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DETERMINATION OF THE BIGOT CURVE BY DRYING TESTS OF CERAMIC SAMPLES

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Abstract. *The drying process of the clay in the brick and tile industry is a special detail that deserves close attention. Among several significant information to assess the drying of ceramics, one of the most important is the Bigot curve. This particular data represents the variation of linear shrinkage as function of the humidity of ceramic samples. Therefore, this work aims to set up an experiment to analyze the drying behavior of ceramics and build the Bigot curve, as well as to appraise the results in order to indicate a way to optimize the process.*

Keywords: *Bigot Curve, Ceramics, Drying Process.*

1. INTRODUCTION

On the present work, the drying process is analyzed by the Bigot Curve optic. This curve is a graphic representation of water content's variation as a function of linear shrinkage percent. Details of the physical process that occurs during the initial stage of drying, with the major shrinkage of the object, and the final phase are presented by Vieira et al (2003). Thus, in the initial phase a large volume of water is removed from the samples, resulting in great reduction of dimensions, which depends on the material and the initial percent of moisture. In the final stage there is still a decreasing reduction of water content, however with a much smaller volume retraction.

2. THE DRYING PROCESS

The process of drying represents one of the most important stages on the production of structural clay ceramics, as the drying provides stability for the firing stage occurring. Without proper caution before the firing, some problems that may render the ware useless (like distortions and cracks) may arise due to non-uniform shrinkage along the product's body.

According to Callister, 1999, before the drying process begins, the clay particles are apart from one another by a thin film of water, but as the drying advances, this film reduces and the distance between the clay particles decreases until the ceramic is completely dried. As the evaporation occurs on the surface, the drying of the interior regions of the product's body is accomplished by the diffusion of the water molecules to the outer side. If the drying proceeds too fast and the evaporation rate is greater than the diffusion rate, the product will shrink unevenly, as the surface dries and shrink more rapidly than the interior, and there will be a high probability of the formation of several defects. Therefore avoiding issues like this is critical for achieving quality and can be accomplished by drying with controlled conditions such as temperature, humidity, and the rate of airflow.

2.1 Test methodology

At the beginning of the procedure, twelve samples (six prismatic and six cylindrical) were removed from six blocks of pre-cast bricks (blocks 1, 2, 3, 4, 5 and 6) for drying. For the acquisition of the prismatic samples, six pieces have been cut off with dimensions around 10mm x 20mm x 85mm from the blocks with a sharp blade, while for the cylindrical, bigger pieces have been removed from each block and individually re-extruded with a shape of cylinder with approximate dimensions of 27 mm diameter and 60 mm high. Each sample has had its weight and dimensions noted, and has been placed on a special tray, covered with aluminum foil, as in Fig. 1. A grid has served as a support to ensure a good air circulation around all surfaces. At each test, the set was placed in a pre-heated ventilated oven (as shown in Fig. 2) at 60ºC and each sample had its weight, and length of corners measured frequently.

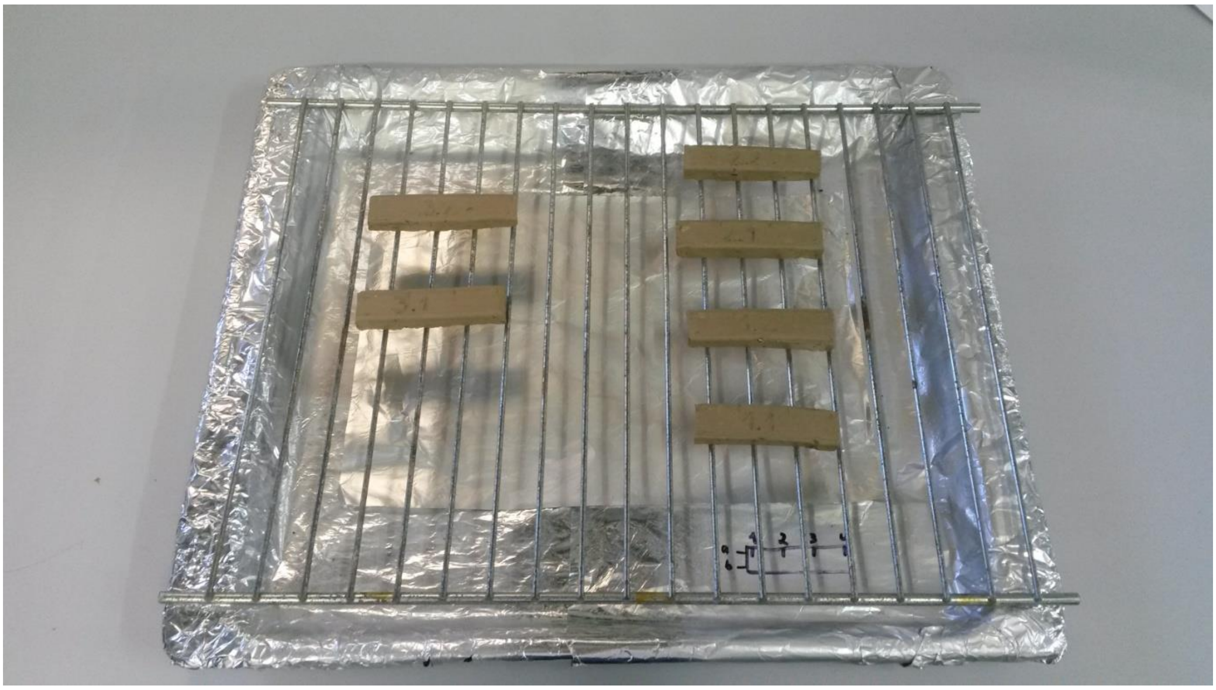

Figure 1. Samples on the grid and coated in aluminum foil tray for drying.

Figure 2. Tray with samples inside the greenhouse for drying.

Each prismatic sample has received two marks on its width and four on its length, while each cylindrical piece has received four on its length and three on its diameter to assist in the measurements. The final measures of each noting period were obtained from the average of the width, height and length.

After calculating the initial volume (V_0) and the volumes of each measurement taken (V), the estimated percentage of volume retraction (R) for each measurement is calculated by Eq. (1).

$$R = 1 - (V / V_0)^\wedge(1/3) \tag{1}$$

The total retraction (TR) is calculated at the end of the drying, and obtained by the difference of the retraction of the final measurement (FR) and the retraction above calculated (R), as shown in Eq. (2). It indicates the retraction that will occur from each measurement until the end of drying. These values will be used for the graph's abscissa of the Bigot curve.

$$TR = FR - R \tag{2}$$

In the drying stage, nothing besides water is removed from the original piece, therefore all the water lost is equivalent to the mass lost. The amount of water present (U) is calculated as the difference of the mass noted on each period (M) and the final mass taken (FM), according to the Eq. (3).

$$U = FM - M \tag{3}$$

2.2 Test results

2.2.1 Test results of prismatic samples

In Fig. 3 it is possible to observe the mass loss over time, which was relatively similar in all samples. It is also possible to observe that in the first hour, all samples lost a large amount of the total water content present. Smaller progressive reductions can be observed from the second hour and on of drying.

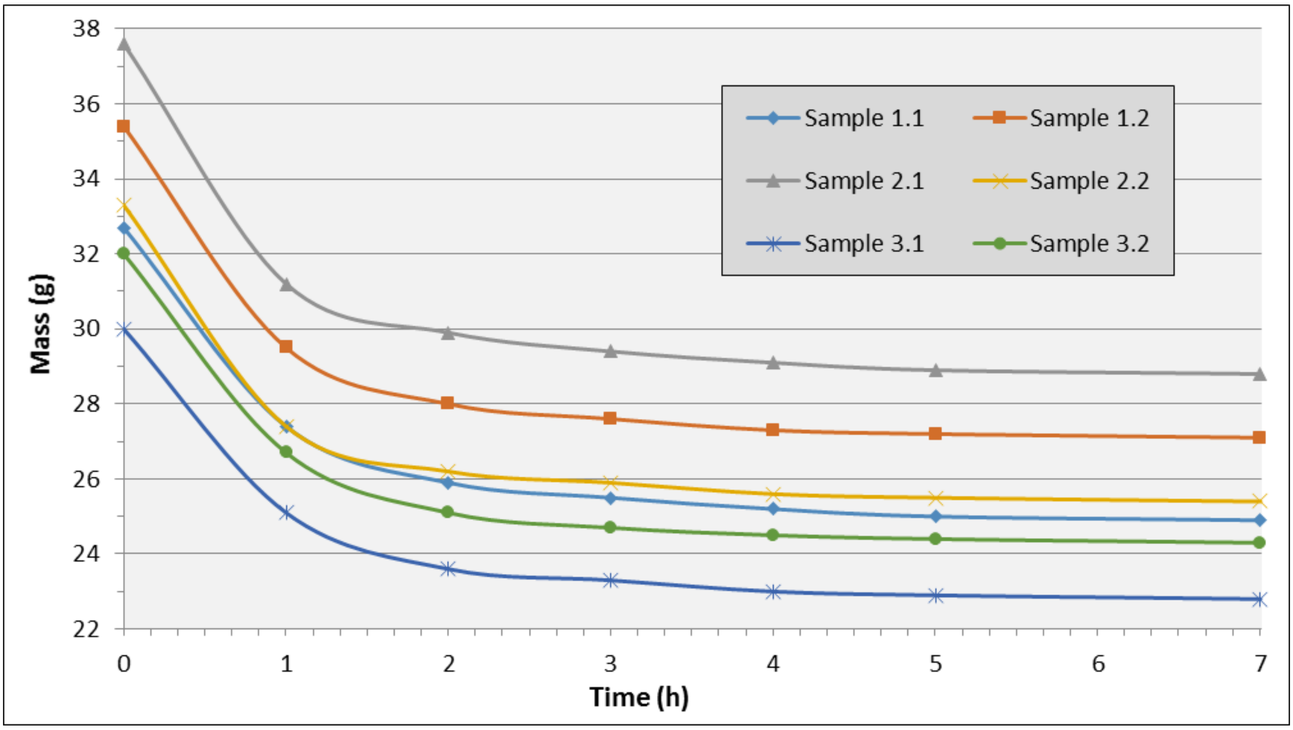

Figure 3. Diagram of the reduction of mass over time.

Figure 4 shows the reduction of the volume of every sample, with a similar behavior of the mass loss. The retraction of volume indicates the shrinkage as a consequence of water evaporation.

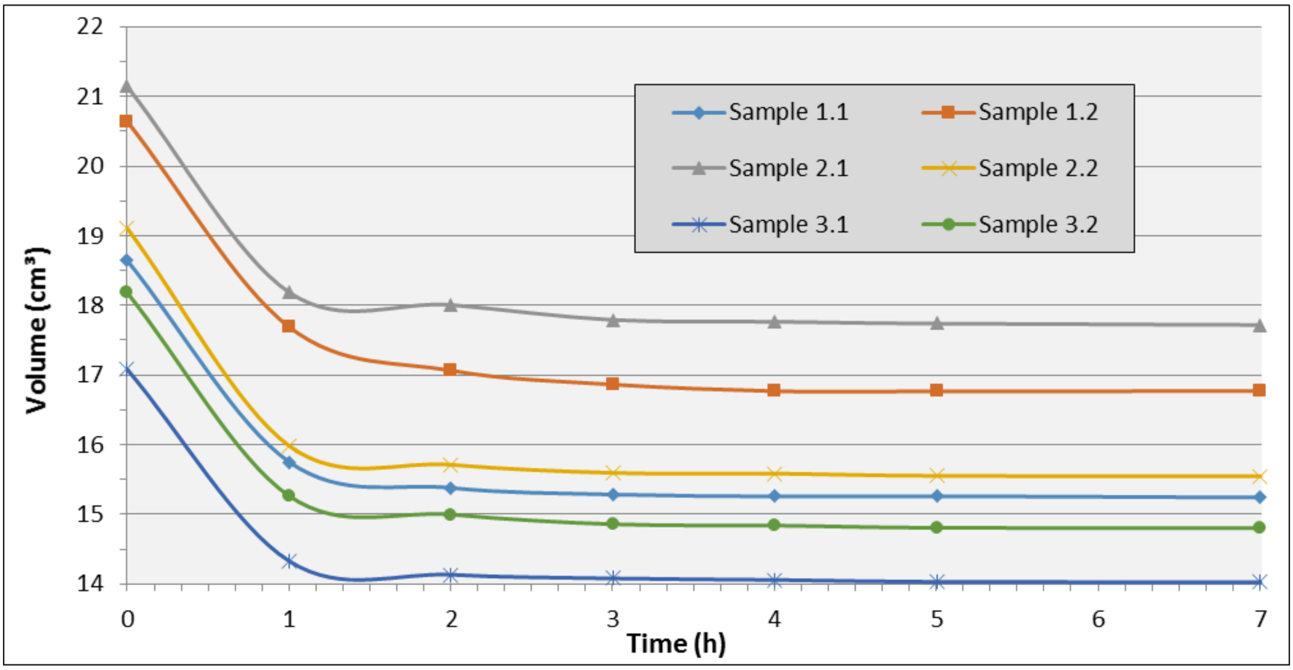

Figure 4. Diagram of shrinkage of volume over time.

Figure 5 presents the volumetric moisture content, expressed as mass of water per volume unit, showing that this content has also progressively reduced. Because it is a specific value, its calculation results on the superposition of the curves of the six samples, with some initial values dispersed.

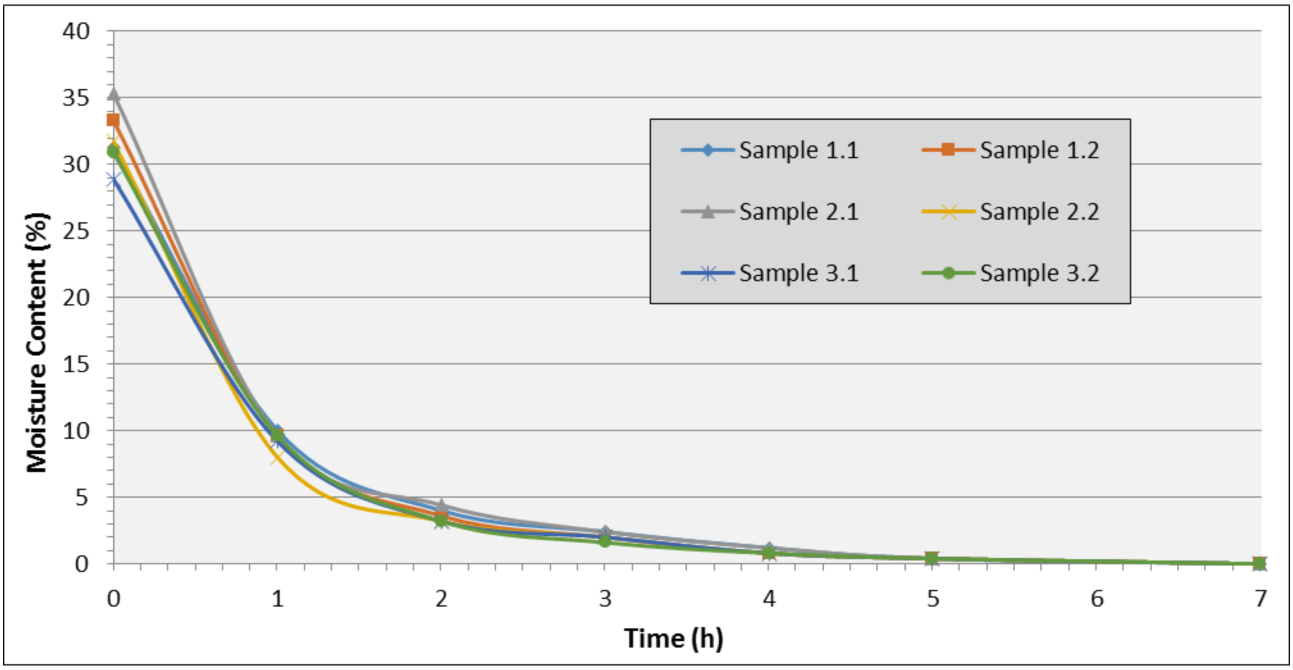

Figure 5. Diagram of lose of moisture content over time.

With the presented results, it is easy to obtain the values on Tab. 1, whose points refer to the regression line of the Bigot curve's linear retraction portion. This part is also known as the first stage of drying and is where samples have the largest retraction as moisture content decreases. The intersection point represents the value of the ordinate (humidity [%]) when sample's retraction reduces significantly. The slope of line represents the ratio between the moisture percentage variation and the shrinkage percentage and it shows a linear relationship between the two variables.

Table 1. Data about the regression line of Bigot curve for each sample.

Sample	1.1	1.2	2.1	2.2	3.1	3.2	Average	Dispersion
Intersection Point (%)	3.36	1.23	2.63	2.43	3.34	2.58	2.59	0.51
Slope of Line (% MC/ % TR)[1]	4.35	4.81	5.76	4.44	4.06	4.33	4.63	0.44

(1) MC – Moisture Content; TR – Total Retraction

Figure 8 refers to the average Bigot curve of the six samples considering the values of Tab. 1. This curve allows the analysis of samples' retraction as they lose water content. The retraction is virtually constant as the moisture is removed, showing that the spaces left by water are being occupied by clay - Schwartz et al. (2005). This behavior continues until the moisture content reaches about 3%, from that on the moisture is still being removed, but with no more significant retractions.

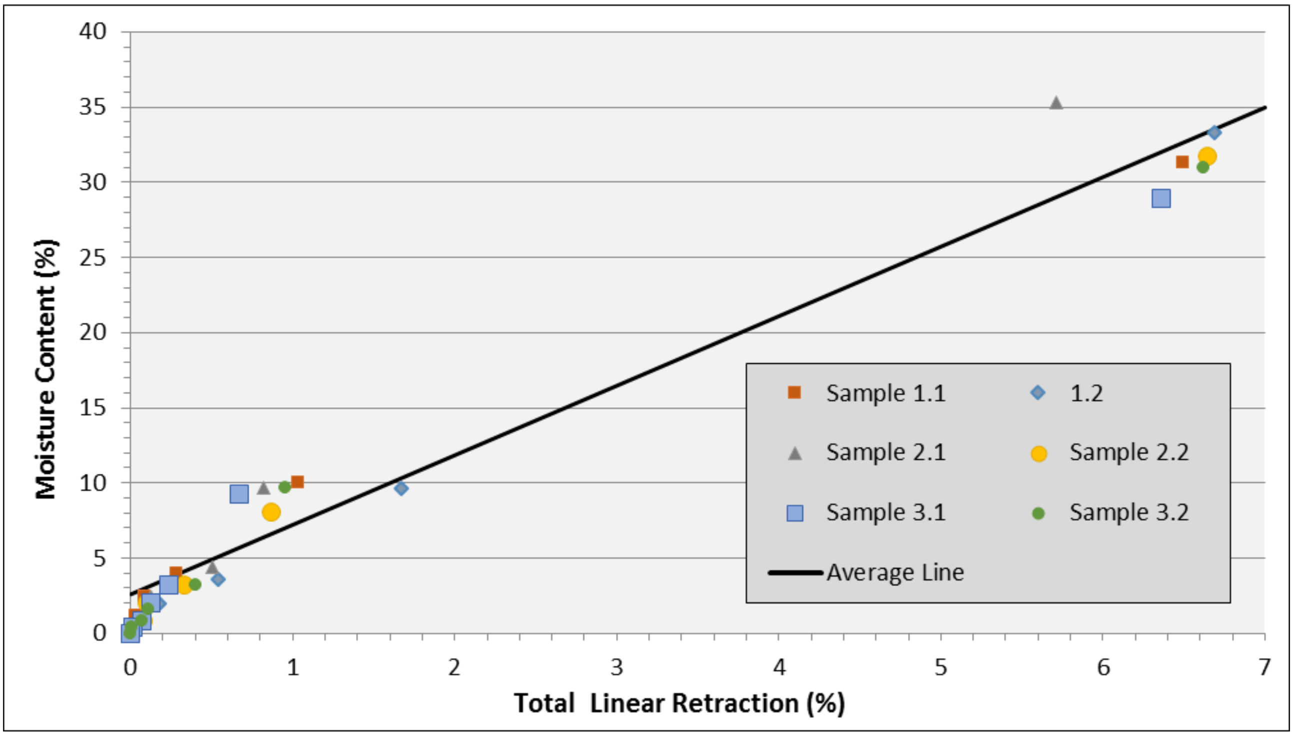

Figure 6. Average Bigot Curve, obtained from six samples.

Figure 6 also shows that there is a certain dispersion of the results, partly due to the imperfections of the samples, which were obtained by slicing pieces from the production line. Although they have been measured hourly during drying, the dots indicate that there is a need for a greater frequency of measurement at the beginning of the process, to better define the slope of the curve at the largest retraction, as done with the cylindrical samples.

2.2.2 Test results of cylindrical samples

In this case, cylindrical samples were obtained in the laboratory, using a mold designed for this purpose. The change took place due the difficulties of measuring prismatic pieces, which were cut directly from extruded pieces.

In Fig. 7 and Fig. 8, the charts show the samples' mass variation over time of drying. In the first case the ceramics were dried for more than 50 hours, while in the second case, they were dried between 26 and 30 hours. However, the largest mass variations occurred during the first 3 hours, with a fast reduction of the curve's slope after 6 or 7 hours. The points were concentrated in the early hours, because these are the most important for later analysis and because after 20 hours of drying nothing more happens, as seen in Fig. 9.

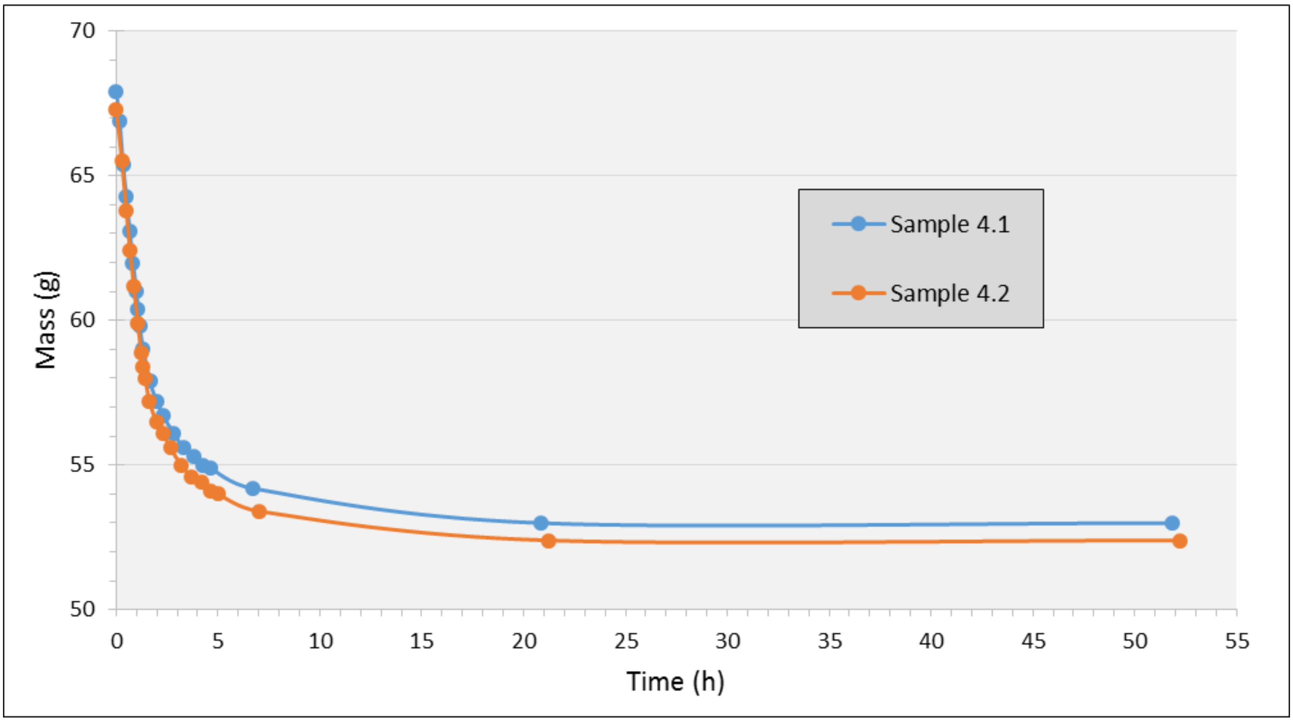

Figure 7. Reduction of masses over time, for 25 to 30 hours of experiment.

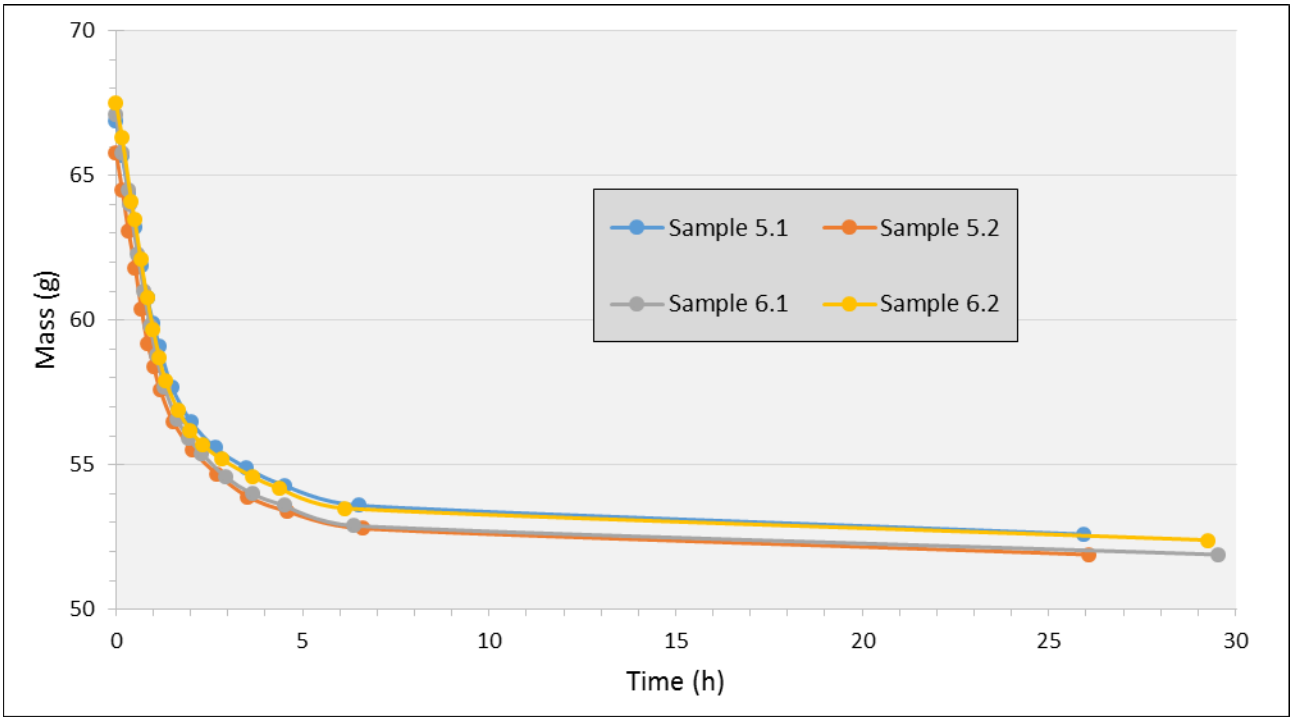

Figure 8. Reduction of masses over time, for about 50 hours of experiment.

In Fig. 9, it is possible to evaluate the cumulative shrinkage over time. Major retractions can be observed in the first two hours, when they reach an average of 5% of the original body. Some samples retract shortly thereafter, but nothing greater than 0.3%.

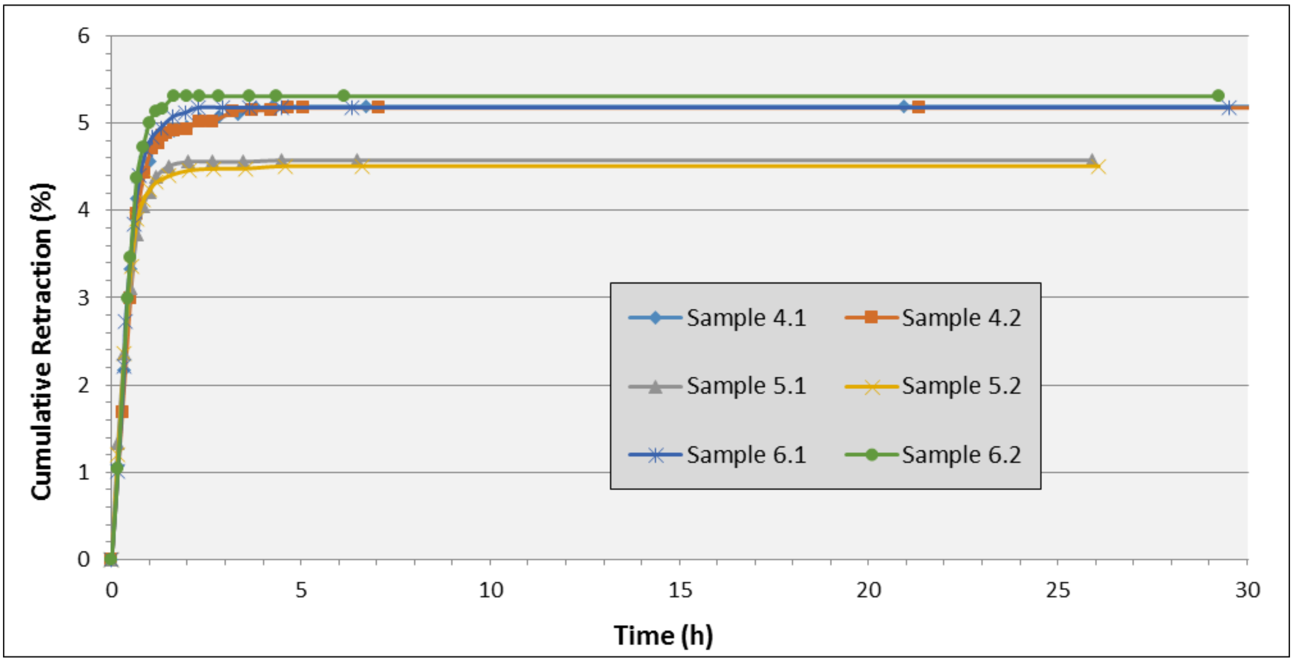

Figure 9. Diagram of cumulative linear retraction over time, for the first 30 hours.

In Fig. 10, the chart presents the percentage of moisture in the samples, whose curves are similar to total masses' charts. The curves indicate that from a moisture content of 29% to almost fully dried piece, the process took around 20 hours. From this point on, variations cease on every physical aspects.

After 3 hours, the samples reach moisture content around 5% and after 6 hours, a value near 2%, which is practically the acceptable moisture content to the pieces that leave the dryer. With this amount, the piece could be forwarded to the kiln for burning, because from this point on, as could be seen on Fig. 9, it retracts barely nothing and shrinking is no longer a problem, as it does not compromise the integrity of the structure anymore. There aren't many points after 7 hours because no longer occur large variations from one point to another and they are already very close to the origin points on the Bigot curve, on Fig. 11.

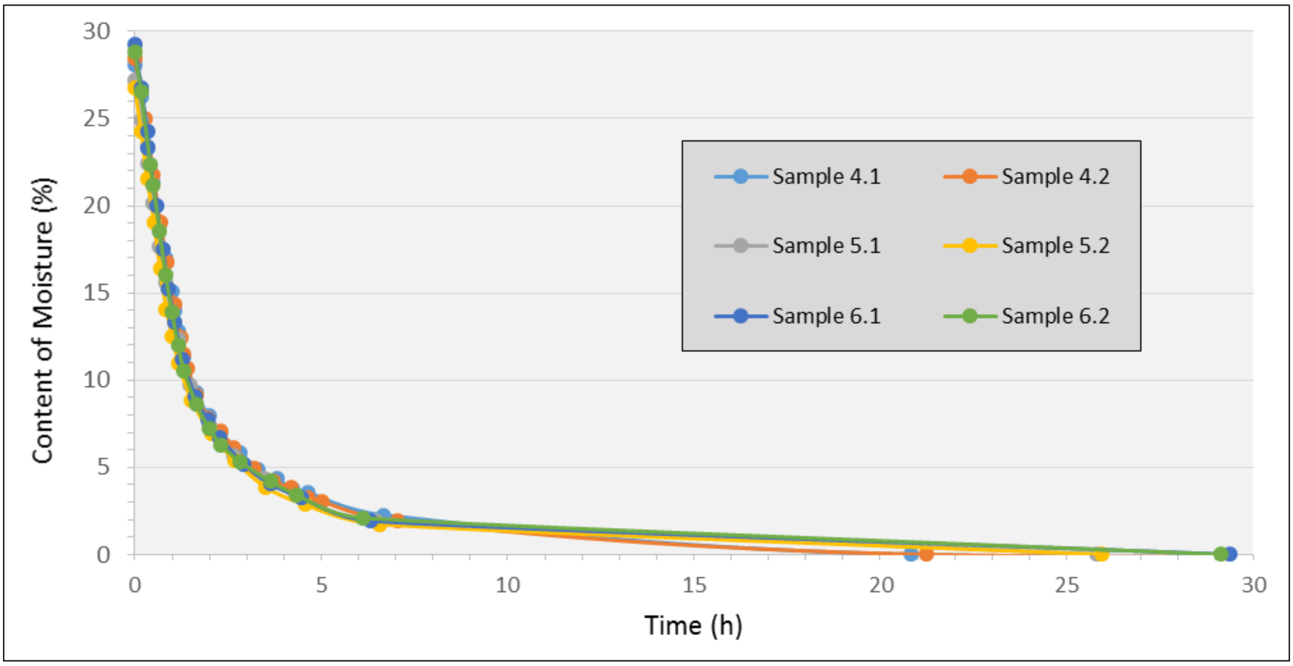

Figure 10. Diagram of moisture content over time, for 30 hours.

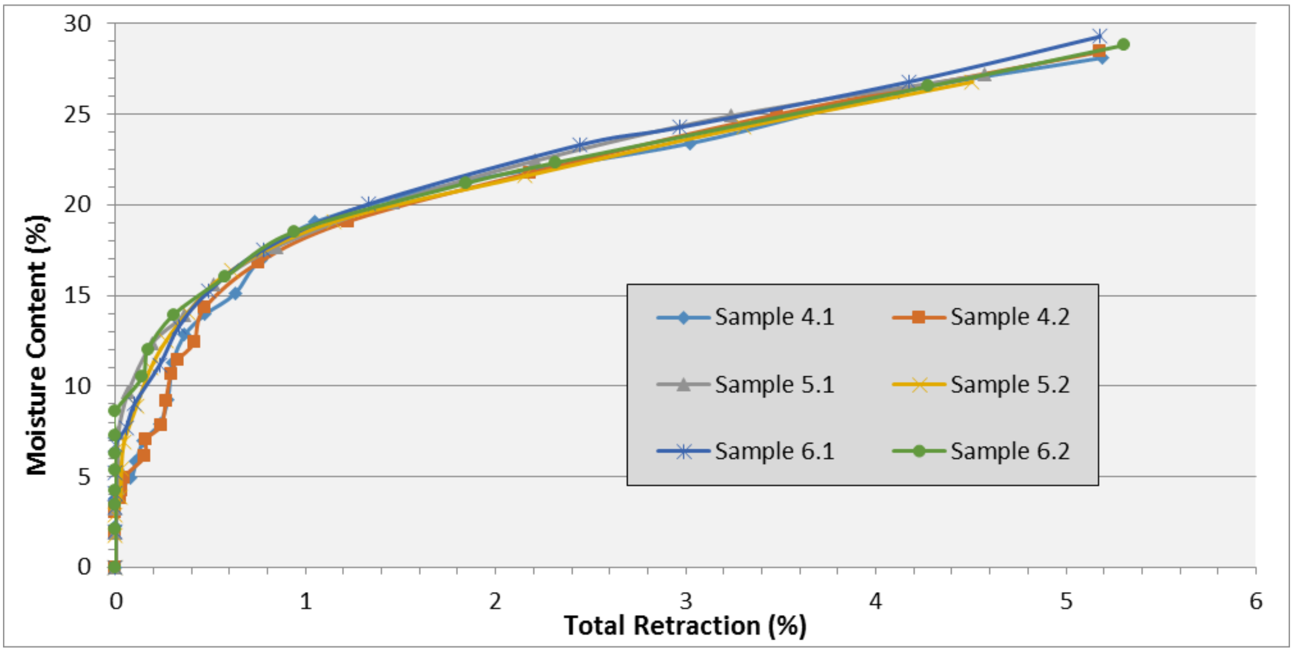

Figure 11. Bigot curve.

The moisture content as a function of the total shrinkage is shown in Fig. 11, which features the Bigot curve. This chart shows that the most important aspect in terms of retraction occurs with moisture content up to 15%, which on previous curves indicates the first 2 to 3 hours of the total drying period. These results are more expressive than those from Fig. 6, in which there are no intermediate points, providing only a simplified form of the curve of Bigot, as presented on the references.

3. CONCLUSION

The results are similar, since the samples are made of the same clay and start with almost the same initial moisture content. Even so, on prismatic samples, there were problems with the geometry when cutting the pieces off, while on cylindrical samples this geometric issue was not so relevant. However, there was some difficulty in obtaining a uniform compaction for the cylindrical ones, once the filling of the extruding mold was made by hand. With this in mind, it is possible to assume that these shall be the reasons of the results' dispersion.

The large initial shrinkage on the drying can present a risk to clay made objects, since it can promote the emergence of cracks. Thus, it is recommended a reduced initial drying rate, as a way of reducing risks to the material. As the geometry is an important factor, this work cannot estimate data for samples with complex geometry; however, for small, thin samples in a well-ventilated space at around 60°C it is acceptable to increase the drying rate after 3 hours, once most of the shrinkage has already happened.

4. ACKNOWLEDGEMENTS

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6. RESPONSIBILITY NOTICE

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