



## Experimental behavior of natural clay during convective drying: Comparative study between two shapes

Kaouther KHALFAOUI<sup>1,3</sup>

Saber CHEMKHI<sup>2,3</sup>

Fethi ZAGROUBA<sup>3</sup>

<sup>1</sup>Department of Energy Engineering, National Engineering School of Monastir, Tunisia.

<sup>2</sup>Department of Mechanical Engineering, Higher Institute of Applied Sciences and Technologies, Kairouan, Tunisia.

<sup>3</sup>Research Laboratory of Environmental Sciences and Technologies, Higher Institute of Environmental Sciences and Technologies, Borj-Cedria, Tunisia.

[Kaouther.khalfaoui@gmail.com](mailto:Kaouther.khalfaoui@gmail.com)

[saberchemkhi@yahoo.fr](mailto:saberchemkhi@yahoo.fr)

[fethi.zagrouba@isste.rnu.tn](mailto:fethi.zagrouba@isste.rnu.tn)

### Abstract:

Drying is a key step in clay-based manufacturing, in which the shrinkage has to be controlled. Dealing with this geometrical phenomenon, the shape (during the shaping step) remains to be discussed so we can manage the drying experimental parameters (temperature, heat flow, power...). A series of convective drying experiments were carried out for different experimental conditions, using natural clay extracted from a region in Tabarka and basing on cylindrical and parallelepiped shapes, in order to conclude on their effects on the mass loss and decrease in volume. As results, the shrinkage is independent of the shape (same final shrinking rate). However, the drying kinetics differs: the dynamics of the constant and falling rate periods are reversed.

### Keywords:

Convective Drying; Clay; Shape; Drying Kinetics; Shrinkage.

### Introduction

Several researchers have contributed to the understanding of the drying modeling of shrinkable products [1-6]. The clay materials, a timeless material in constant evolution, occupy a preponderant place in the building construction (tiles, bricks and flooring, ceramics...). Responding mainly to the respect of the environment and economy (an industry classified as an energy consumer), the study of the fired products has interested the researchers in modeling studies [2, 4, 6-9] and experimental characterization [3, 6 and 10]. To specify, we have chosen to concentrate on the shrinkage phenomenon known as deformations in different directions causing, in many critical cases, the cracks in the product.

Dealing with the drying as step following the shaping one, the sample's geometric form could be an important factor to discuss in order to optimize the process (time, drying parameters...). This idea is developed in this experimental work. In fact, we have tried to highlight the shape effect on the behavior of the samples during convective drying: mass loss to describe the drying kinetics and the decrease in volume to characterize the shrinkage phenomenon for natural clay.

To do it, we will present the experimental device developed for the drying kinetics as well as the measurement method used for the follow-up of volumetric shrinkage. We will detail some results of comparison to conclude on the shape effect.

### 1. Materials and Methods

The model material is natural clay extracted from Tabarka, a region in Tunisia. It is found formed mainly of kaolin (see [11] for more details of mineral characterization). Cylindrical and parallelepiped samples were prepared by varying the size ratio in order to show the scale effect.

### 1.1. Behavior during drying: drying kinetics

Samples are prepared by mixing the powdered clay with controlled quantity of water. The prismatic shape is made with cake paste respecting the desired dimensions. The cylindrical ones are prepared using syringe with different dimensions.

Convective drying tests are carried out using convective drying loop available at the Thermal Processes Laboratory (LPT) at the Borj Cedria Technology Center. The experimental installation is shown in figure 1.



(a) Temperature control



(b) A view of the drying loop



(c) Data acquisition balance

Figure 1: Convective drying loop (Thermal Processes Laboratory (LPT) in Borj Cedria)

Drying parameters (Temperature ( $T$ ), Relative Humidity ( $RH$ ) and Air Velocity ( $V_{air}$ )) are fixed and controlled (Figure 1(a)). The experimental protocol is standard one used in many references [9, 11 ...]. However, the boundary conditions differ. To clarify it, schematic representation is proposed in Figure 2. In fact, the sample is placed on the support above the scale (Figure 1(c)). The drying process is then started: the specimen is subjected to a convective air flow whose temperature took values ranging from 30 °C to 70 °C. The subsequent mass measurements of the sample are tracked and recorded via a computer interface (Figure 1(b)). The test is stopped when the sample mass is kept constant (value of equilibrium with the external conditions). The specimen is then dried in an oven at 105 °C during 24 hours to get the dry mass (and so to calculate the corresponding water content).

This procedure was repeated for cylindrical and parallelepiped specimens with very close water content. Curves of kinetics drying (mass loss versus time) are set out for different drying conditions. In order to be able to compare the different evolution curves, it was recommended to use the concept of reduced water content: we refer the quantity of water content to the initial value for each drying test.

We mention that we have concentrated only on temperature and relative humidity variations in this experimental work.

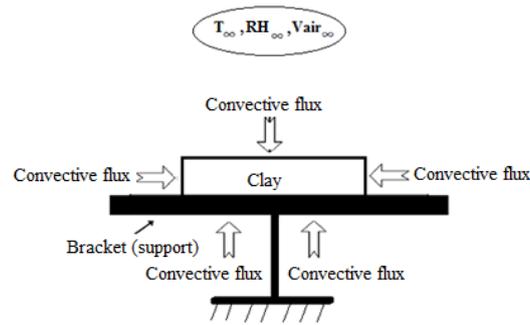


Figure 2: Boundary conditions of convective drying

Series of experiences were carried out by varying the drying conditions in order to describe and compare kinetics drying. Some conditions are announced in Table 1.

Table 1: Experimental conditions of convective drying

Test n°	Air velocity (m/s)	Temperature (°C)	Relative Humidity (%)
1	1.5	30	30
2	1.5	60	5
3	1.5	70	5
4	1.5	70	10
5	1.5	60	10
...	...	...	...

## 1.2. Behavior during drying: Measuring shrinkage

As "the volume shrinkage is a macroscopic characteristic dependent on the quantity of water existing in the material for low and medium temperatures" [4], the experiments were performed on specimens with water content close to 40% (dry basis) at a temperature equal to 70° C.

The procedure is summarized in sample volume monitoring during drying, using a caliper. It was put in an oven and discharged at different drying times in order to measure its dimensions and mass. The tests were undertaken on cylindrical and parallelepiped specimens. After taking the necessary data for tracing curves, shrinkage was calculated from the ratio of volume compared to the initial volume of the sample ( $V/V_0$ ).

## 2. Results and Discussions

We will focus in this part on the experimental results, by comparing the curves profiles after changing the form and dimensions, in some cases, of the samples.

### 2.1. Behavior during drying: drying kinetics

#### 2.1.1. Profile of the drying curve

Curves of the water content versus time are drawn. An example for a cylindrical form is given in Figure 3. We can see that the profile follows a decreasing pattern: It's a loss mass phenomenon because of the drying.

After a setting up period named as the initiation period, we often observe a drying period at constant speed and then one or two periods of decreasing rate [4, 7, 11...]. Figure 3 shows clearly the second and the third phases. Note that the absence of the first period can be justified by the samples dimensions: The sample is quite small; the time of temperature homogenization is incomparable to that total of drying. It's recommended to represent the mass flux density versus time if we want to show this detail.

Figure 3 represents also the temperature's influence on the drying speed: For a high temperature, the product takes less time to reach equilibrium water content with the external drying environment.

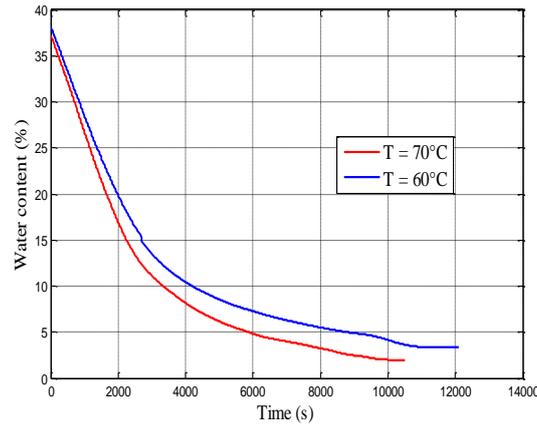


Figure 3: Water content versus time for cylindrical sample (RH=10%;  $V_{\text{air}}=1.5\text{m/s}$ )

### 2.1.2. Dimensions effect on the drying curve

Cylindrical samples with different size ratios were prepared in order to conduct drying experiences and conclude on the dimensions effects (see Figure 4). The size ratio ( $r$ ) is defined as the one between the diameter ( $d$ ) and the height ( $H$ ) of the cylinder clay.

For a ratio  $r = 1$ , the cylinder dimensions are chosen equal to  $d = H = 20\text{mm}$ .

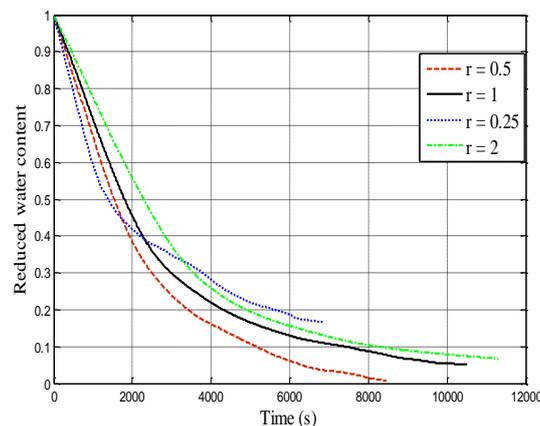


Figure 4: Size ratio ( $r$ ) effect on the drying of cylindrical clay ( $T=70^\circ\text{C}$ ; RH=5%;  $V_{\text{air}}=1.5\text{m/s}$ )

We can assume that the voluminous is the product; the longer is the drying time. If we are interested in discussing the drying physical configuration (from Figure 4), we can note that there's a difference in the process dynamics. In fact, when we talk about privileged material transfer sense similar to the convective drying flux ( $r=0.25$ ), the phase at constant speed is achieved in shorter time. However, compared to the configuration when the material transfer sense is perpendicular to the convective drying flux ( $r=2$ ), its last drying phase occurs with less accelerated speed.

### 2.1.3. Reproducibility of the drying experiences

Similar drying tests ( $T = 70^\circ\text{C}$ , HR = 10%,  $V_{\text{air}} = 1.5\text{m/s}$ ) were carried out on identical parallelepiped samples of the natural clay (Figure 5). The comparison of these experiments certifies the reproducibility of the measurements made. The deviations are essentially due to data recording errors.

If we compare the profiles of the two chosen geometries at the same drying conditions, we can see that the phase at constant speed ends after 2 hours of drying for parallelepiped specimen clay (Figure 5). Nevertheless, it's achieved with shorter duration for cylindrical specimen (Figure 3). This period is marked by contraction phenomenon of the solid structure in order to fill the voids left by the evaporated water: we talk about the volumetric shrinkage which will be discussed in next paragraph.

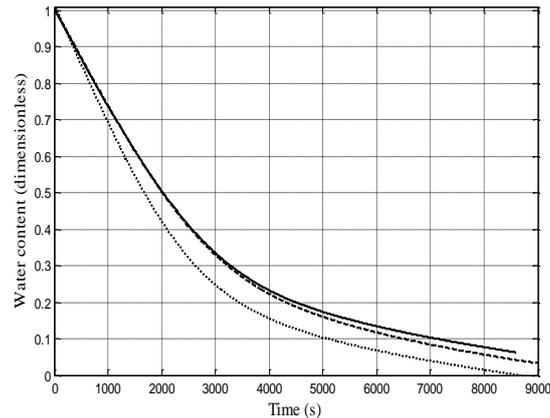


Figure 5: Water content evolution for parallelepiped samples. Reproducibility of our tests ( $T=70^{\circ}\text{C}$ ;  $\text{HR}=10\%$ ;  $V_{\text{air}}=1.5\text{m/s}$ )

#### 2.1.4. Comparison of the drying kinetics for the two different geometries

Convective drying process has been applied to two different samples under similar drying conditions ( $T = 60^{\circ}\text{C}$ ,  $\text{HR} = 5\%$  and  $V_{\text{air}} = 1.5\text{m/s}$ ). Figure 6 describes schematically the dependence of the kinetics drying on the sample's form (or geometry).

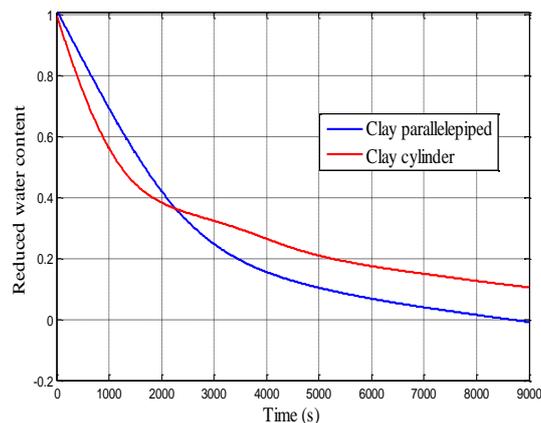


Figure 6: Water content profiles for two different samples geometries ( $T=60^{\circ}\text{C}$ ;  $\text{HR}=5\%$ ;  $V_{\text{air}}=1.5\text{m/s}$ )

Plotted on the same axes, the curves evolutions testify the results announced above: a cylinder takes less time to reach the end of the second phase named period at constant speed. However, the kinetic for the discussed form slows down during the last drying phase. We remind that the hydrothermal transfers are accompanied by transformations in the solid matter of the studied medium: we are talking about volumetric shrinkage. This phenomenon will be discussed and analyzed in the next part of this paper.

## 2.2. Behavior during drying: Measuring shrinkage

### 2.2.1. Profile of shrinkage

The shrinkage follows a decreasing trend until canceling out (Figure 7). The water content corresponding to zero shrinkage is called the shrinkage limit (noted in this work  $w_s$ ) [4]. From this value, the product does not change volume.

Figure 7 below shows the change in volume (relatively 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's shown that the final volume measured at the end of drying shows 75% of the initial volume of the studied natural clay. This means that the loss in volume presents only 25% of the initial volume. These values can be discussed in accordance with the behavior of clay in general [4, 10, 11...], 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 (90%) on dry basis.

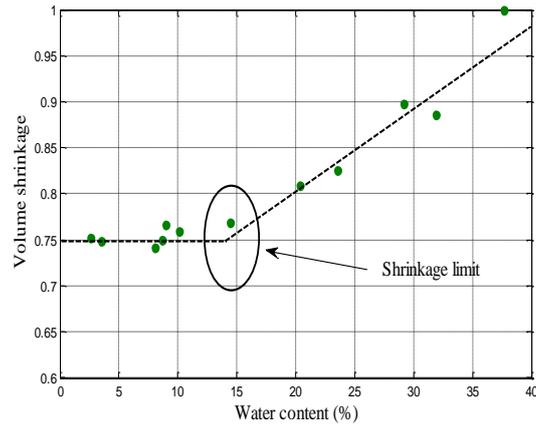


Figure 7: Variation of the volume shrinkage as a function of moisture content. Shrinkage limit highlighting

### 2.2.2. Shrinkage profiles for two different samples geometries

To study the effect of changing the shape, Figure 8 is represented to plot the evolution of the volume for a parallelepiped sample. It shows the same results obtained for a cylindrical sample and analyzed above.

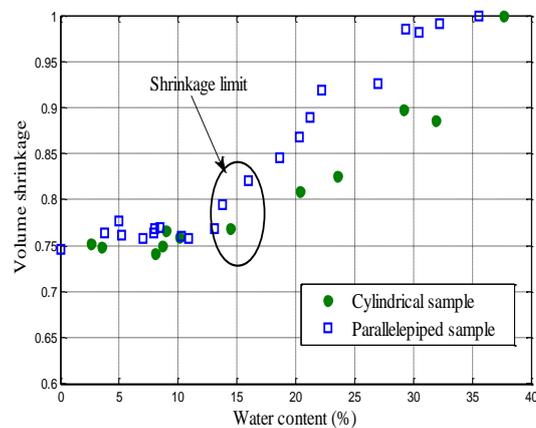


Figure 8: Geometry's effect of the sample during monitoring the volume decrease

### 2.2.3. Reproducibility of the shrinkage phenomenon

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 two parallelepiped samples (same preparation) at the same temperature condition ( $T = 70^{\circ}\text{C}$ ) using the same measuring instruments (scales, calipers). The curves are plotted in Figure 9.

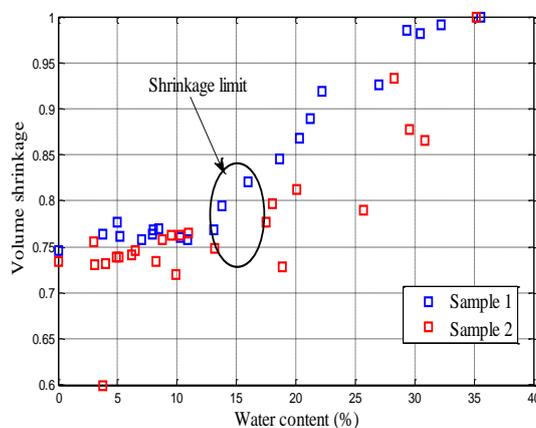


Figure 9: Reproducibility of the shrinkage phenomenon

According to Figure 9, these independent experiments show that the results are remarkably reproducible and clearly consistent with the results of characterization studies of porous media.

## Conclusion

The objective of this study was to determine the shape effect during drying of natural clay. A similar work was done by [12]. It was numerical study to improve the thermal performance of red clay holed bricks (modeling parameters of the heat transfers). The author focused on the protuberances presence (material added to the bricks for wall buildings) using the same red clay and the same thickness.

This present work is an experimental observation of the shape influence on the drying kinetics and volume shrinkage for filled clay material samples during convective drying. The results demonstrate that the reproducibility of the experiments give us similar behaviors. We conclude that the shrinkage is independent of the shape (same final shrinking rate and same shrinkage limit approximately). However, the drying kinetics differs: the dynamics of the constant and falling rate periods are reversed. This finding could influence the thermal performance by managing the heat transfer parameters.

## Nomenclature

Notations can be used with different meanings. In general, the context removes any confusion.

Symbol	Name, unit
d	diameter, <i>m</i> (or <i>mm</i> )
H	height, <i>m</i> (or <i>mm</i> )
r	size ratio
RH	relative humidity, %
T	temperature, °C
V	velocity, <i>m/s</i>
V	volume, <i>m</i> <sup>3</sup> (or <i>mm</i> <sup>3</sup> )

## References

- [1] W Jomaa and J R Puiggali, Drying of shrinking materials: Modelling with shrinkage velocity. *Drying Technology*, Volume 9, N°5, Pages 1271–1293, 1991.
- [2] S Chemkhi, F Zagrouba and A Bellagi, Mathematical model for drying of highly shrinkable media, *Drying Technology*, Volume 22, Pages 1023–1039, 2004.
- [3] S J Kowalski, K Rajewska and A Rybicki, Mechanical effects in saturated capillary-porous materials during convective and microwave drying, *Chemical Engineering Science*, Volume 22, N°10, Pages 2291–2308, 2004.
- [4] S. Chemkhi, F Zagrouba And A Bellagi, Drying of ceramics: modeling of the thermo-hydro elastic behavior and experiments, *Industrial ceramics*, Volume 22, N°2, Pages 153-163, 2005.
- [5] F Couture, S Laurent and M A Roques, Drying of two-phase media: Simulation with liquid pressure as driven force. *AIChE Journal*, Volume 53, Pages 1703–1717, 2007.
- [6] I. Hammouda et al., Changes in the physico-mechanical characteristics of a ceramic paste during drying, *C.R.Mécanique*, 2015, <http://dx.doi.org/10.1016/j.crme.2015.06.001>
- [7] S Chemkhi, F Zagrouba, Development of a Darcy-flow model applied to simulate the drying of shrinking media, *Brazilian Journal of Chemical Engineering*, Volume 25, N°3, Pages 503-514, 2008.
- [8] K Khalfaoui, S Chemkhi and F Zagrouba, Modeling and stress analysis during drying of a deformable and saturated porous medium., *Drying Technology*, Volume 31, Pages 1124-1137, 2013.
- [9] M Ben Abdelhamid, D Mihoubi, J Sghaier and A Bellagi, Strain-stress formation during stationary and intermittent drying of deformable media, *Drying Technology*, Volume 32, Pages 1245-1255, 2014.
- [10] I Hammouda and D Mihoubi, Thermodynamic and mechanical characterization of kaolin clay, *Polish Journal of Chemical Technology*, Volume 16, N°1, Pages 28-35, 2014.
- [11] S Chemkhi, K Khalfaoui and F Zagrouba, Physico-chemical and mechanical behavior of natural clay as a porous medium during convective drying, *American Chemical Science Journal*, Volume 6, N°3, Pages 126-135, 2015.
- [12] V.A.F Costa, Improving the thermal performance of red clay holed bricks, *Energy and Buildings*, Volume 70, Pages 352-364, 2014.