Effect of firing temperature and mineral composition on the mechanical properties of silty clays

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ABSTRACT

The physical and mechanical properties of ceramic blocks are affected by the firing process of the primary raw material, in this case, clay or clay minerals. Consequently, this study sought to characterize different types of silty clays for ceramic block production and determine the effect of firing time and mineral content on their mechanical and physical properties. Characterization was conducted in two stages: pre and post-firing. Pre-firing evaluations were granulometric distribution, Attenberg limits, chemical composition, mineral composition and thermal behavior. Post-firing evaluation made use of 30 specimens subjected to temperatures of 800 °C, 900 °C and 1,000 °C. Results showed that firing temperatures greatly affected mechanical properties. Higher burn temperatures increased tensile strength and reduced water absorption while causing linear shrinkage. This increase in shrinkage raised the possibility of damage to the ceramic material and reduction of overall quality.

Keywords: Ceramic; Silty clays; Firing temperature; Mechanical properties.

1. INTRODUCTION

The manufacture of structural ceramic blocks requires a mixture of clays to achieve the required desired characteristics such as compression and tensile strength, linear shrinkage and plasticity. To this end, it is necessary to determine the chemical and physical characteristics of clays [1].

The formation of clay and mineral clays is related to the decomposition and weathering of feldspar and other aluminosilicate minerals in situ or near a geological formation. Variations in the mineral composition of the source rock or in the physical, chemical and biological effects of weathering results in several different types of mineral clays [2, 3]. Clays have a granulometry of less than 4 µm and are made of quartz, feldspar and other minerals. On the other hand, mineral clays such as kaolinite, illite, esmectite or vermiculite are made of aluminum, magnesium or iron hydrated phyllosilicates. These minerals are seldom amorphous and tend to have crystalline lamellate particles in fine grains, also of granulometry of less than 4 µm [4, 5]. Clay minerals combined with clays have important physicochemical characteristics such as plastic behavior when mixed with water and shrinkage when dried at high temperatures [4]. These characteristics are used to ensure that ceramics acquire specified desired characteristics post-firing [3, 6]. For example, the most adequate mineral groups suitable to produce earthenware (also known as "brick ceramic") are illite and montmorillonite [7].

PUREZA [8] reported that final properties of ceramics were related to physical and chemical transformations that occurred during the firing process. Residual water and zeolite water were removed at temperatures of up to 200 °C. Sulfides dissociated between 350 °C and 650 °C, releasing CO_2 and SO_2 and combusting organic matter. Mineral clay lattice broke down between 600 °C and 850 °C, releasing constituent water from within. Carbonate decomposed between 800 °C and 950 °C with CO_2 release. Silica and alumina reacted with other elements between 900 °C and 1,000 °C, forming complex aluminum silicates which lent ceramics their physical and mechanical properties. Finally, above 1,000 °C, aluminum silicates softened and fused, leading to a vitreous phase whose least fused particles conferred hardness, compactness and impermeability to the ceramic.

The firing process of earthenware requires a high amount of energy [9]. Consequently, fuel and the operation of kilns are the costlier factors in the manufacture process with firing time having a direct effect in cost [10]. SALEIRO and HOLANDA [9] evaluated an earthenware quick-firing cycle and concluded that it

was both technically and economically viable. However, the bending strength of the blocks produced with this technique was slightly lower than others from a conventional firing process.

Since clays have varying mineral content, classification and identification is difficult and results in multiple branching nomenclatures such as silty clay, sandy clay etc. [11]. Nonetheless, characterization of clay is a fundamental step in defining the production process for a ceramic block: characterization leads to verification of properties, which leads to definitions on its use in the production of ceramics in accordance with design specifications [12].

Characterization of clays has some limitations: some tests can be performed on the raw material but others are only possible after the production of ceramic elements. Granulometric analysis and Atterberg limits are necessary to physically characterize clays while chemical and mineral characterization are obtained from X-ray diffraction, thermal analysis, optical microscopy and IR spectroscopy [7].

The technique of X-ray diffraction is widely used in the identification of mineral clays. The diffraction profile is characteristic of each crystalline structure and results are obtained faster than other methods [10]. This technique was recommended by MASHEANE *et al.* [1] to determine the mineral composition of clay and evaluate the contribution of each component of the mass. However, for highly pure clays, further techniques such as thermal and chemical analysis should be used to conduct a more precise identification [13].

Thermal analysis evaluates the effects of gain or loss of heat of a material. In clays, this method yields data such as fusion, crystalizing and phase change temperatures. DENARI and CAVALHEIRO [14] pointed out differential thermal analysis (DTA) as a technique that evaluated the difference in temperature between a sample and a reference material heated equally. Thermogravimetric analysis (TGA) determines the mass of a substance as a function of control temperatures. This parameter is used in obtaining data regarding thermal stability, reaction speed and composition of clays. Due to the complexity in analyzing thermogravimetric curves, derivative thermogravimetry (DTG) is used to precisely determine the temperature corresponding to the maximum loss of mass [15].

Considering this scenario, the red ceramic industry occupies a prominent position in the Brazilian civil construction sector. Due to the variability of clays available for the manufacture of ceramic pieces, industries need to carry out previous studies to determine the chemical and physical properties of the raw material. Those tests can estimate the ceramic behavior and if the product will achieve the regulatory requirements. Thus, this study is related to the evaluation of the mineralogical composition of pre- and post-burning clays, comparing the results obtained with those mentioned by other authors.

This study sought to characterize different types of silty clays for ceramic block production and determine the effect of firing time and mineral content on their mechanical and physical properties.

2. MATERIALS AND METHODS

Samples of the silty clays used in this study are shown in Figure 1. These clays are used as raw material in the manufacture of several types of ceramics in the south region of Brazil.

Silty Clay type A1   Silty Clay type A2   Silty Clay type A3

Silty Clay type A4   Silty Clay type A5   Silty Clay type A6

Figure 1: Silty clay samples used in this study.

2.1. Physical analysis

Granulometric distribution and Attenberg limits of the samples were evaluated. Granulometric analysis was conducted through sifting and sedimentation as recommended in standard NBR 7181 [16]. Equipment used for this analysis were a mechanical agitator with sieves of 2.0 mm, 1.2 mm, 0.6 mm, 0.42 mm, 0.25 mm, 0.15 mm and 0.075 mm. Additionally, 6 graduating cylinders, 1 hydrometer, 1 thermometer, 1 electrical homogenizer, 1 pycnometer and a vacuum pump were also used. Attenberg limits were determined according to the procedures of standards NBR 6459 [17] and NBR 7180 [18]. Plasticity measurements made use of a graduated comparison cylinder 3 mm in diameter and 100 mm in length as set by standard NBR 7180 [18] while liquidity was determined with a Casagrande apparatus as required by standard NBR 6459 [17].

2.2. Chemical and mineral analyses

Chemical analysis was conducted through energy-dispersive X-ray spectroscopy (EDX) with a Shimadzu brand, model EDX 720 apparatus. Mineral composition was determined through X-ray diffraction (XRD) with Cu-Kα radiation, Bragg-Brentano HD incident beam geometry, 40 kV tension and 40 mA current. The sweep had a 2θ variation from 5° to 80° with a sweep speed of 0.01°/s and an area detector PIXcel 3D-Medpix3 with 255 channels. This measurement was taken with a Malvern Panalytical Empyrean apparatus. Thermogravimetrical (TGA) and differential thermal (DTA) analyses were conducted to determine the best burn temperature of each sample to produce ceramic blocks. These analyses were conducted in a Perkin Elmer brand, model STA 800 apparatus.

2.3. Specimen production and firing process

Five ceramic blocks measuring $18 \times 27 \times 86$ mm (height × width × length) were produced for each type of clay and firing temperature for a total of 30 specimens. A clay vacuum extruder with gage pressure of 700 mmHg was used to produce the specimens. Specimens were dried in two stages: the first stage consisted of drying at ambient temperature for 72 h while the second stage was carried out in a kiln at 110 ± 5 °C for 24 h [9].

The firing process was conducted in a laboratory kiln with the following combinations of temperature and firing times: 800 °C for 520 min, 900 °C for 570 min and 1,000 °C for 620 min. Heating rate was 2 °C/min and peak temperature was maintained for 2 hours for all burns [19].

Mechanical properties were evaluated in accordance with their respective standards: bending strength with ASTM C1161 [20], linear shrinkage with COLLATO [21] and water absorption with ASTM C373 [22]. Based on the results, water absorption and linear shrinkage were combined in a vitrification curve for each sample to better evaluate their behavior as a function of firing temperature [23].

3. RESULTS AND DISCUSSION

3.1. Physical characterization

Sample identification and granulometric distribution are presented in Table 1 while Table 2 shows properties obtained from the Attenberg limits analysis. Overall, results showed that there was little granulometric variation in between the clay samples used in this study.

3.2. Chemical and mineral characterization

Chemical composition results are presented in Table 3.

Table 3 shows that all samples contain mostly SiO_2, Al_2O_3 and Fe_2O_3. According to GUIMARÃES [24], these oxides are typically found in raw material clays used in earthenware. The presence of SiO_2 decreases plasticity and contributes to shrinkage during firing [25]. However, with respect to mechanical strength, SiO_2

Table 1: Granulometric distribution (%) of clay samples of this study.

SAMPLE	LESS THAN 2 mm	2 mm TO 20 mm	OVER 20 mm
A1	52	21	27
A2	34	36	30
A3	50	21	29
A4	56	13	31
A5	48	18	34
A6	51	20	29

Table 2: Attenberg limits analyses of clay samples of this study.

SAMPLE	LIQUIDITY LIMIT (%)	PLASTICITY LIMIT (%)	PLASTICITY INDEX (%)
A1	66	33	33
A2	44	29	15
A3	48	36	12
A4	66	42	24
A5	67	54	12
A6	60	40	20

Table 3: Clay samples chemical composition.

ELEMENT	A1	A2	A3	A4	A5	A6
Na_2O	1.10	1.03	0.35	0.58	0.1	0.1
MgO	0	0.11	0	0	0	0
Al_2O_3	5.76	2.89	3.43	5.38	6.57	4.61
SiO_2	26.7	27.8	24.69	18.51	20.13	20.92
P_2O_5	0.13	0.19	0.05	0	0.11	0.1
SO_3	1.12	1.10	1.12	1.1	1.12	1.12
K_2O	0.53	1.73	0.82	0.05	0	0.9
CaO	0.02	0.45	0.52	0	0	0.17
TiO_2	0.75	0.57	0.99	0.81	0.92	0.95
MnO	0	0.03	0.18	0.17	0.21	0.25
Fe_2O_3	6.63	4.07	11.23	15.18	12.1	12.51
L.I.	7.5	5.7	9.77	12.2	11.1	9.7
Others	49.8	54.3	46.85	45.95	47.64	48.6

Note: L.I. – loss on ignition.

increases the dilatation coefficient of the ceramic and may lead to a loss in strength in glazed ceramics [26]. The presence of Al_2O_3 may also increase the refractory properties of the resulting ceramic [27].

The reddish-orange tint of earthenware arises from Fe_2O_3 content after firing. The presence of this oxide minimizes shrinkage and assists in drying [25]. The traces of alkaline and alkaline-earth metals (K_2O and Na_2O; CaO and MgO, respectively) contribute to the liquid phase formation that consolidates particles. This results in an increase in linear shrinkage [28]. Also sintering K_2O and Na_2O in temperature ranges of 950 °C to 1,000 °C increases ceramic density and provides an increase in mechanical strength [24]. The diffractogram of clay samples A1 and A2 are shown in Figure 2 while samples A3 through A6 are shown in Figure 3.

Figure 2 shows that samples A1 and A2 reach high peaks of approximately 16,000 cts, which characterizes crystalline materials with little trace of mineral clays. On the other hand, Figure 3 shows that samples A3 through A6 have a considerable reduction in detector count number, peaking only at 3,600 cts. This lower count number indicates a larger presence of mineral clays. Samples A1, A4, A5 and A6 contain kaolinite, which is mostly used in porcelains (also known as "white ceramics") which are fired at temperatures higher than 1,100 ºC. Samples A2 and A3 contain montmorillonite and are more suitable for ceramic blocks.

Figure 4 shows thermogravimetric (TGA) and derivative thermogravimetry (DTG) curves of each sample. Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) allow the assessment of change in mass in a sample as a function of temperature difference, which allows the optimization of the firing process of ceramics.

As noted by DEER *et al.* [4], quartz, feldspars (albite and sanidine) and hematite allowed little or no hydration in their structure and lattice spaces. This resulted in insignificant variations in mass due to elimination of absorbed water or dehydration in the range of temperatures of this study. Consequently, DTG peaks became
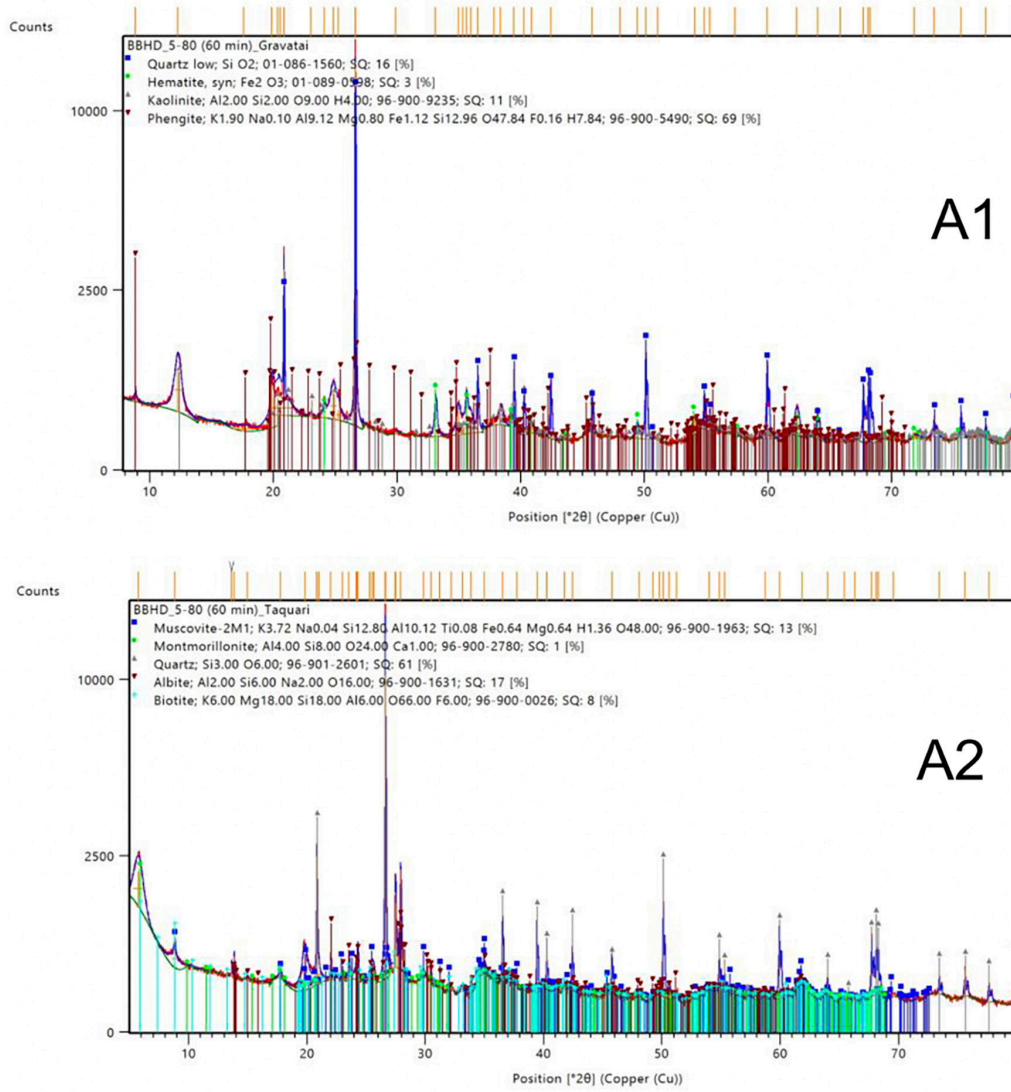
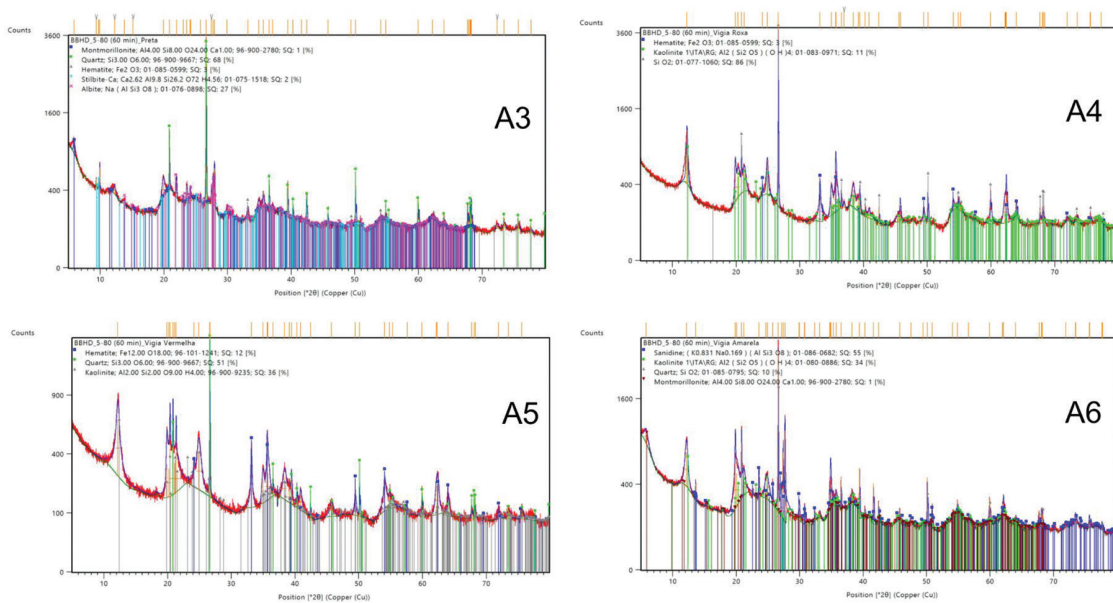**Figure 2:** Diffractograms of clay samples A1 and A2 in powder form.

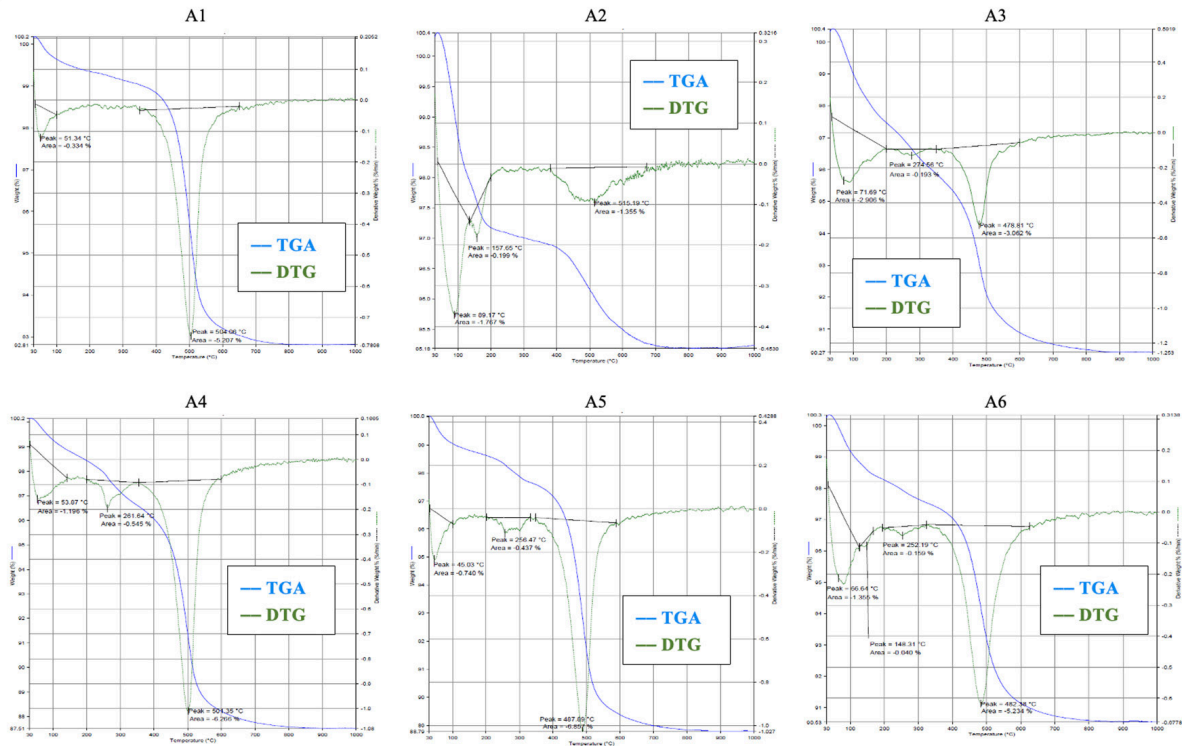**Figure 3:** Diffractograms of clay samples A3 through A6 in powder form.

Figure 4: DTG and TGA curves for all samples of this study.

more discreet and harder to identify. Mica minerals (muscovite, phengite and biotite) and stilbite allowed some water in the empty spaces of crystalline structures. Thus, during calcination which occurred within the temperature range of this study, changes in mass could occur. Taken together, both mineral contents formed the fusion phase in which they fused and later solidified to create internal bonds between mineral clay grains.

In comparison, kaolinite and montmorillonite were hydrated silicate clay minerals which lost absorbed and constituent water when heated. This resulted in shrinkages, mass loss, reduction in porosity and increases in mechanical strength [24]. Consequently, the firing process must be adjusted to achieve total water removal from the material.

Based on TGA and DTG data, two main intervals were identified in the DTG curve. The first interval occurred between 30 °C and 350 °C and was related to the removal of colloidal water and organic matter, with a corresponding lower percentage of mass loss. The second interval occurred between 400 °C and 650 °C and accounted for the removal of structural water and a corresponding larger mass loss. This was an expected result since clays, especially montmorillonite were hydrated minerals. The samples used in this study were found to partially match expected mineralogical temperature ranges with discrepancies attributed to intrinsic variations in content of natural materials and other phases not identified through XRD.

The TDA of Figure 4 demonstrated that samples A1, A4, A5 and A6 had the same thermal behavior with two similar and well-defined temperature intervals. Sample A2 had a greater loss of mass in the first interval when compared to the other samples. This was attributed to less efficient drying or greater presence of organic matter. In the second interval, sample A2 had a less defined curve with a less inclined TGA, which could be related to a lower relative proportion of mineral clays. As a special note, sample A3 had a thermal behavior that diverged from the other samples with respect to loss of mass. This divergence occurred in similar fashion in both intervals of the DTG and could be an indication that this sample had a more balance proportion of accessory minerals and mineral clays.

Overall, complete dehydration of mineral clays and fusing of accessory minerals was known to result in an effective increase in mechanical strength, depending on whether the material was subjected to a quick or slow firing process. In this case, the granulometry distribution between 2 µm to 20 µm in the samples of this study was quite wide and negatively affected the firing process as large grains might not have fully crystalized and resulted in an unstable nucleus inside a shell.

3.3. Mechanical characteristics (flexural bending strength)

Flexural bending strength tests were conducted to determine sample strengths with respect to each firing temperature. Average (ft,med) and maximum (ft,max) strengths results are shown in Table 4. Table 4 shows that sample A3 has the highest ftmax for firing temperatures of 800 °C and 1,000°C.

Figure 5 displays a surface plot combining the clay samples, firing temperature and flexural bending strength.

The surface plot of Figure 5 indicates that samples A1, A2, A5 and A6 have strengths below 5 MPa, which are below the recommended value of 5.5 MPa for adequate ceramic blocks [7]. Sample A3 surpassed this value for a temperature of 900 ºC and reflected the behavior identified in the DTA of Figure 4. Additionally, sample A3 also presented 3 endothermic regions: the first between 30 °C and 140 °C with a loss of mass of 2.42%; the second between 140 °C and 200 °C with a loss of mass of 0.85% and the third between 380 °C and 670 °C with a loss of mass of 1.89%. The losses of mass in the first and second region were related to residual water while the third region was due to the presence of organic matter and release of constituent water in the mineral clay [8]. The total loss of mass in the TGA curve was of 5.16%.

The mechanical performance of sample A3 could be related to its montmorillonite content identified through XRD as shown in Table 4. Montmorillonite is a type of silty clay adequate for ceramic blocks and consists of smaller particles than usually found in mineral clays [29]. The particles are shaped as plaques or thin plates with irregular profiles which tend to coalesce during drying and have a good potential of delamination when placed in contact with water [30]. While Sample A3 had suitable concentrations of montmorillonite, Sample 2 had low concentrations which conferred low plasticity to the ceramics produced with it.

Table 4: Flexural bending strength of the samples used in this study with respect to firing temperatures.

SAMPLE	FLEXURAL BENDING STRENGTH (MPa)							
	$F_{T,MED}$				$F_{T,MAX}$			
	23 ºC	800 °C	900 °C	1,000 °C	23 ºC	800 °C	900 °C	1,000 °C
A1	2.21 ± 0.12	2.30 ± 0.22	3.83 ± 0.32	4.46 ± 0.38	2.38	2.65	4.45	4.98
A2	1.94 ± 0.40	2.57 ± 0.15	3.77 ± 0.27	4.73 ± 0.22	2.62	2.72	4.39	5.02
A3	4.10 ± 0.34	4.72 ± 0.30	6.4 ± 0.49	7.72 ± 0.87	4.95	5.15	7.50	9.31
A4	1.28 ± 0.11	2.67 ± 0.47	3.33 ± 0.51	4.64 ± 0.35	1.56	3.85	4.08	5.42
A5	2.39 ± 0.05	2.87 ± 0.23	3.68 ± 0.19	4.06 ± 0.23	2.46	3.21	3.94	4.54
A6	2.07 ± 0.01	2.39 ± 0.23	2.75 ± 0.19	3.18 ± 0.17	2.09	2.62	3.12	3.35
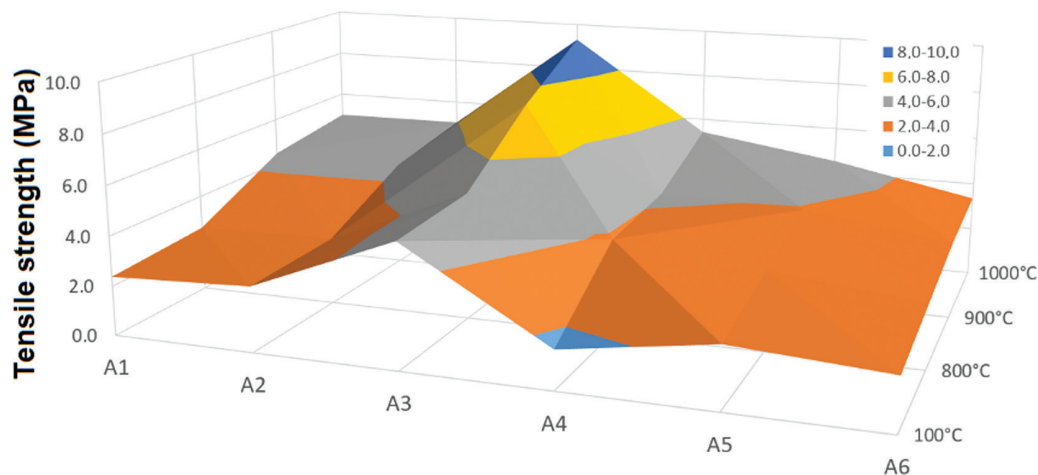
Figure 5: Flexural bending strength plot of clay samples of this study with respect to temperature.
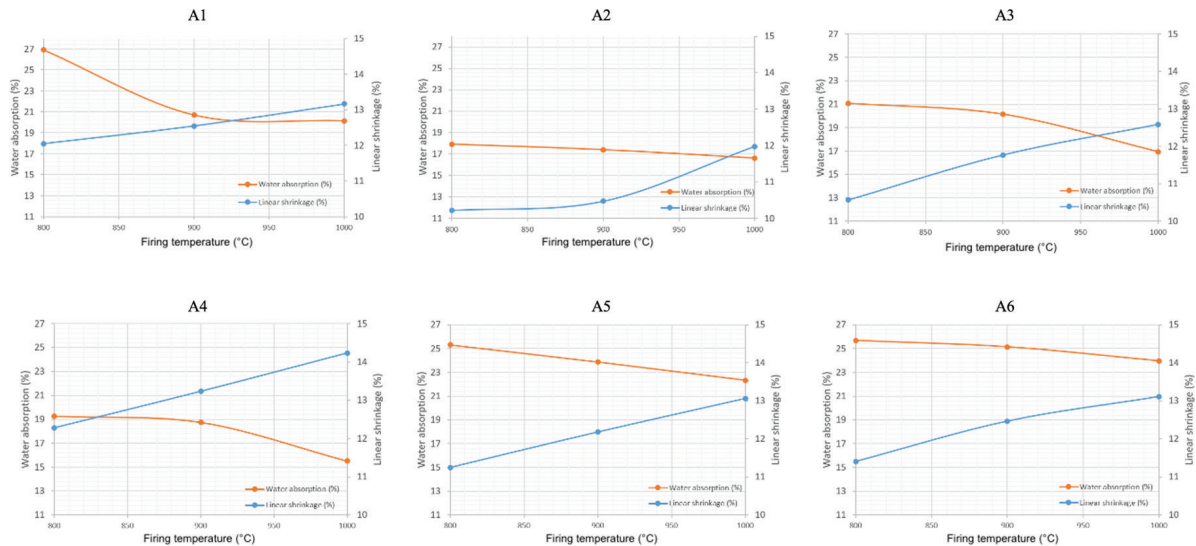
Figure 6: Vitrification curves of clay samples used in this study.

3.4. Vitrification of samples

Samples A1, A3, A5 and A6 presented water absorption equal or higher than 21% at 800 °C, which was the value recommended by standard NBR 15270-1 [31]. On the other hand, this limit was not reached by samples A2 and A4 at any of the temperatures tested. Within each sample, the variation of linear shrinkage was between 1.0% and 2.1% for the temperature range between 800 ºC and 1,000 ºC. Individually, sample A2 presented the largest variation of shrinkage from 10.2% at 800 ºC to 12.3% at 1,000 ºC. Conversely, sample A1 had the smallest variation of shrinkage from 12.1% at 800 ºC to 13.1% at 1,000 ºC. In terms of total shrinkage, all mineral clays exceeded 12% at 1,000 ºC which, according to KOHL *et al.* [32], increased the potential of fissures and deformations to the point of compromising the quality of the ceramic.

Figure 6 also demonstrates that shrinkage increases with increasing firing temperature. This was a result of sintering, which caused a compaction of clay molecules and decreased pore size and volume of material. Sintering required high temperatures to induce sufficient atomic molecular mobility [33]. Samples A1, A4, A5 and A6 had linear shrinkages higher than 12.5% and had similar TGA curves in Figure 4. This grouping of samples had a distinct behavior when compared to samples A2 and A3 which had less linear shrinkage. These behaviors were due to the mineral content detected through XRD. Under sintering, the difference in content formed different compounds and different structures with distinct responses [34].

4. CONCLUSION

The following conclusions were the drawn from the results of this study:

- Considering the firing level of 900 °C, it was verified that clays A1 and A3 presented the highest mechanical strength values for compression and flexion. The water absorption and linear shrinkage values were very close to the limit. Only A1 clay surpassed the linear shrinkage limit recommended in the literature, reinforcing the importance of moderately adding non-plastic material to the ceramic mass.

- As for the Atterberg Limits, all samples have a Plasticity Limit greater than 25%. The granulometry confirms that all samples, except sample A6, have a high clay fraction index, giving plasticity to the clayey mass and increasing mechanical resistance. However, it increases water absorption and linear shrinkage.

- Chemical and mineralogical characterizations showed that the samples have typical compositions of red raw materials. All clays had silica (SiO_2), alumina (Al_2O_3), and iron oxide (F_2O_3).

- From the performance of post-fire tests, it was observed that the increase in temperature decreases water absorption, increases mechanical strength and linear shrinkage of ceramics. With this, it was possible to define the ideal firing level to produce ceramic pieces.

- The silty clays analyzed had different chemical and physical properties which affected the mechanical and physical performance of the resulting ceramics. Ceramics, in general, had better performance characteristics when fired at higher temperatures.

5. ACKNOWLEDGMENTS

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