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PHYSICAL-MECHANICAL CHARACTERIZATION OF A CLAY SAMPLE THROUGH PRELIMINARY TESTS

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Abstract. *This work presents the physical-mechanical characterization of a clay sample by means of preliminary tests. Initial tests of moisture percentage, linear retraction at a temperature of 110 ºC, granulometric distribution, chemical elements verification and determination of bending rupture stress were performed. As results, it was found the values of moisture, linear retraction and the denomination of the clay type, according to the chemical elements present that will serve to determine the possible use of the clay sample in the industries.*

Keywords: *Clay. Characterization. Preliminary tests.*

1. INTRODUCTION

The knowledge of the raw materials, in terms of their characteristics, is of utmost importance for obtaining the ceramic products, in view of the quality standards pre-established by the companies. The good performance of the ceramic process stages depends very much on the controls performed on the raw materials. These controls begin with the sampling system, which consists of the sampling procedures, including the natural drying of the clays, sampling, scaling and ending with the preliminary ceramic tests (Dondi, 2006).

The ceramic process will be successful if it is backed by the guarantee of the raw materials quality, resulting in products of great quality, with reduced manufacturing costs. In this way, preliminary ceramic tests aim to study the physical and mechanical characteristics of a material to define its application in the industry and to predict the individual uses of raw materials from the simple ceramic tests (Rêgo, 2010; Norton, 1973).

Laboratories that study raw materials are often asked to study unknown clays, predicting, whether or not, they may have industrial use. It is generally used both for plastic raw materials (clays, kaolins, etc.) and for ceramic compositions (coating masses, sanitary ware, etc.), and by the results of the tests the raw materials can be classified by comparison its applicability in ceramics.

Preliminary tests have as their main objectives to predict, adapt or suggest new uses for raw materials that are used only for an industrial purpose and to compare raw materials already used with new raw materials, aiming at the substitution and characterization of ceramic masses. As a primary advantage, the preliminary tests use a common ceramic approach and unsophisticated equipment. Thus, the present work seeks, through preliminary tests, to perform a physical-mechanical characterization of a clay sample for industrial applications.

2. MATERIALS AND METHODS

To begin the preliminary tests, a portion of material or a group of units is withdrawn from a large mass or assembly which will subsequently be reduced to smaller samples by the screening process. Subsequently, approximately 10 kg of a sample of clay material was collected and placed in a clean place for the homogenization of the samples (SENAI, 2005). Then a cone-shaped mound is made, and the mound is scattered with a shovel. The scattered material is divided into four equal parts in a cross-section, two of these four parts being discarded and the remaining parts and the process is restarted from the homogenization until a representative sample is obtained, corresponding to a sufficient quantity of clay to carry out the laboratory tests (SENAI, 2006).

Then, twelve samples were prepared for laboratory tests. The specimens were extruded (Fig. 1a) at a vacuum of 25 in/Hg, measuring approximately 12.0 cm x 5.0 cm x 1.5 cm (length, width and thickness, respectively) and burned at a high temperature (950 °C) to characterize the samples. After this, the specimens were oven dried at 110 °C (Fig. 1b).

The firing was carried out in a laboratory oven at a temperature of 950 °C. The heating rate was 3 °C/min., and the dwell time at the landing temperature was 60 min. Cooling was performed by turning the oven off and maintaining the specimens inside. From these samples, six were selected for the tests at the Materials Testing Laboratory of the Federal Institute of Piauí (IFPI).

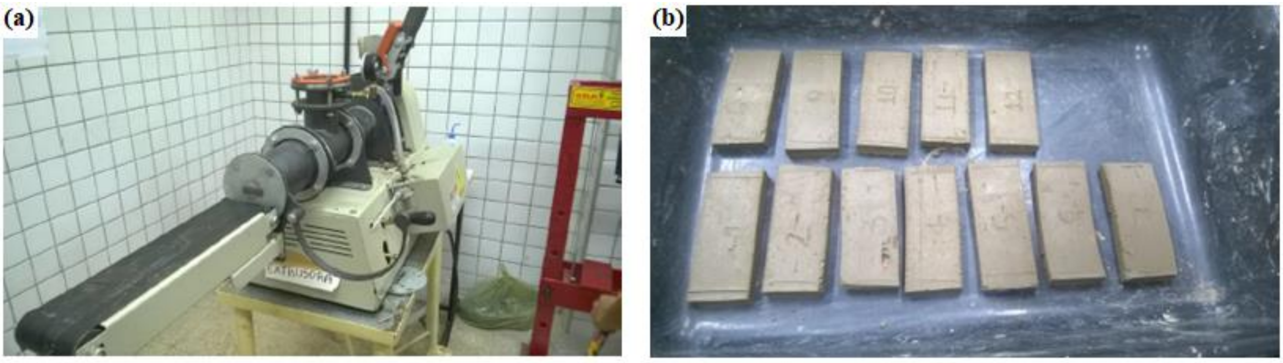

Figure 1. (a) Extruder - Laboratory of Ceramic Testing – IFPI; and (b) Specimens after drying at 110 °C.
Source: Authors, 2019.

2.1. Moisture determination test (%M)

There are three methods of determining moisture: Stove; Infrared; and Speedy or humidifier.

2.1.1. Stove Method

This method consists of coding a clean and dry aluminum capsule using the retro - projector brush. Then, weighing the capsule and recording the weight (in the capsule). After, to weight 100g of wet sample on the scale with 0.1g precision and note the wet mass (Wm). Then, to place the capsule in the oven for 2 hours at 110 ºC; remove the capsule, allowing cooling in the desiccator, and weighing the capsule with dry material. At the end, note the total weight (dry material + capsule weight), deduct the total weight, the capsule weight, obtaining the dry mass (Dm) and calculate the percentage of moisture by the following equation:

$$\%M = [(Wm - Dm)/Dm] \cdot 100 \quad (1)$$

2.2. Test of retained residue in sieve #325 (wet way)

This test aims to separate impurities from clay minerals (clay itself) from the clayey mass. The residue is composed of the mineral impurities present in the clays, which have larger particle size than these.

The procedure for the test is done by: 1) collecting approximately 300g of the crude samples through the quartet; 2) weighing 200g of the sample collected; 3) drying the sample in the oven at 110 °C for 2 hours; 4) after drying, weighing 100g of the dried sample, transferring to the 1L container, and place approximately 600mL of water; and then, 5) add a few drops of ammonium hydroxide; 6) allow the sample to stand for 24 hours to deflocculate it; 7) shake the sample with the stick; 8) fit the sieve into the iron holder in the sink, under the tap with running water; 9) carefully transfer the sample to the sieve, with the aid of the sieve, thoroughly pass the sample in the sieve, using the hairbrush and the fingers to dismantle the clods, when necessary; 10) wash the sample container completely, deflocculating the residue from the sieve into the aluminum dish with the aid of the pestle; 11) drying completely the residue in the oven; 12) weigh the sample dry and write down the weight (capsule + dry residue); and 13) Calculate the percentage of the residue, using the following equation:

$$\%R = (P_{caps}. + Resid._{dry}) - P_{caps}. \quad (2)$$

2.3. Testing body preparation

Three processes are generally used in the preparation of the testing body: extrusion; pressing; and bonding.

2.3.1. Testing body preparation by extrusion

This process is used to predict the use of a clay in the manufacture of red ceramic products such as tiles and blocks. For the preparation, it is recommended to follow steps: 1) to use pre-quarted and dry sample; 2) grinding through hammer mills, gauge or mortar; 3) sieve ± 15 kg of material in #48 (0.297 mm opening); 4) moisten the material by adding 20 to 22% moisture (depending on plasticity); 5) to rest for 24 hours in a plastic or aluminum container (muffled with plastic bag); 6) after this period, pass through the extruder. The vacuum should be about 25 inches of mercury (in/mg) until proper plasticity is achieved.

2.4. Determination of linear retention after drying at temperature T (110 °C)

This test is governed by the MB-305 Brazilian Method (ABNT, 1987). Linear retraction or shrinkage evaluates the shrinkage of the clayey masses when they lose colloidal and residual water during the drying and burning process. By contraction, the granulometric composition of the clay can be estimated, that is, the greater contraction indicates very fine granulometry, which in turn requires more water for the kneading. According to Dondi (2006), the optimal variation is 5% to 8% and the acceptable variation is 3% to 10% (Dondi, 2006).

Linear retraction after drying: Altering the dimensions after drying is a consequence of the elimination of the water used in the conformation of the product, therefore, the higher the amount of water, the greater will be the retraction.

$$\%R1_S = [(Ci - Cf)/Ci] \cdot 100 \quad (3)$$

$\%R1_S$ = Percentage of linear retraction after drying at temperature T; Ci = Initial length of test specimen, measured shortly after forming; Cf = Final length of test specimen, measured after drying at temperature T, which is generally 110 °C.

2.5. Granulometric Distribution

The analysis of grain size distribution aims to determine a grain size curve. For such an embodiment, a sample of granular material is sieved in a standard series of sieves, the aperture of which has a predetermined sequence.

Then the mass of material retained in each sieve is determined and the results are plotted on a graph where the abscissa axis corresponds to the mesh opening on a logarithmic scale and the ordinates to the percentage of the material passing through. This assay is standardized in Brazil by NBR 7181 (ABNT, 2016).

Table 1. Granulometric distribution analysis. Source: Authors, 2019.

Granulometric distribution Test					
Sieve number	**Aperture (mm)**	**Retained material (g)**	**% Retained**	**% Accumulated**	**% Passant**
10	2.00	0.22	1.10	1.10	98.9
30	0.60	0.76	3.80	4.90	95.1
50	0.30	1.28	6.40	11.30	88.7
100	0.15	4.63	23.20	34.50	65.5
Bottom	0.15	13.06	65.50	100	0
	Σ	**19.95**	**100**		

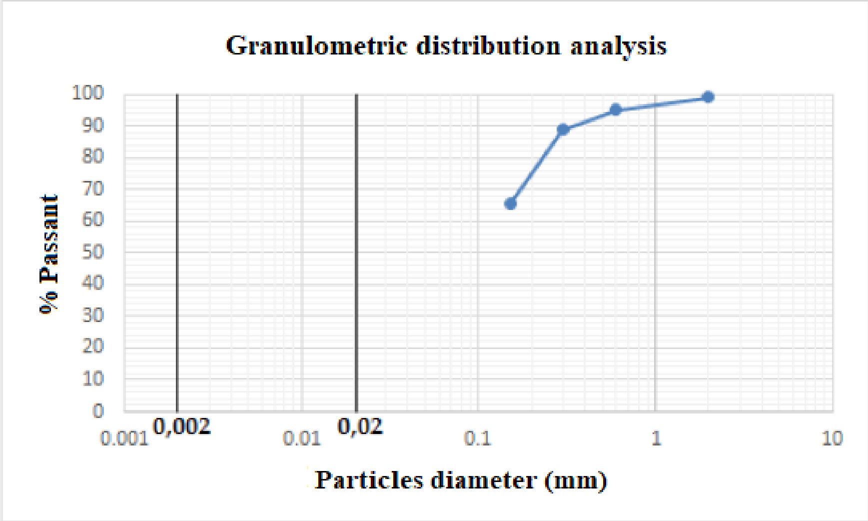

Figure 2. Granulometric distribution analysis.
Source: Authors, 2019.

Table 2. Granulometric composition of red ceramic products. Source: SENAI, 2005.

Regions	**Granulometric composition (%)**		
Produts types	**< 2 µm**	**2 a 20 µm**	**> 20 µm**
A. Materials with quality but with difficulties to produce	40 to 50	20 to 40	20 to 30
B. Roof tiles	30 to 40	20 to 50	20 to 40
C. Drilled bricks	20 to 30	20 to 55	20 to 50
D. Solid bricks	15 to 20	20 to 55	20 to 55

When the soils have very fine granulometry, less than 0.075 mm, this percentage is treated in a differentiated way through the sedimentation test, whose details can be seen in the same NBR 7181 (ABNT, 1987). This operation aims to determine the granulometric distribution of the fines through the fall velocity of the soil particles in a liquid medium, based on Stokes' Law, which correlates the velocity of falling of the spherical particles with their diameter.

For the sample of clay studied, it is intended to use it for the production of tiles, therefore, a value between 20 and 40% in its granulometric composition should be present.

2.6. Determination of bending rupture stress

Mechanical strength is one of the properties of clays, which is of great interest to the ceramist, since it allows the processing of the ceramic pieces without breaking them. In this case, it is one of the routine tests, which is the mechanical resistance to raw and after the burning of the specimens. Bending rupture stress (BRS) is defined as the force per unit area required to break a body, expressed in kilogram-force per square centimeter (kgf /cm^2) or mega-Pascal (MPa). The bending rupture stress occurs when a body is subjected to a bending force causing compression and traction. The mechanical strength of the ceramic masses depends mainly on granulometric distribution, mineralogical composition, and plasticity.

3. RESULTS AND DISCUSSIONS

3.1. Moisture determination test (%)

The test data for the determination of moisture, in percentage, carried out according to the methodology mentioned in sub-item 2.1.1, are shown in Tab. 3.

Table 3. Data for mountain analysis and linear retraction. Source: Authors, 2019.

Sample	**Before Drying**	**After Drying**

	Mu (g)	La (mm)	Ms (g)	Ld (mm)
1	143.64	100	122.08	93.95
2	142.10	100	120.05	94.20
3	135.03	100	115.76	94.97
4	141.37	100	120.26	93.83
5	144.65	100	123.91	95.09
6	139.75	100	118.41	94.63
Average	**141.09**	**100**	**120.08**	**94.45**

According to the data in Tab. 3, and using the formula presented in sub-item 2.1.1, the percentage of moisture in the sample (%M) is 17.53%.

3.2. Test for determination of linear retraction after drying at temperature T (110 °C)

The test data for determination of linear retention after drying at temperature T (110 °C), in percentage, performed according to the methodology quoted in 2.4, are shown in Tab. 3. According to the data in Tab. 3 and using the formula presented in sub-item 2.4, the percentage of linear retraction after drying at temperature T (110 °C) is 6.33%.

3.3. Granulometric distribution test

The results of the test to determine the granulometric distribution, in percentage, follow in Tab. 4.

Table 4. Results for the granulometric distribution test.

Sieve Number	Slot (mm)	Retained material (g)	%
10	2.00	0.22	1.1
30	0.60	0.76	3.8
50	0.30	1.28	6.4
100	0.15	4.63	23.2
Bottom		**13.06**	**65.5**
Total		**19.95**	**100.0**

3.4 Testing of chemical elements in clay

During the verification test of chemical elements in the clay, a yellowish coloration was observed in the specimens, indicating the presence of iron oxide, which according to the literature, can be classified as red ceramics.

3.5. Determination test of bending rupture stress

The results of the test to determine the bending rupture stress performed according to the methodology presented in sub-item 2.5, with test bodies 5 and 6, are shown in Fig. 3 and 4.

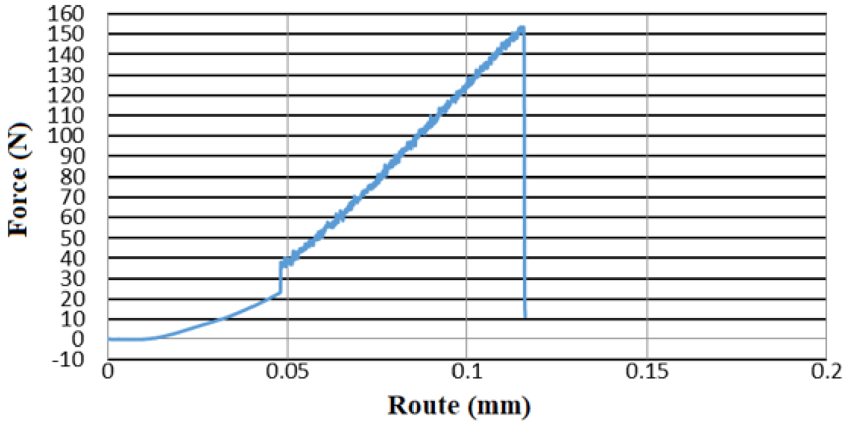

Figure 3. Bending test performed on the test body 5.

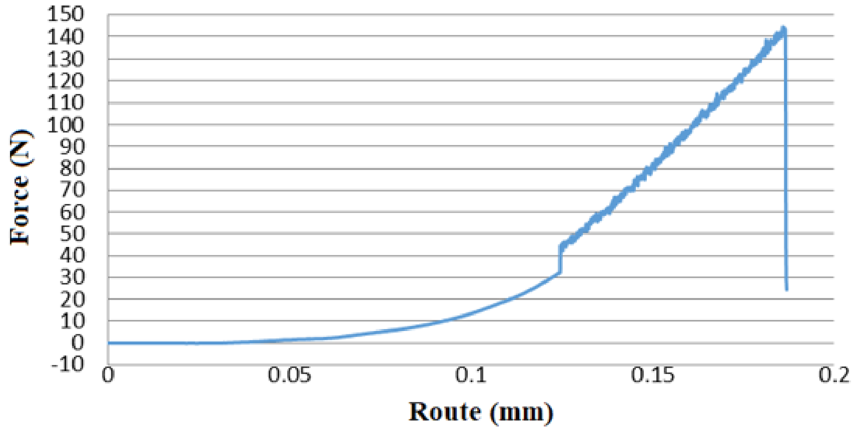

Figure 4. Bending test performed on the test body 6.

According to the results of the test, the maximum tensile strength of the test piece was 144N, whereupon the tensile strength was calculated, the value of which was 2.29 N/mm^2.

4. CONCLUSIONS

The preliminary ceramic tests aim to study the physical and mechanical characteristics of a material to define its application in the industry and predict the individual uses of raw materials from the simple ceramic tests. After the accomplishment of all the technological tests, it can be affirmed through the analysis of the results obtained in the studied samples, that the physical and mechanical characteristics of the collected material present good applicability for the manufacture of red ceramic tiles, for use in the civil construction.

5. ACKNOWLEDGEMENTS

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