Thermal behavior evaluation of clays from algodonal formation and their impact on technological properties

G Guerrero-Gómez¹, N J Escobar-Mora² and C Nolasco-Serna³

¹ Grupo de Investigación en Tecnología y Desarrollo en Ingeniería, Universidad Francisco de Paula Santander seccional Ocaña, Colombia
² Grupo de Investigación en Bioingeniería, Universidad Pontificia Bolivariana, Medellín, Colombia
³ Grupo de Investigación de la Facultad de Educación, Artes y Humanidades, Universidad Francisco de Paula Santander seccional Ocaña, Colombia

E-mail: gguerrerog@ufpso.edu.co, Nelson.escobar@upb.edu.co

Abstract. It is presents the characterization of the clays from the algodonal formation and the mechanical properties of the products manufactured in brickyard “Hora”, located in Ocaña, Norte de Santander, Colombia. Tests of scanning electron microscopy, thermal gravimetric analysis, differential scanning calorimetry, initial rate of absorption, 24-hour immersion, compressive strength, and modulus of rupture were performed. The results indicate that the clay contains a considerable amount of silico-aluminate, while the products have low mechanical strength and high percentage of water absorption. The clays used present difficulty in the process of shaping and compacting the products. None of the samples comply with the values established in the standard and do not allow their use as non-structural masonry products for interior or exterior use.

1. Introduction

Clays are minerals found in nature generally accompanied by organic matter [1,2] and are composed of silicoaluminates and minerals such as feldspars, quartz, carbonates, among others [3,4]. They are also used in the construction of civil works [5,6], for their low costs and thermal insulation [7], and acquire during the firing properties and desired characteristics set by the standards.

An important ceramic industry has developed in Ocaña, Colombia due to the good quality of its clay formations, but empirical from a technological point of view. Nowadays, most of the brickworks carry out a completely handcrafted work. In spite of that, it generates a significant number of direct jobs; becoming one of the main development alternatives for the region. For this reason, it is of great importance the technification in its production processes, improving the quality of the products offered in order to reach the competitiveness to remain in the current market, complying with the regulations of the government agencies regarding the emission of particulate matter into the environment.

Up to now, there are few thermal and mineralogical studies of the clays of the region. Descriptive studies of the production process of ceramic materials and mineralogical studies have been carried out [8].

Norte de Santander internal agenda for productivity and competitiveness proposes that by 2020, the ceramic industry should be the national and bi-national leader in the field of high-quality ceramic products [9]. To achieve this goal, the ceramic industry in the region must be technified. Also, thermal
studies of raw materials must be carried out in order to know the transformations they undergo during firing process and the properties of the corresponding raw and fired products. This research allows knowing the thermal behavior of the clays from Algodonal formation and the mechanical properties of the products manufactured in brickyard “Hora”, located in Ocaña, Colombia, following the guidelines methods for sampling and testing of masonry units and other clay products [10] and the compliance with the properties established in the standard for masonry units of fired clay, bricks, and ceramic blocks [11]. This serves for future research in order to optimize the manufacturing processes of ceramic materials and promote the proper exploitation of it.

2. Methodology
The samples selected are the result of the preparation of the plastic-texture clay mixture [12], from the Algodonal formation and fired bricks in the brickyard “El Estanco”, located in Ocaña, Colombia.

The clay sample was dried, crushed, ground, and sieved to a particle size of less than 2 mm [2].

In the evaluation of the mechanical properties of the products, 5 samples were selected in each test, for a total of 20 brick samples according to the stipulated in the methodology and selection and sample preparation processes established in the Colombian technical standard [11], to which standardized non-destructive quality control tests such as initial absorption rate and 24-hour water immersion were performed, measuring the amount of water absorbed by the brick in 1 minute and for 24 hours respectively, and destructive tests such as compressive strength and modulus of rupture or bending in accordance with the aforementioned standard.

2.1. Tests
To determine the chemical components of the clay, a scanning electron microscopy was carried out, where the sample to be analyzed was coated with gold in a Denton Vacuun Desk V equipment, then images were taken in the Sem Jeol JCM 6000 Plus equipment under high vacuum conditions, in which secondary electrons and 15 kv were used, during the test a scattering energy spectroscopy was carried out.

For thermal characterization, thermal gravimetric analysis and differential scanning calorimetry tests were performed on a TGA SDTA851 Mettler Toledo and TA instruments SDT Q600, respectively, using inert nitrogen gas with a flow rate of 40 ml/min and a heating speed of 10°C/min until reaching 1200°C [13].

While for the mechanical properties of the products, standardized non-destructive and destructive quality control tests were carried out on the masonry blocks established in the Colombian [10], methods for sampling and testing of masonry units and other clay products.

For the initial absorption rate and 24 hour water immersion tests, the samples were dried in a muffle furnace for 24 hours at 110°C, then placed in a cooling chamber for 4 hours at 24°C± 8°C and moisture between 30% and 70%; The dry mass of the samples was then determined, then they were immersed in clean water for 1 minute in the initial absorption rate test and 24 hours in the immersion test for 24 hours at a temperature between 15°C and 30°C, finally, the final mass of the sample was determined after the test Figure 1 (a).

For the compressive strength and modulus of rupture or bending tests, the samples were dried in a muffle furnace for 24 hours at a temperature of 110°C, then placed in a cooling chamber for 4 hours at a temperature of 24°C± 8°C and humidity between 30% and 70%; Subsequently, a 3 mm thick layer of gypsum was applied to the opposite sides of the load. Finally, the samples were taken to a universal testing machine with a capacity of 1000 kN, until the sample was taken to the failure [14,5], recording the breaking load and determining the maximum compressive stress [15] and the bending modulus. The sample in the rupture test is placed between two supports and subjected to a point load at the center of the top face of the sample by means of a steel plate [16], see Figure 1 (b).
3. Results

Figure 2 shows the scanning electron microscopy at different magnifications, where the homogeneity in the size of the aluminosilicate layers and their laminar morphology is observed.

Figure 3 and Table 1 shows the chemical composition of the clay resulting from the analysis of energy-dispersive X-ray spectroscopy differential scanning calorimetry derived from the scanning electron microscopy, in which it can be determined that the sample is made up mainly of silicon in 27.96%, aluminum in 13.81% and the rest is oxygen in 58.22% from the environment, forming considerable amounts of silicoaluminates [17], and may be associated with the presence of kaolin.
Table 1. Chemical composition of the clay

| Element | (keV) | Atom% | Mass% | Sigma |
|---------|-------|-------|-------|-------|
| O K     | 70.71 | 0.53  | 58.22 | 1.82  |
| Al K    | 9.95  | 1.49  | 13.81 | 0.71  |
| Si K    | 1.74  |       | 27.96 | 1.07  |

Figures 4 and Figure 5 show the thermal gravimetric analysis and differential scanning calorimetry curves in which it is observed that, between 0°C and 75°C, there is an endothermic peak related to the elimination of hygroscopic water. At 280°C, there is a second exothermic peak related to the decomposition of organic matter, and between 280°C and 900°C, there is an endothermic reaction related to the loss of water from crystallization or dihydroxylation of the clay [17]. Finally, between 900°C and 1,200°C, there is an exothermic reaction related to the formation of the crystalline phase known as mullite.

Figure 4. Thermal gravimetric analysis.

Figure 5. Differential scanning calorimetry.

The result of the initial absorption rate and 24 hour water immersion tests can be observed in Figure 6. The initial water absorption rate ranges from 0.15 g/cm²/min to 0.44 g/cm²/min with an average of 0.30 g/cm²/min; while the 24 hours immersion varies from 12.86% to 34.04%, with an average of 21.30%.

The result of the compressive strength and modulus of rupture tests can be observed in Figure 7. Compressive strength ranges from 2.82 MPa to 4.25 MPa, with an average of 3.76 MPa; while the modulus of rupture fluctuates between 0.59 MPa and 4.50 MPa, with an average of 1.98 MPa.
4. Conclusions
In the thermal characterization carried out on the clay, it was observed that, in order to reduce the contractions or expansions in the products, the material must enter completely dry to the firing process, in addition to establishing the ideal temperature and time curve, leading to the optimization of the firing process.

The Colombian technical standard states that for structural and non-structural masonry products the value at the initial water absorption rate is 0.25 g/cm²/min. But in the selected samples the average value is 30 g/cm²/min. Therefore, the products manufactured in the brickyard “el Estanco” do not comply with what is established in the standard; which generates the presence of superficial pores allowing the brick to absorb a great amount of water from the environment, affecting the adherence and consistency of the mortar.

The average value in the 24-hours immersion test is 21.30% and is above the maximum water absorption value according to standard, which must be 17.50%, which is why this type of brick cannot be used for interior and exterior use.

The average value of the compressive strength in solid fired clay brick samples is 3.76 MPa lower than that established in the 14 MPa standard and its restriction to use only 80% of this strength, that is to say 11.2 MPa, which indicates the high degree of vulnerability to compression in solid brick walls used mainly in housing. Therefore, it does not comply with the mechanical strength values established in the standard. The average value in the modulus of rupture in samples of solid brick is 1.98 MPa, being above the range of values of 0.29 MPa and 0.884 MPa established in the standard, therefore, it complies with the values established in the standard.

References
[1] Díaz Rodríguez L A and Torrecillas R 2002 Arcillas cerámicas: una revisión de sus distintos tipos,
significados y aplicaciones Boletín de la Sociedad Española de Cerámica y Vidrio 41 459–470

[2] Santos Amado J D, Malagón Villafrades P Y and Córdoba Tuta E M 2011 Caracterización de arcillas y preparación de pastas cerámicas para la fabricación de tejas y ladrillos en la región de Barichara, Santander Dyna 78 53–61

[3] Muñoz Meneses R A, Muñoz Chaves J A, Mancilla P and Rodríguez-Páez J E 2007 Caracterización fisicoquímica de arcillas del municipio de Guapi – costa pacífica caucana (Colombia) Revista de la Academia Colombiana de Ciencias Exactas, Físicas y Naturales 31 547–544

[4] Gelvés Díaz J F, Sánchez Molina J and Peña Rodríguez G 2009 Comportamiento de las arcillas del área metropolitana de Cúcuta sometidas a proceso de moldeo por extrusión Revista Resuestas 14 32–38

[5] Rozo S, Sánchez J y Alvarez D 2014 Propiedades físico mecánicas de bloques H10 fabricados en el área metropolitana de Cúcuta Ciencia e Ingeniería Neogranadina 24 67-78

[6] Asociación Colombiana de Ingeniería símica (AIS) 2010 Reglamento colombiano de construcción sismo resistente, NSR-10 (Colombia: Asociación Colombiana de Ingeniería símica)

[7] Aranguren A, Sancho J M, Sanz B and Planas J 2011 Método de caracterización de las propiedades mecánicas de la fractura del ladrillo Anales de Mecánica de la Fractura 2 725–730

[8] Afanador García N, Guerrero Gómez G and Monroy Sepulveda R 2012 Propiedades físicas mecánicas en ladrillos macizos cerámicos para mampostería Ciencia e Ingeniería Neogranadina 22 43-58

[9] DNP-Agenda Interna 2007 Agenda Interna para la Productividad y la Competitividad: Norte de Santander (Colombia: Departamento Nacional de Planeación)

[10] Instituto Colombiano De Normas Técnicas (NTC) 2005 Métodos para muestreo y ensayos de unidades de mampostería y otros productos de arcilla, Norma Técnica Colombiana, NTC4017 (Colombia: Instituto Colombiano De Normas Técnicas)

[11] Instituto Colombiano De Normas Técnicas (NTC) 2000 Unidades de mampostería de arcilla cocida, ladrillos y bloques cerámicos, Norma Técnica Colombiana, NTC4205 (Colombia: Instituto Colombiano De Normas Técnicas)

[12] Cely Illera L 2014 Comportamiento térmico y mecánico de una arcilla de la región de Norte de Santander (Colombia: Universidad Francisco de Paula Santander)

[13] Fernández Abajo M 2000 Manual sobre fabricación de baldosas, tejas y ladrillos (Barcelona: Beralmar)

[14] Cuellar E, Portillo J, Renderos M and Vides F 2006 Evaluación de la resistencia a la fractura de los ladrillos de barro fabricados por compresión (Universidad Centroamericana José Simeón Cañas)

[15] Kaushik H B, Rai D C and Jain S K 2007 Stress-Strain Characteristics of Clay Brick Masonry under Uniaxial Compression Journal of Materials in Civil Engineering 19 728–739

[16] Takeuchi C 2007 Comportamiento en la mampostería estructural (Bogotá D.C.: Universidad Nacional de Colombia)

[17] Mahmoudi S, Bennour A, Megueblí A, Srarsra E and Zargouni F 2016 Characterization and traditional ceramic application of clays from the Douiret region in South Tunisia Applied Clay Science 127 78–87