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Caracterización Físico-Química De Un Material Arcilloso Proveniente De La Región Nororiental De Colombia, Para La Fabricación De Bloques H-10

Physical-Chemical Characterization Of A Clay Material Coming From The Northeast Region Of Colombia, For The Manufacture Of H-10 Blocks

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ABSTRACT

Keywords:

Blocks H-10; hydrometry; mixed; ceramics; brick; DRX.

In this work, the physical-chemical characterization of clay used for the manufacture of H-10 ceramic blocks in the municipality of Ocaña, Norte de Santander, Colombia is presented. The collection of samples was carried out from the quarry of a company dedicated to the manufacture of masonry products for construction. Initially, in the physical characterization a stratified sampling was made according to the texture and appearance of the clay, to identify the presence of carbonates; In addition, the wet granulometry test was carried out for each sample in order to determine the percentages of sand, silt, and clays. The chemical characterization was carried out by X-ray diffraction (DRX) to the samples mixed with the procedure used by the company, to determine the phases formed in the material and the degree of Mosh hardness, in order to buy the values with respect to the final product manufactured by the company. Finally, the results of the final properties of the blocks are analyzed with respect to the Colombian NTC 4205 technical standard procedure, against the physical-chemical characteristics of the clay and the properties of the mixture used in the process, to propose strategies and recommendations on the selection of the raw material that allows blocks with greater mechanical properties and less production defects, thus complying with the requirements established by Colombian technical standards.

RESUMEN

Palabras clave:

Bloques H-10; hidrometría; mezclado; cerámica; ladrillera; DRX.

En este trabajo, se presenta la caracterización físico-química de una arcilla usada para la fabricación de bloques cerámicos H-10 en el municipio de Ocaña, Norte de Santander, Colombia. La recolección de muestras se realizó desde la cantera de una empresa dedicada a la fabricación de productos de mampostería para la construcción. Inicialmente, en la caracterización física se realizó un muestreo estratificado de acuerdo a la textura y apariencia de la arcilla, para identificar la presencia de carbonatos; además, se llevó a cabo el ensayo de granulometría por vía húmeda para cada muestra con la finalidad de determinar los porcentajes de arenas, limos y arcillas. La caracterización química se llevó a cabo por difracción de rayos X (DRX) a las muestres mezcladas con el procedimiento utilizado por la empresa, para determinar las fases formadas en el material y el grado de dureza Mosh, con la finalidad de comprar los valores respecto al producto final fabricado por la empresa. Finalmente se analizan los resultados de las propiedades finales de los bloques respecto a la norma técnica Colombia NTC 4205, frente a las características físico químicas de la arcilla y a las propiedades de la mezcla utilizada en el proceso, para proponer estrategias y recomendaciones sobre la selección de la materia prima que permita bloques con mayores propiedades mecánicas y menos defectos por producción, cumplimento de esta forma con los requisitos establecidos por normas técnicas colombianas.

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Introduccion

The ceramic process is mainly composed of three phases, which are; preparation of ceramic paste, molding of the piece and cooking. In the first phase of the preparation process, the composition and plasticity are modified by adding other clays in order to obtain a homogeneous ceramic paste, then molded according to the desired shape under pressure or extrusion. Once, the molded part has been obtained, it is dried by controlling the evaporation rate of water in order to avoid defects in the piece. Subsequently, the piece is sintered; that is, it undergoes the cooking process in order to decrease porosity, increase density and mechanical resistance [1][2][3].

According to production capacity and technological development, brick industries have been classified as chircal, small, medium and large bricks [4]. In general, these industries maintain the production process as can be seen in Figure 1.

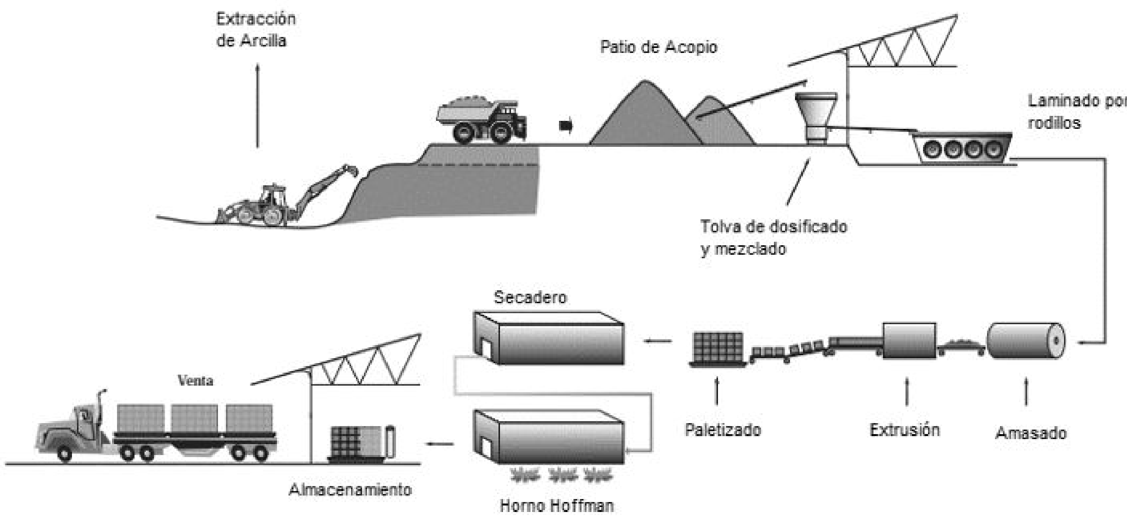


Figure 1. Block manufacturing scheme [5][6].

Generally, three components are used that play important roles in optimizing the performance of the final properties of the blocks and ceramic materials. The first component is clay, so its plasticity facilitates the structure of the product, while the second is feldspar or alumina (Al2O3) which is used for flux and the third is silica (SiO2) that is used as a filler and stabilizer [7][8]. These compositions are determined by chemical composition, which is the basis of modern classification of minerals and the approximation of mixtures with ternary diagrams [9][10], that in the case of many factories producing blocks or bricks do not take into account the proportions of each of these chemical components.

The physical-chemical characterization is carried out in order to observe the behavior of the material in the drying stage, mainly determining the contraction rate of the sample at the different selected temperatures and relating the losses due to drying with the dry shrinkage percentage of the test tubes formed in the laboratory [11][12][13][14]. In order to determine the firing behavior of clays, it is necessary to apply the chemical characterization techniques by X-ray Diffraction to certain specific conditions to determine the phases present in the system [15][16][17][18].

It is for the above, that the present investigation is based on the analysis of a clay mixture obtained from different clay samples by performing an experimental procedure in order to improve the characteristics of the final product through different laboratory tests with the that the behavior of the raw material (clay) can be predicted and thus optimize the company's resources.

Methodology

For the development of this research, a channel-type sampling was performed to classify the clays of the quarry, with an experimental type of investigation; taking into account that the clay samples were collected directly in the mines of a brick by direct observation. Likewise, from the results of the analyzes that were obtained from the samples, the variables to be studied for the approach and implementation of the experimental studies were determined. In addition, laboratory tests were carried out in order to determine the variables that were studied, to identify those that have the greatest impact and thus design the optimal mixture in the manufacture of H-10 blocks [19][20]. Taking into account the above, the procedure shown in the following figure 2 was performed:

Figure 2. Methodology used.
Source: Authors

After the collection of the samples, these were prepared according to the procedures established by the NTC 4017 standard to perform the physical tests for the determination of the presence of carbonates and granulometry by wet route; in addition to the X-ray diffraction test (DRX) to determine the phases present and their corresponding degree of Mosh hardness. With the results obtained, the contraction curve of the block during burning and the cooking curves of the samples at different temperatures were obtained, as well as the final mechanical properties of the blocks such as the compressive strength and the modulus of flexion or breakage determined according to the technical standard Colombia NTC 4205.

The stratified sampling of each of the existing layers in the quarry were classified by color and granulometry, as shown in Table 1. Likewise, cross-sections of the different mantles (channel type) were made, taking care to clean the material that is exposed to the weather and taking fresh material (without weathering) for physical-chemical characterization.

SAMPLE	DESCRIPTION	OBSERVATIONS
1	Waste of production	-
2	White	A mantle thickness of 25 m
3	White sandy	A mantle thickness of 5 m
4	Green sandy	A mantle thickness of 5 m
4A	Green clay with black pints	A mantle thickness of 5 m
4B	Green clay with iron oxides	A mantle thickness of 5 m
4C	Green clay under the tanks	A mantle thickness of 6 m
5	Sandy White Clay	A mantle thickness of 2.2 m
6	White Clay	A mantle thickness of 5.45 m
7	Red clay	-
8	Green clay	-

Source: Authors

Table I. Nomenclature of the Samples.

Results And Discussion

For the development of the physicochemical characterization each of the samples was dried at a temperature of 110 °C for 10 hours. Once dried, the quartet and homogenization were carried out, dividing each sample to perform the different tests. The grinding of the pulp was carried out approaching in particle size to the current pulp of production.

HCL test to identify carbonates

This test was carried out by chemical attack with dilute hydrochloric acid HCl, to determine if carbonates are present within the chemical composition of the clay. When the clay contains carbonates, better grinding is required to avoid problems in the finished product such as caliches and efflorescence. In addition, if carbonates are in large percentages, it is necessary to adjust the cooking curve to ensure their total combustion.

The test of attack with diluted hydrochloric acid HCl for the samples tested, was negative for all samples, being negative discarded the presence of carbonates within the mineralogical composition of the materials.

Granulometry by wet way.

The determination of the percentages of sands, silts and clays, was carried out by wet cutting the dry samples on three meshes, the 120 mesh (125 µm) that retains the fractions of very thick sand, coarse sand, medium sand and fine sand; 230 mesh (63µm) that retains very fine sand and 325 mesh (45µm) in which there are superfine sands and some nodules of certain minerals. Table 2 shows the results obtained.

SAMPLE	RESIDUE			
	M 120 (%)	M 230 (%)	M 325 (%)	Pass (%)
Green 1	12.11	4.35	2.10	81.42
Green 1A	23.67	6.65	1.81	67.86
Green 1B	23.77	8.50	2.30	65.43
Green 1C	31.56	7.55	190	58.95
Yellow 1	12.21	5.95	2.25	79.58
Yellow 1A	27.70	8.38	2.56	61.36
Yellow 1B	33.62	9.68	2.99	53.75
White	46.87	14.54	3.35	35.24

Source: Authors

Table II. Results of granulometries.

The Granulometry data shows that green clays can be classified into two groups:

• **Green 1:** Sample with lower percentages of sands retained on meshes 120, 230 and 325 and higher percentage of through material on mesh 325, which should behave differently in drying and cooking.
• **Green 1A, Green 1B and Green 1C:** Raw materials that can be grouped by their similar particle sizes.

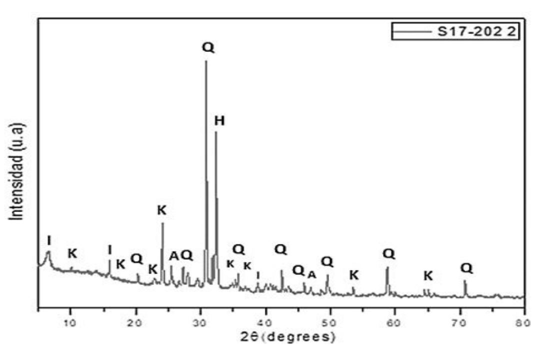
Also, the granulometry of the yellow clays are divided into two groups:

• **Yellow 1:** Sample with good plasticity and average granulometry values on the different meshes.
• **Yellow 1A y Amarilla 1B:** Medium to high particle size and lower plasticity materials.

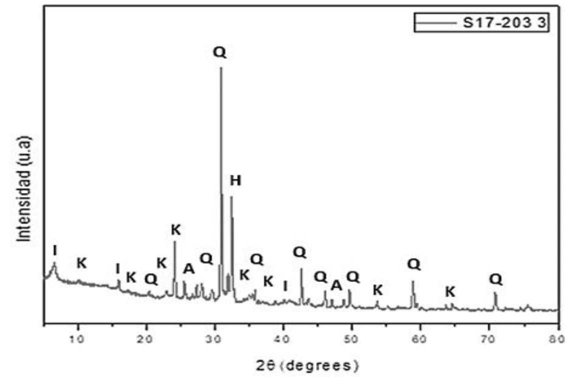
The white material has a medium to high grain size and is classified as an individual material; It should be noted that on the residues grains that appear to be feldspar are observed, so this material could have good gresification. The texture results show that with the exception of green sample 1A, which is categorized as purely clayey, the remaining samples fall within the recommended group for perforated blocks and bricks.

X-ray diffraction analysis (XRD)

All green clays are classified as purely clay materials, yellow clay 1 is classified as a silty-silty material, yellow clays 1A and 1B and white clay are classified as degreasing or thick clay materials. Figures 1 and 2 show the diffractograms of silts and degreasers respectively. Also, table 3 is presented on the Mosh hardness scale for the phases present in the clay samples for the set used, as well as the designation of each of the elements.



a) Yellow



b) White

Figure 3. Diffractogram for selected samples **a) Yellow** and **b) White**

Source: Authors

PHASES	DENOMINATION	CHEMICAL COMPOSITION	HARDNESS MOSH
Hematita	H	Fe_2O_3	5.5-6.5
Illita	I	$(KH_3O)(AlMgFe)_2(SiAl)_4O_{10}$	12
Caolinita	K	$Al_2(Si_2O_5)(OH)_4$	2-2.5
Cuarzo	Q	SiO_2	7
Anatasa	L	TiO_2	5-6

Source: Authors

Table III. Mosh hardness for the phases present in the samples.

In general, clays used in construction are called ceramic clays or common clays; which are composed of two or more clay minerals, usually illite, kaolinite and smectic, with significant amounts of other minerals that are not phyllosilicates (carbonates, quartz, etc.) and are generally used for the manufacture of building materials and aggregates [21].

The samples have a diverse composition, of which certain trends are appreciated. Some clays find their main field of application in the absorbent sector because they can retain water or other molecules in the interlaminar space (smectites) or in the structural channels (sepiolite and paligorskite). The hydration and dehydration of the interlaminar space are characteristic properties of smectites, and whose importance is crucial in different industrial uses. Therefore, smectites in the form of montmorillonite are considered to absorb a large amount of water between their interlaminar spaces, which are their main characteristic [22][23]. As for the clay minerals, it is observed that the majority of the samples are constituted by the illite in an important proportion (it is generally the second most important mineral after quartz).

In the analysis by DRX, it indicates that the clay minerals consist mainly of silica and hydrated aluminosilicates with the presence of some impurities, such as Na, Fe, K and Ca. The diffractograms reflect the phases present in the materials and highlights the high content of microcline and muscovite, which justifies its yellow color [24]. Also, this type of analysis is essential to determine their quality based on the content of SiO2 and Al2O3. It can be analyzed that the silt phase has more H (Hematite) content than degreasers, whose hardness is between 65-72 HRC. In addition, both silts and degreasers have a high Quartz content that has a hardness of 80 HRC.

Determination of Cooking Contraction

Figure 3 shows the laboratory curve used to perform the burning to be performed with the different pastes corresponding to each sample.

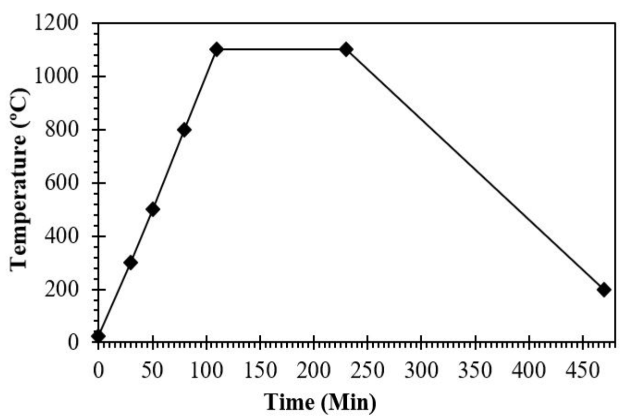


Figure 3. Burning curve performed in the laboratory.

Source: Authors

For this test, samples were organized into five types as follows: the first with green clay 1, the second with the mixture of green, the third with yellow clay1, the fourth with the mixture of yellow and the fifth with the white [25][26]. Likewise, the contraction percentages at different temperatures were determined and the respective curves are observed in Figure 4.

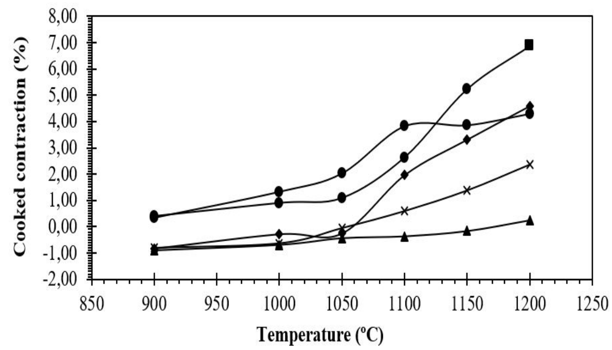


Figure 4. Contraction percentage vs temperature of the different samples

Source: Authors

Cooked contractions for green clay 1 and yellow clay 1 are high and have a marked increase from 1050 °C, without ever contracting at 1200 °C. The mixture of green clays expands to 1000 °C and after this temperature begins to contract, this behavior is typical in materials with sandy characteristics. Losses due to calcination of all samples are within normal ranges for raw materials used in the manufacture of such products. The fact of having low losses due to calcination foresees not having problems in cooking due to excess organic matter in the composition of raw materials.

Weight loss percentage.

To carry out this test, blocks extracted from the artificial dryer of the Ocaña brick with a humidity percentage of 7% were selected. Samples of 10x10x2 (cm) of 200 grams were extracted. Using a precision scale, the variation of the weight was recorded as a function of the temperature, which reached 1000 ° C, measurements were made every hour for 12 continuous hours of burning, obtaining the results for each type of clay that were subsequently averaged obtaining the data shown in table 4.

Temperature (°C)	Time (h)	Weight (gr)
100	1	199.11
200	2	192.41
300	3	191.11
400	4	190.99
500	5	186.96
600	6	181.23
700	7	180.70
800	8	180.31
900	9	177.76
1000	11	176.20
900	12	176.29

Source: Authors

Table IV. Weight variation table.

Compressive strength.

This mechanical property of the material is one of the most important because the quality of the ceramic block is determined. The tests were carried out with five cooked blocks corresponding to each of the three types of clay (purely clay, clay-silty and clay degreaser or thick), following the guidelines of the Colombian technical standard NTC 4205, the results are shown in the figure 5.

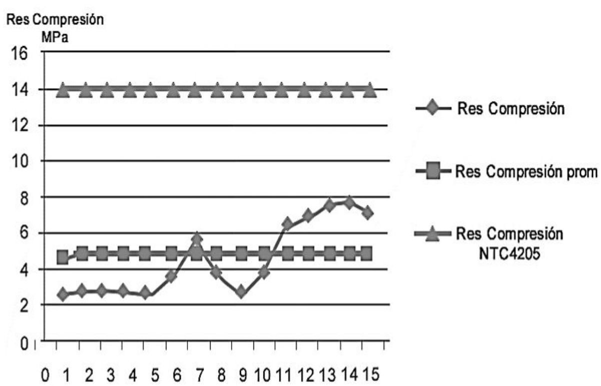


Figure 5. Compression resistance of cooked block.
Source: Authors

The minimum compressive strength of the non-structural masonry block established in NTC 4205 is 14 MPa, and a much lower average value of 4.87 MPa was found, with a maximum value presented of 7.91 MPa and a minimum value of 2.82 MPa.

Bending or breaking module

The rupture or flexural test (MR) module is a mechanical property that determines the ability of the block to withstand bending loads, stresses to which such products are subjected. The test was performed according to the guidelines of the NTC 4205 standard with five samples for each type of clay and the results are shown in Figure 6.

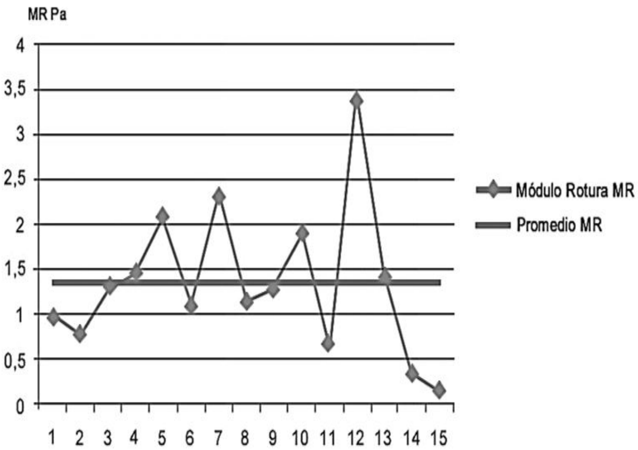


Figure 6. Cooked block flex or break module

Source: Authors

The results of this test show that the blocks have a low resistance, taking into account that the standard does not establish average values to measure the quality of this variable. These low values are due to the fact that the selected clay samples have high percentages of sand that cannot be homogenized with the other contiguous elements of the mixture in the cooking process.

Conclusions

Currently, in the artisanal sector, the mixture of clays for the manufacture of bricks is 60% of sandy clay and 40% of plastic clay, this mixture being one of the possible causes of the poor quality of the block such as: cracks, poor cooking, color non-uniform, among other factors that affect the finished product by an unsuitable dosage [27][28][29].

Of the different types of clay found in the quarry and initially classified by color and appearance, four different types of green clay, three types of yellow clay and one type of white clay were defined, which later by means of granulometry and X-ray diffraction tests , were reclassified in: purely clay, clay-silty and clay degreaser or thick, of which it was found that according to their composition and hardness they are suitable for the manufacture of openwork blocks or bricks to be used in masonry.

The results of the granulometry tests indicate that most of the samples have a 45% fine sand index; which is of great importance for the manufacture of ceramic pastes, because it allows the samples to be classified as low compaction materials, low plasticity, which should not be used alone as a production paste because they will have low dry and cooked resistance, as well as high water absorption. Also, sand is necessary for the extrusion of masonry products for construction, because it helps reduce drying time and prevents the formation of cracks in the pieces.

It can be affirmed that a good clay to be used in the production of masonry products are those samples that operate at low temperatures, that is, between 950 ºC and 1050 ºC. Where they also present water absorption values that comply with the standard of finished product that is to be produced, as established by NTC 4017 and 4205.

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