high-precision console. To simulate quasi-static conditions and minimize the impact of viscous effects during the test, a constant displacement rate of 0.1mm/min was applied.

2.5.2 Determination of the Secant Modulus

The determination of the Young's modulus in clayey materials is complex due to the non-linearity of the stress-strain relationship, which is highly sensitive to the magnitude of deformation. According to the small versus large deformation hypotheses [23], a traditional linear approach is often insufficient. We adopted the secant-modulus approach. This modulus is defined as the slope of the line connecting the origin of the stress-strain curve to a specific point. To ensure that the measurements remained within the elastic domain, the slope was calculated over a strain range of 0 to 1%. This specific interval was selected based on the findings [24], who demonstrated that for clay plates of identical mineralogical composition, this range captured the fundamental elastic signature before the onset of plastic-deformation or micro-cracking.

2.5.3 Correlation with Structural Transitions

This experimental approach's integration of mechanical data with the clay's structural evolution is a significant innovation. We seek to correlate the abrupt increase in material stiffness (rigidity) with the transition seen in the volume shrinkage and Atterberg limits by charting the profiles of Young modulus against water content. This analysis specifically aimed to show that the shrinkage limit acted as a mechanical "Inflection point," signifying the change from a plastic, deformable medium to a rigid, semi-solid structure.

3. Results and Discussions

3.1 Clay Material Characterization and Identification

The collected samples of the natural clay were ground using a mechanical grinder at the laboratory scale. The collected fine powder has a particle size distribution varying between 5 and 35 μm , which corresponds to industrial powders (standard ceramics).

X-ray diffractometer allowed identify the main clay mineral components [19,25]. In fact, the results demonstrated that the main component were quartz and kaolinite. Some illite clay was also detected. The elementary characterization was performed by atomic absorption spectrophotometer (type Perkin Elmer 560), major elements contents were determined. The loss in ignition (L.I.) at 1000 $^{\circ}\text{C}$ was determined also and it is about 14.44% of the sample initial mass. The results were detailed in the Table 1.

Table 1. Chemical composition of the natural clay in mass percentage.

Oxides	SiO ₂	K ₂ O	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	CaO	MgO	MnO	L.I
Mass (%)	39.4	15.61	13.85	14.10	0.68	1.14	0.56	0.13	14.44

3.2 Physical Characterization

3.2.1 Determination of Liquid Limit (W_L)

Experimentally, the liquid limit W_L corresponds to a closure of the groove after 25 shots. After removing the water content values for a range of impacts already determined, a straight average of experimental couples ($\ln N$, W_L) was drawn in Figure 5. The limit of liquidity was determined by projecting the abscissa $\ln(N)$ for $N = 25$ strokes on the curve. It was found equal to: $W_L = 46.58 \pm 1\%$.

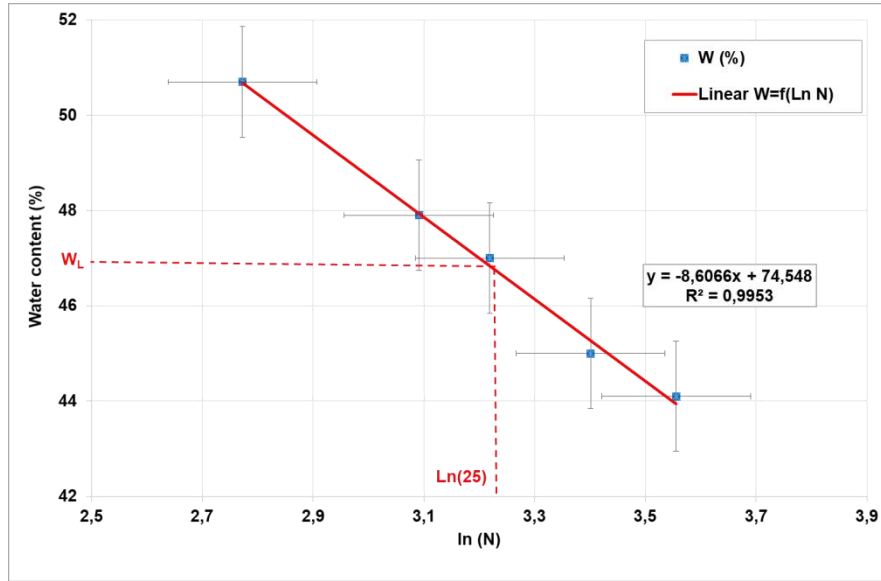


Figure 5. Determination of liquid limit (W_L).

3.2.2 Determination of Plasticity's Limit (W_P)

The limit of plasticity has been given by the average of at least three results values, to find it for the clay samples equal to: $W_P = 16.27\%$.

3.2.3 Rheological Behaviour: Interpretation of Atterberg Limits

In the purpose of modelling the rheological behaviour of a porous medium, we deduced from the states derived from Atterberg limits a proposal for behaviour laws (Figure 6) by intervals of water contents. Note that W_s has an approximate value in Figure 6 below.

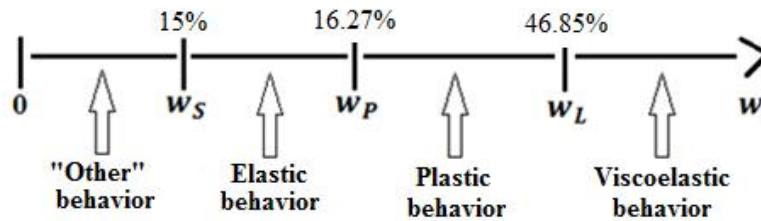


Figure 6. Proposition of rheological behaviour modelling according to the limits of Atterberg.

We can remark that the shrinkage limit can indicate a change in the rheological behaviour, which will be reflected in the writing of theoretical models relating to the study of porous media during drying. The choice of the laws stated in Figure 6 was reasoned as follows:

(1) The liquid state generally uses the viscosity but since we treated a material formed of a solid matrix, we introduce a law of viscoelasticity. The elasticity was chosen for its character of reversibility that could be suitable with the presence of a large quantity of water.

(2) The contents, on the one hand, between the limit of liquidity and that of plasticity, and on the other hand, the plasticity limit and the solid limit correspond to respective laws to the states described in Figure 3.

(3) The assignment of the character of elasticity described according to the limits of Atterberg for contents lower than that of plasticity could bring to mind several questions. Indeed, this hypothesis make that the deformations are reversible, a fact that is not obvious experimentally. However, in order to remove this ambiguity, we assumed that this is a domain of small perturbations (or transformations), a hypothesis validated in this case by the elasticity law.

(4) The behaviour in the last domain of water content (less than W_s) remains an element to be discussed since it may have limits of use in specific manufacturing cases (less than 10% dry basis, which generally is not directly and exclusively affected in the drying phase of the entire manufacturing process). The material at this stage loosed its ductility and becomes fragile considering the complete elimination of the plasticity character [26].

We can mention in this stage that the values of these physical limits could be probably changed if we have used another tool to determine them. Kayabali et al. [11] reported from that the Casagrande cup method and the fall-cone method do not give the same values of the liquid limit: we can talk about a margin between +10% and -200% [27]. According to Kayabali et al. [11], a newly devised mud press machine is more advantageous because it eliminates a number of uncertainties involved in conventional methods. This machine could be used to determine the liquid limit and the plastic limit in the same operation. Concerning the conventional methods described in our work, they proved experimentally that it could be retained because of their efficiency and accuracy.

The temperature is also a parameter influencing theses physical limits evolution. In fact, Abu Zreig et al. [28] demonstrated that the physical and mechanical properties are strongly influenced by the temperature when it ranged from 100 °C to 300 °C: for example, the liquid and plastic limits steadily decrease with increasing temperatures. Heating the clay at 400 °C gives rise to material with plastic limit equal to zero [28]. To conclude, the results announced in our work could be experimentally proved and used for drying processes at low temperatures less than 100 °C.

3.3 Apparent Density

Figure 7 represented the variation of the apparent density as a function of the product water content. It is clear that a maximum density value (almost equal to 2030 kg/m³) is recorded for a moisture content equal to 15% (dry basis). This profile's change can be explained as follows: for a relatively high value of water content, the migration of the water is macroscopically interpreted by the shrinkage phenomenon. In this case and by referring to the equation (2) above, a volume decrease causes increase in the apparent density of the sample. However, when it exceeded the shrinkage limit (equal to 15% for our studied clay), it was the mass loss that carried for a case of zero volume shrinkage. Consequently, the density decreased following the decrease of the product mass.

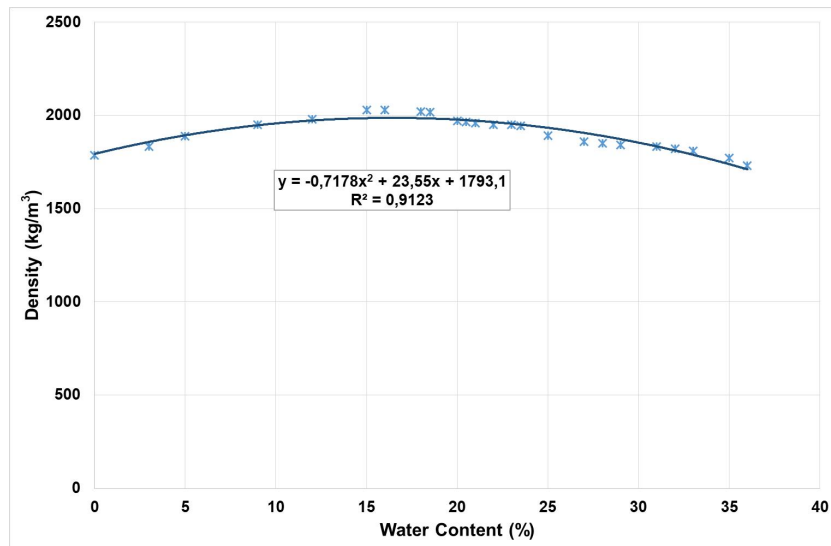


Figure 7. Experimental evolution and the fitting curve of the clay density versus water content.

The fitting of the experimental results drawn in Figure 7, give the following relation between the density and the water content:

$$\rho(W) = \rho_b - b.W^2 + c.W \quad (6)$$

With: the bulk density $\rho_b=1793.1$ kg/m³. $b=0.7178$ and $c=23.55$

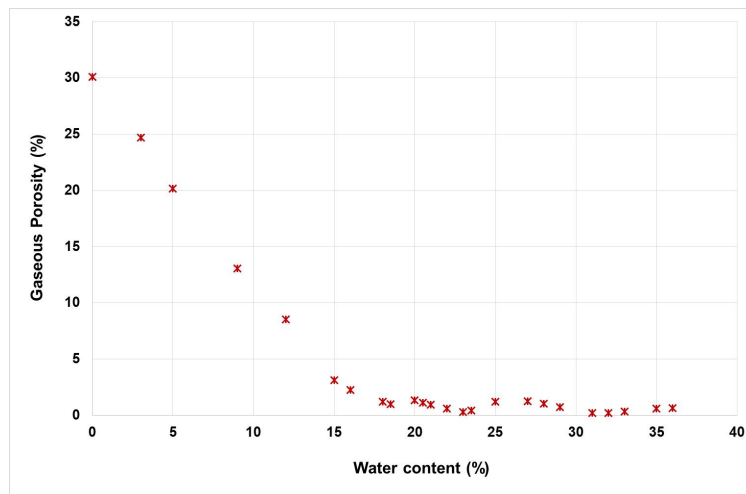
The initial properties of the clay material were presented in the Table 2 below.

Table 2. Properties and initial values for the natural clay.

Properties	Symbol	Value and unit
Initial water content	W_0	36 %
Initial density	ρ_0	1720 kg/m ³
Bulk density	ρ_b	1793.1 kg/m ³
Intrinsic solid density	ρ_s	2350 kg/m ³
Initial gaseous porosity	ϕ_0	0.65 %

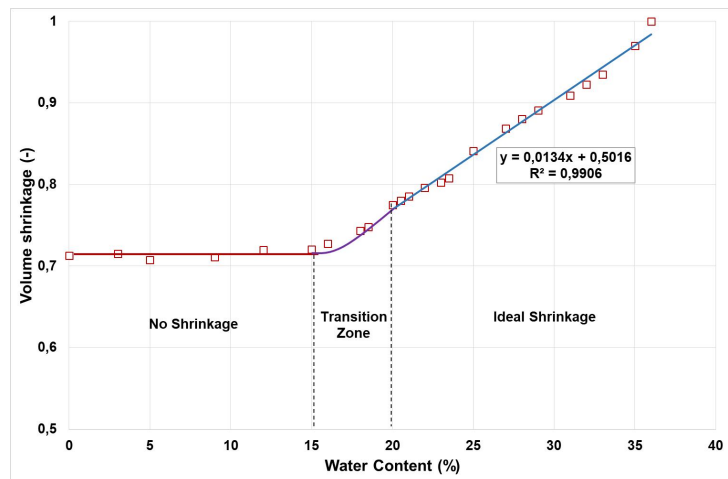
3.4 Porosity

During drying, the water content decreases, highlighting a hole in the material's structure filled with increasing volumetric shrinkage to record water content using the presence of a third phase other than solid and liquid. The gas begins to pass through the pores of the material. The Figure 8 confirmed that the gaseous phase is negligible until a water content close to ($W=17\%$) then it increased linearly to 30% at the end of drying. This value corresponds to the gaseous porosity of fully dry solid. Figure 8 confirmed the hypothesis of a saturated medium (only two phases: liquid and solid) until the end of the first drying period to pass to an unsaturated medium.

**Figure 8.** Experimental evolution of the gaseous porosity versus water content.

3.5 Volume Shrinkage

Figure 9 represented the change in volume (relative to the initial volume) for a cylindrical natural clay sample according to the dry base moisture content. In response to drying, clay water volume decreases and the soil shrinks. It has shown that the final volume measured at the end of drying shows 71% of the initial volume of the studied natural clay. This means that the loss in volume presents about 29% of the initial volume. These values can be discussed in accordance with the behaviour of clay in general, contrary to the case of gels for example where the initial water content (which will be released in large quantities at the end of drying) can reach ninety percent on dry basis.

**Figure 9.** Volume shrinkage: Experimental measurements and fitting curves.

The shape of Figure 9 showed the existence of three characteristic zones representing the shrinkage variation [17,18,25], in the drying direction:

- (1) A first zone of development where the shrinkage is almost ideal. It is essentially caused by the mass loss. The material in this phase is saturated to a marked limit w_{c1} . For the studied clay in this work, that value is $w_{c1}=20\%$ (*dry basis*);
- (2) An intermediate transition zone (between w_{c1} and w_{c2}) where the loss in mass and volume down are no longer proportional: It is the presence of unsaturated media. For this study, the second limit value is equal to $w_{c2}=15\%$ (*dry basis*) and this is in fact the interested value resulted from this work: the shrinkage limit;
- (3) A third zone where the shrinkage is zero. A mass loss is observed but the sample volume remains unchanged.

To study the effect of changing the shape, Figure 10 is represented to plot the evolution of the volume for a parallelepiped sample. It shows the same results obtained for a cylindrical sample. Then, we can conclude that the shrinkage limit is a value property of the material, which does not depend on the sample shape.

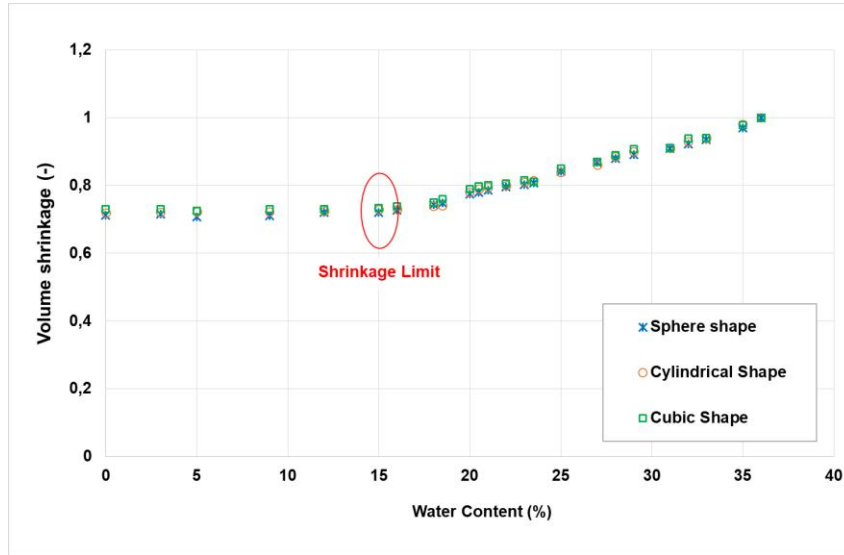


Figure 10. Reproducibility of the tests with three sample shapes: Profiles of volume shrinkage versus moisture content.

To generalize the results stated above, concerning the existence and uniqueness of the shrinkage limit value, and to conclude on the reproducibility of the shrinkage phenomenon, we repeated the experiment for three samples of different shapes (same preparation) at constant temperature conditions ($T=40\text{ }^{\circ}\text{C}$) and the same measuring instruments. The first sample has a spherical shape (1 cm radius), the second has a cylindrical shape (1cm radius and 1cm height) and the last one corresponded to a cubic shape (1cm side length). According to Figure 6, these independent experiments showed that the results are remarkably reproducible and clearly consistent with the results of characterization studies of porous media [22,25].

Finally, we can conclude that the shrinkage limit is an intrinsic property of the material. It can be determined experimentally, independently of external factors to the dried material. We underline once again the importance of this value in managing the drying process (Flow and duration of exposure in the drying chamber).

3.6 Mechanical Behaviour

We will discuss in this part the influence of the shrinkage limit on the rigidity evolution for natural clay undergoing a change in water content. It is characterized by the longitudinal elastic constant (Young modulus) which has been determined from the experimental data.

The fitting of the experimental results drawn in Figure 11, give the following relation between Young modulus and the water content:

$$E(W) = a - b \cdot \exp(-c \cdot W^{-d}) \quad [\text{MPa}] \quad (7)$$

With: $a=15.187$, $b=423.83$, $c=2.79$ and $d=0.3$

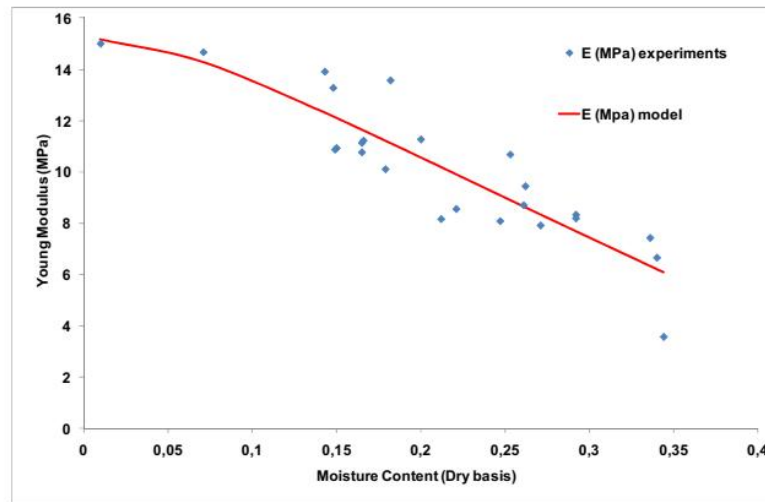


Figure 11. Evolution of elasticity modulus versus water content.

It is worth mentioning that the shape of the curve depicted in Figure 11 matches with that found in the literature on the subject [29,30]. The curve's profile shows that the Young modulus increases significantly in the drying direction (for decreasing water content), up to a value close to 15% of water content where its value becomes almost constant (approximately 15MPa). The results were undertaken for the same operating drying conditions.

We can find that the stiffness gain is also discussed in the case of heating during dehydration for higher temperatures (up to 250 °C) [13]. The difference is that the gain occurring in this work is about 200% increase of the Young's modulus, a remarkable value compared to that deduced during a continuous heating process (only 76% increase in [13]). We could assume that, logically, the porosity formation could affect the Young's modulus: The presence of vacuum during the drying process could make the material less rigid. However, the shrinkage phenomenon causes deformations, which consolidate the material. Therefore, the shrinkage impact exceeds the impact of the pores in this case. This trade-off remains valid until reaching the shrinkage limit (equal to 15% for the studied clay) where the deformations stop taking place and so the rigidity becomes constant.

We recall that the stiffness is used as parameter for structural design criteria. As an exploitation of this result, the determination of the shrinkage limit and thereafter the measurement of the stiffness for a sample having this value of water content will make it possible to deduce its rigidity which will be used for the construction and any other useful sizing purpose.

4. Conclusions

The volume shrinkage of natural clay material during convective drying is characterized in this work by a shrinkage-limit. It is a water content from which, in the direction of drying, the volume shrinkage becomes zero or the sample no longer changes volume. The determination of this value by a single experiment of the shrinkage follow-up (which we have proved to be strictly intrinsic to the material itself) will enable us to conclude on its physical state (purely solid state, no longer deformed), rheological behaviour (of which the law is necessary for the theoretical study of clay materials drying), its density evolution (usable for calculating the weight of a structure or building pieces for example) and its rigidity (needed for shaping and even dimensioning of structures). The shrinkage limit has been highlighted in this study as a critical factor controlling the kinetics of convective drying of natural clay. This critical threshold denotes the point at which the internal structure of the material reaches its maximum compaction and volume shrinkage stops.

The findings emphasize that the evolution of density and porosity was determined by the intrinsic shrinkage-limit found at approximately 15% water content for the studied clay material. We have demonstrated a direct connection between the hydric state and the mechanical behavior of the material by relating these physical changes with Atterberg limits. Notably, figuring out this limit make it possible to forecast how the Young modulus will change, which gives the information needed to describe the stiffness and structural integrity of the material. In conclusion, combining these physico-mechanical characteristics into a single experimental framework provided a reliable way to model unsaturated porous media behaviour.

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Data will be available on demand.

Author Contribution

S. Chemkhi: Conceptualization, Methodology, Investigation, Writing, Review & Editing, Supervision & Validation; K. Khalfaoui: Methodology, Investigation, Experimental Measurements; F. Zagrouba: Project administration, Supervision & Validation.

Conflict of Interest

The authors declare no conflict of interest.

Generative AI Statement

The authors declare that no GEN AI was used in the creation of the manuscript. Only, translation and paraphrasing tools were employed to improve the English language quality.

Nomenclature

E : Young modulus, MPa

$L.I.$: Loss in Ignition, %

m : Mass, kg

R_v : Volume shrinkage, %

T : Temperature, °C

V : Volume, m³

W : Water content, %

W_L : Liquid limit, %

W_P : Plastic limit, %

W_S : Shrinkage limit, %

ρ : Density, kg/m³

ϕ : Porosity, %

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