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# Technological characterization of a clay used in the manufacture of masonry products for construction

## Caracterización tecnológica de una arcilla utilizada en la fabricación de productos de mampostería para la construcción

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## ABSTRACT

**Keywords:**

Clays,
Raw Material,
Ceramics,
Bricks,
Optimization.

In the present investigation, the physical, mechanical, and chemical characterization of the raw material used in two companies dedicated to the manufacture of solid bricks in the region of Cesar, Colombia, was carried out. Initially, the hydrometric test was performed on five clay samples from different quarries with which the percentages of sand, silt, and clays were determined. These percentages were placed in the Winkler diagram to identify the types of existing clays according to their texture and the types of products that can be manufactured. Subsequently, the chemical characterization was carried out through the X-ray diffraction and fluorescence tests, to determine the phases and elements present in two of the optimal samples for the manufacture of the bricks that may arise during the cooking process. The results of the test of mechanical resistance to compression in solid brick units showed that the clays currently used by the company do not meet the minimum requirements for the production of solid clay bricks, according to the parameters of the Colombian technical standard NTC 4205-09. With this research, we expect to predict the behavior of the raw material in the stages of the production process, which will obviously improve the environmental and economic resources of the companies. The result of the characterization of the raw material will allow us to predict the structural behavior and to carry out indispensable design and reinforcement models in building and attention to the community.

## RESUMEN

**Palabras clave:**

Arcillas,
Materia Prima,
Cerámica,
Ladrillos,
Optimización.

En la presente investigación se llevó a cabo la caracterización física, mecánica y química de la materia prima utilizada en dos empresas dedicadas a la fabricación de ladrillos macizos en la región del Cesar, Colombia. Inicialmente, se llevó a cabo el ensayo de hidrometría a cinco muestras de arcilla de diferentes canteras con los cuales se determinaron los porcentajes de arenas, limos y arcillas. Estos porcentajes fueron ubicados en el diagrama de Winkler para identificar los tipos de arcillas existentes según su textura y los tipos de productos que se pueden fabricar. Posteriormente, se realizó la caracterización química a través de las pruebas de difracción y fluorescencia de rayos X, con la finalidad de determinar las fases y elementos presentes en dos de las muestras óptimas para la fabricación de los ladrillos que pueden surgir durante el proceso de cocción. Los resultados del ensayo de resistencia mecánica a la compresión en unidades de ladrillo macizo, demostraron que las arcillas utilizadas actualmente por la empresa no cumplen los requerimientos mínimos para la elaboración de ladrillos de arcilla macizo, acorde a los parámetros de la norma técnica Colombiana NTC 4205-09. Con esta investigación se espera poder pronosticar el comportamiento de la materia prima en las etapas del proceso productivo, con lo que evidentemente se mejoraran los recursos ambientales y económicos de las empresas. Resultado de la caracterización de la materia prima permitirá predecir el comportamiento estructural, y realizar modelos de diseño y reforzamiento en edificación indispensables y de atención a la comunidad.

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## Introduction

Currently the ceramic industry in Colombia plays an important role in the economic and social sectors of the country. In studies carried out in 2015 by the Asociación Nacional de Productores de Ladrillo y Derivados de Arcilla (ANFALIT) found that the brick manufacturing industry in Colombia reports a national production of 376,947 tons per month of brick (4,523,367 tons per year). Likewise, there are 1,924 brick producing companies, of which 88% are operating, 2% are liquidated and 10% are temporarily closed. [1]. Taking into account the above, it can be inferred the wide capacity that the country has in the production of brick and other masonry products for construction with very good quality and in this way generate competitiveness at the national level in this market.

The clay transformation process is mainly composed of three phases: preparation of the ceramic paste, molding of the piece and firing. The first phase corresponds to the preparation process, in which the composition and plasticity are modified for a homogeneous ceramic paste; The second phase is molding, where the brick shaping process must be taken into account, either by pressure or extrusion; and the third phase is the drying phase, where the water evaporation rate is controlled in order to avoid cracking in the piece; In addition, it aims to eliminate the percentage of moisture, reduce porosity, increase density and mechanical resistance. [2][3][4][5][6][7].

The companies dedicated to the manufacture of clay, according to their production capacity and technological development, are classified into chircales, small brickyards, medium brickyards, and large brickyards [8]. The clay transformation process consists of the following stages in the production process as shown in Figure 1.

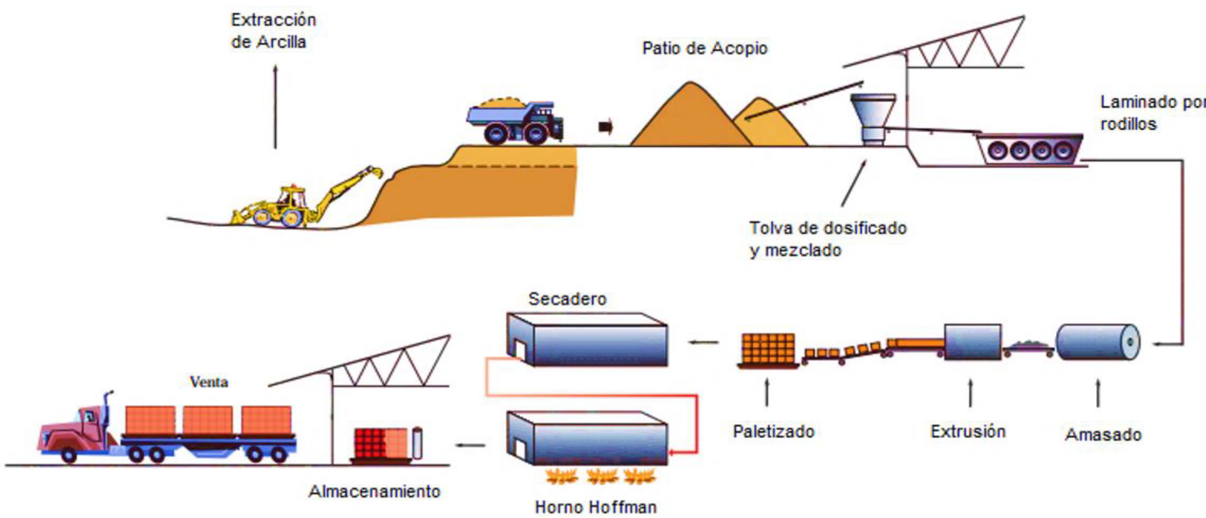


**Figure 1.** Production process of a brickyard.
**Source:** [9][10].

The clay mixtures for the manufacture of bricks are 60% sandy clay and 40% plastic clay, this composition is attributed to the poor quality of the bricks in terms of cracks, poor firing, lack of uniformity in color, among other factors that affect the finished product. These defects are associated with the sizes of the sand particles and the mineralogical composition of the raw material. [11][12][13].

The physical-ceramic characterization of the raw material is carried out to observe the behavior of the material in the drying stage, mainly determining the contraction speed of the sample at the different selected temperatures and relating the losses due to drying with the percentage of dry shrinkage of specimens formed in the laboratory [14][16][17]. This characterization of the selection of the raw material in the large ceramic industries is carried out in order to establish if it is suitable for the elaboration of the brick and in this way to be able to obtain good quality products [17]. On the other hand, the chemical characterization of raw materials is of great interest to determine the phases that can be formed during the clay shaping processes. The most common analyzes are: X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Thermal Analysis (ATD / TG) [18][19][21][22].

Taking into account the above, in the present research the physical, mechanical, and chemical characterization of two types of clays from different companies used in the manufacture of solid bricks in the Cesar region - Colombia was carried out, to improve the quality of the final product through laboratory tests that allow a global visualization of the behavior of the raw material.

## Materials and methods

The research with descriptive depth was used in the development of the project, which determines the most important variables that intervene in the selection of the raw material for the manufacture of masonry products for construction (solid bricks). The identification of the effects of the variables studied took into account an experimental design taking into account the hydrometric composition of the clays (Sands - Silts - Clays). The methodology applied for taking samples is shown in Figure 2.

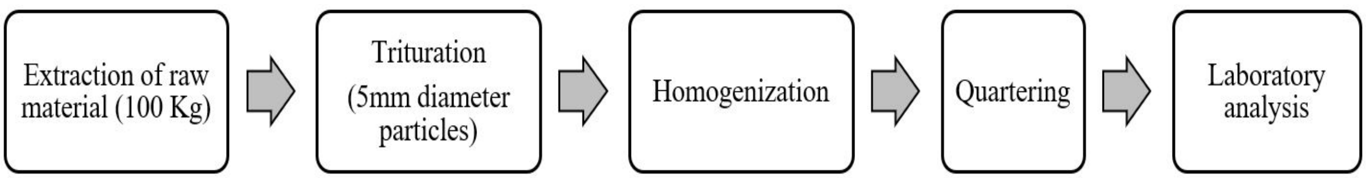


**Figure 2.** Methodology applied in the selection of raw material.

The taking of the samples was channel type, selecting several clay veins from the quarries of the El Cielo and Las Casitas companies in the town of Valledupar, Cesar - Colombia. At this stage, physical analyzes were carried out, which allowed the selection of two (2) samples, one from each quarry. Subsequently, X-ray diffraction and fluorescence analysis were carried out. Finally, a mechanical analysis was carried out to be able to evaluate if the raw material meets the technical quality standards according to the NTC 4205-09 standard, with repeatability of five times, to guarantee the statistical validity of the results. The identification of the samples was established as shown in Table 1.

**Table 1.** Analysis unit and samples (clay).

| Quarry | Analysis unit | Samples |
|---|---|---|
| Q1: El Cielo | P1 | P1M1 |
| | P2 | P2M2 - P2M3 |
| | P3 | P3M4 |
| Q2: Las casitas | P4 | P4M5 - P4M6 |
| | P5 | P5M7 - P5M8 |

### *Technological analysis*

### *Physical Characterization.*

The analysis was carried out taking into account the procedure of standard NTC 4630 and INV.E - 125 and 126 [23][24][25]. 1000 g of ASTM No 40 mesh through sample (425 µm) were taken and subdivided into four samples of 150 and 200 g each. Each sub-sample was kneaded with the amount of water necessary for the consistency of the resulting homogeneous mass to be adequate to start the test. In order to complete its homogenization, the dough is left to rest


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for 24 h. Once this period of time had elapsed, each sub-sample was kneaded again for 2 min and the test was started. By carrying out this test, the amount of water that must be added to the mixture in the mixing process before entering the extruder is determined.[26][27].

On the other hand, to determine the plasticity index it is necessary to calculate the liquid limit and the plastic limit of the samples since this corresponds to the difference between these two values. When the value is equal to zero (0) or less, the soil is considered to be non-plastic and therefore difficult to extrude. For construction activities, the ideal condition is a high plastic limit (LP) and a low liquid limit (LL).

***Chemical characterization.***

For the preparation of the samples, the parameters of the ASTM C323-56 standard were followed, to obtain a particle size of the raw material of around 38 µm.

X-ray diffraction or XRD allows the identification of the crystalline phases present in a ceramic or metallic sample [27]. To carry out this test, the Bruker D4 AXS equipment was used, with LYNXEYE detector, X-ray tube Cu 1.9Kw (Kα-λ = 1.5406) with Nickel filter at 40 KV 40 mA, with a normal electron beam and 2θ incidence angles : 0-120 ° (powder mounting) and a speed of 0.4 s/θ.

Likewise, X-ray fluorescence or X-ray fluorescence, also called X-ray spectrography, is a physical method for the analysis of clay materials, its final objective is to qualitatively and quantitatively determine the chemical composition of a sample. [27]. To carry out this test, the Bruker S4 Explorer equipment was used, with Pro4 detector, X Rh test tube at a voltage of 40 KV at 25mA.

***Mechanical Characterization.***

Taking into account the specifications of the NTC 4205 standard for the approval or rejection of a masonry unit [28], the normative compressive strength values are shown in Table 2. The resistance in one of the five units tested may be less than 10% than indicated as long as the indicated average value complies with the specified minimum value. If any of the units does not comply, the batch is re-sampled and the compressive strength test is repeated. If the result is null again, the test lot is rejected.

**Table 2.** Compressive strength in masonry units.

| Masonry type | Minimum compressive strength (MPa) | |
|---|---|---|
| | Average 5 Units | Unit |
| Structural | | |
| M (1) | 20 | 15 |
| Non structural | | |
| M (1) | 14 | 10 |

**Note: Where M are solid units. Source:** [29].

## Results and discussions

***Physical characterization***

Granulometry. In the first step to determine percentages of sands, silts, and clays of the two companies C1 and C2 that are part of the composition of the clay paste, the analysis of granulometry by sieving and hydrometry was carried out, it is observed in Figure 3.

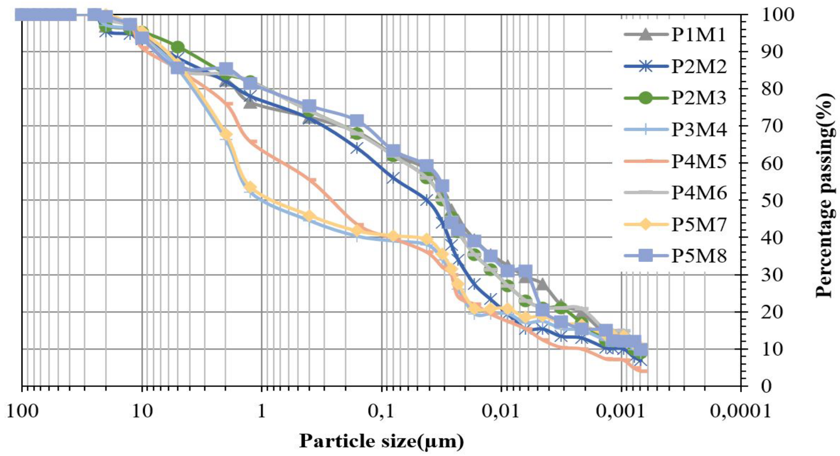


**Figure 3.** Granulometry results for the selected samples of the companies studied.

From Figure 3 the percentages of sands, silts and clays were determined, obtaining Table 3 of results.

**Table 3.** Granulometric composition of the samples.

| Percentage | El Cielo | | | | Las Casitas | | | |
|---|---|---|---|---|---|---|---|---|
| | P1M1 | P2M2 | P2M3 | P3M4 | P4M5 | P4M6 | P5M7 | P5M8 |
| Clay <2 µm | 21.16 | 25.40 | 34.64 | 24.85 | 37.40 | 26.81 | 26.00 | 25.44 |
| Silt >20 µm | 61.64 | 42.20 | 44.66 | 39.55 | 8.60 | 23.69 | 18.50 | 33.36 |
| Sand 2 a 20 µm | 17.20 | 32.40 | 20.70 | 35.60 | 54.00 | 49.50 | 55.50 | 41.20 |
| Obtained texture | Loam-Silty | Loam | Loam-Clay | Loam | Clay-Sandy | Loam-Clayey-Sandy | Loam-Clayey-Sandy | Loam |

The texture was obtained from the triangular diagram of the basic textural classes of the Winkler soil and the percentages of sands, silts, and clays for each of the studied samples. It was observed that the type of texture is related to the composition percentage of the soil sample because the P1M1 sample presented a Loam-Silty texture, which suggests that the sample has a greater amount of silt, Table 3 confirms its textural classification because silt has a higher proportion with a value of 61.64% in its particle distribution. From the data of the granulometric composition, the different applications of the raw material can be known. Likewise, Figure 4 shows the types of products that can be manufactured taking into account the results obtained by granulometry and hydrometry (Table 3).


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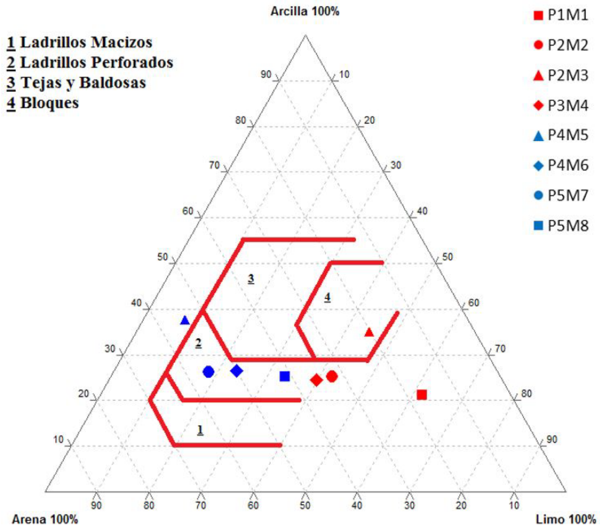


**Figure 4.** Types of products that can be manufactured with company samples.

Taking into account the Winkler percent triangle, the texture results indicate that most samples are suitable for the manufacture of masonry products, except P4M5 and P1M1. However, in the case of the sample P2M3, it is suitable for the manufacture of blocks because it is in group 4 as indicated in Figure 4. Meanwhile for the rest of the samples, its possible use for the manufacture of perforated bricks, and therefore an optimal raw material for the manufacture of solid bricks.

Moisture content. The determination of the water content is one of the most characteristic physical properties to define the behavior of soil. Among the behaviors are cohesion, volume changes, and mechanical stability, among others.

Figure 5 details the results of the moisture content of the samples under study.

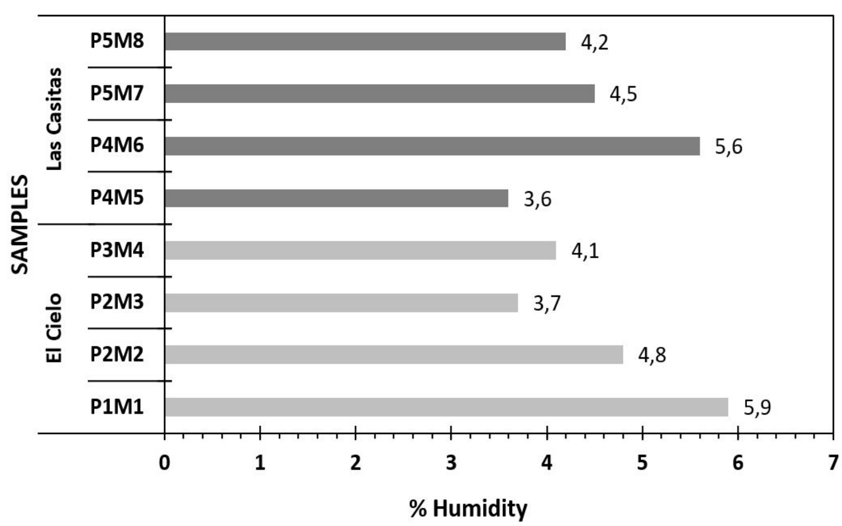


**Figure 5.** Humidity percentages of company samples.

Figure 5 shows that the humidity percentage is maintained in a range of 3.6% as a minimum in the P4M5 sample and 5.9% as a maximum in the P1M1 sample; that is, the sample has a greater capacity to store water among its particles. Clearly, it was possible to show in a general way that brickyards have a minimal variation in moisture content, being the houses that present the highest percentage of moisture, due to their standard deviation (C1=4.61±0.95 y C2=4.48±0.83), therefore, it has less dispersion in its results. This gives it a better property of elastic character that it infers in the use of the ceramic industry, typical in the manufacture of artisan solid brick.

**Consistency or Atterberg limits.** The liquid limit, plastic, and plasticity index are shown in Table 4. These allowed to quantify the plasticity contained in the clay samples, in addition to their quality, nature, and behavior.

**Table 4.** Atterberg limits.

| Brickwork | Sample | Liquid Limit (%) | Limit of plasticity (%) | Plasticity index (%) |
|---|---|---|---|---|
| El Cielo | P1M1 | 30.9 | 23.4 | 7.5 |
| | P2M2 | 24.0 | 18.4 | 5.6 |
| | P2M3 | 34.9 | 25.5 | 9.4 |
| | P3M4 | 31.0 | 21.4 | 9.6 |
| Las Casitas | P4M5 | 31.5 | 19.9 | 11. 6 |
| | P4M6 | 21.2 | 2.1 | 19.9 |
| | P5M7 | 22.0 | 15.5 | 4.5 |
| | P5M8 | 22.6 | 13.8 | 8.8 |

Figure 6 shows the graph of the results obtained in the Casa-Grande diagram, for the samples of the selected companies, to determine the plastic behavior of the raw material.

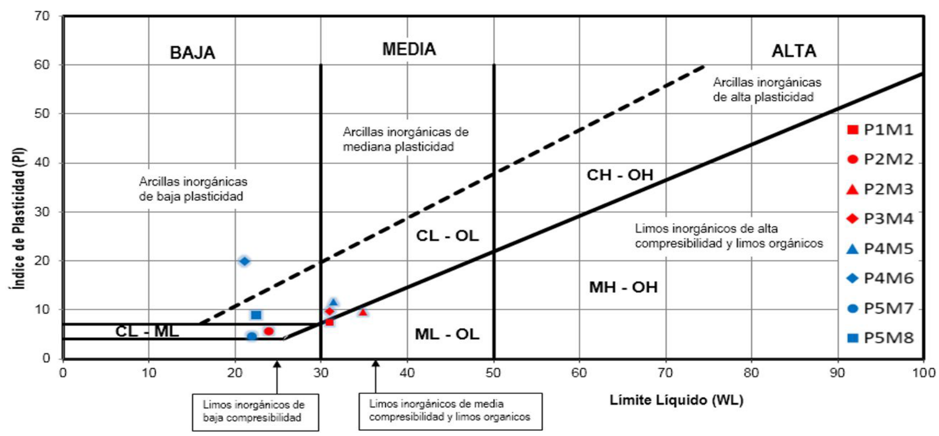


**Figure 7.** Extrusion and / or molding prediction diagram through Atterberg limits.

From Figure 6, the geotechnical properties of a soil sample can be predicted. In this case, according to the Casa-grande diagram, the samples from the El Cielo brickyard: (i) P1M1 exhibits a low to medium plasticity behavior, (ii) P2M2 shows a corresponding behavior with a low plasticity clay similar to silty clay, (iii) P2M3 is in the range of medium plasticity, but below the A-line; which delimits silty soils, and (iv) P3M4 is also in medium plasticity, but above line A that delimits clayey soils. In the brickyard Las Casitas; (i) P4M5 has a medium plasticity behavior, (ii) P4M6 has low plasticity, (iii) P5M7 is considered low plasticity, and (iv) P5M8 is low plasticity. These results are similar to those reported by García-León for a clay from the Norte de Santander region in Colombia [26] and another similar study [30]. The interpretation of the results for each of the samples is observed in Table 5.


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**Table 5.** USCS Classification of Samples.

| Samples | USCS classification | Group name |
|---|---|---|
| **P1M1** | ML o OL | Low compressibility slime |
| **P2M2** | CL – ML | Clay-silty |
| **P2M3** | ML o OL | Low compressibility slime |
| **P3M4** | CL- OL | Low compressibility clay - organic clay. |
| **P4M5** | CL- OL | Low compressibility clay - organic clay. |
| **P4M6** | Low plasticity zone | Low compressibility clay |
| **P5M7** | CL-ML | Clay - silty |
| **P5M8** | Low plasticity zone up (CL) | Sandy or silty clay |

**Note:** C: Clays M: Silts, O: Organic matter, L: Low compressibility, H: high compressibility.

Subsequently, with the same results, the analysis was carried out to predict the behavior of the raw material molding, which took into account the Atteberg limits, as shown in Figure 7.

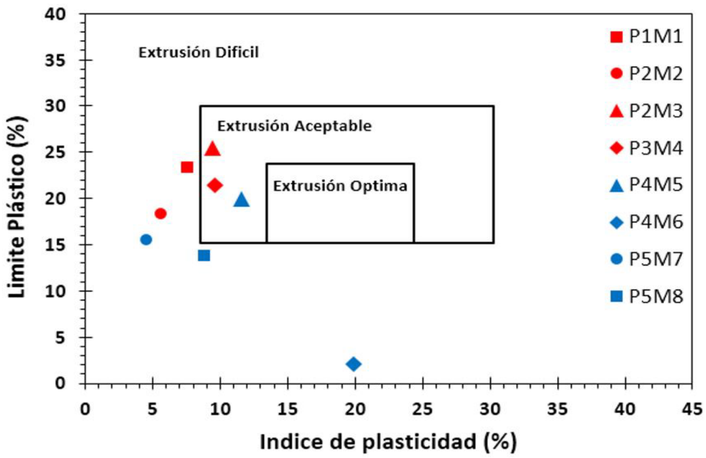


**Figure 7.** Extrusion and / or molding prediction diagram through Atterberg limits.

Figure 7 shows the diagram of the extrusion behavior, it illustrates the prognosis of the molding and / or extrusion for each of the samples. On the other hand, it can be observed that the sample that the samples P2M3 and P3M4 from El Cielo and P4M5 from Las Casitas, are those that present the best behavior in extrusion with a range of acceptable, being for the others, a behavior in difficult extrusion. This generates more operating expenses in terms of the transformation of the raw material in the extrusion process and the final quality of the bricks. When there is a higher content of degreasing material, it can be said that there is less plasticity in the soil.

Additionally, the expansion potential of the samples can be analyzed according to the current regulations outlined in NSR-10, chapter H. All clays have as a general characteristic the property of contracting when they lose moisture and of expanding when they gain moisture again according to environmental conditions. . At present, buildings present fissures and cracks due to soils with expansive properties, this phenomenon is more evident in winter or summer due to thermal changes.

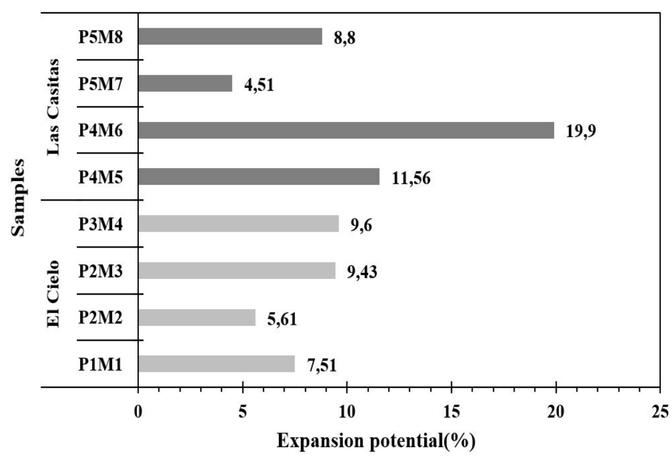


**Figure 8.** Potential expansion diagram of samples.

Figure 8 presents the results of the expansion potential, where the samples may not be classified in the group of expansive clays, due to their ability to increase the volume in the presence of water is between low and medium-low.

### *Chemical and mineralogical composition*

Taking into account the previous results, the Casitas samples P5M7 and P5M8 are selected for the execution of fluorescence and X-ray diffraction studies. Since these are in the same stratum, this will allow later investigations that will focus on making a mixed design to improve the final product based on its chemical and mineralogical composition.

**X-ray fluorescence.** The results of the chemical composition and sediments present in the Table 6.

**Table 6.** Chemical composition of samples.

| NAME OF THE ELEMENT | **COMPOUND** | **P5M7** | **P5M8** |
|---|---|---|---|
| Silicon oxide | $SiO_2$ | 64.01 | 61.07 |
| Aluminum oxide | $Al_2O_3$ | 15.98 | 16.69 |
| Iron oxide | $Fe_2O_3$ | 6.25 | 7.29 |
| Calcium oxide | CaO | 2.62 | 2.50 |
| Potassium oxide | $K_2O$ | 2.65 | 2.55 |
| Magnesium oxide | MgO | 0.90 | 1.79 |
| Titanium oxide | $TiO_2$ | 0.81 | 0.85 |
| Sodium Oxide | $Na_2O$ | 0.85 | 0.95 |
| Barium oxide | BaO | 0.13 | 0.14 |
| Manganese Oxide | MnO | 0.12 | 0.14 |
| Match | $P_2O_5$ | 0.09 | 0.10 |
| Copper Oxide | CuO | 0.03 | 0.03 |
| Vanadium | $V_2O_5$ | 0.02 | 0.02 |
| Strontium Oxide | SrO | 0.02 | 0.03 |
| Losses | LOI | 5.53 | 5.84 |

**Note:** LOI: Loss on Ignition.

The samples have a high content of silica and alumina; Furthermore, the iron content in both samples is above 2-2.5%, allowing us to infer that the color of the final product will have a characteristic red [29]. Because the content of A12O3 was less than 30%, these two materials cannot be classified as semi-refractory. The clay samples are considered a suitable medium for the manufacture of ceramics because due to their values they are not completely suitable; for these samples to be suitable, their percentage of a chemical component must range for the SiO2 between 50% and 60% and Al2O3 between 20% and 30%.

X-ray diffraction. The results obtained regarding the mineralogical components can be seen in Table 7.


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**Table 7.** Crystalline phases identified for the two samples.

| Mineral | Chemical formula | Natural clay. (wt.%) | |
|---|---|---|---|
| | | P5M7 | P5M8 |
| Low quartz | $O_2Si_1$ | 43.5 | 41.0 |
| Microcline | $Al_1K_1O_8Si_3$ | 30.5 | 20.2 |
| Muscovite | $H_{1.79}Al_{2.65}Fe_{0.17}KO_{0.93}Mg_{0.03}\ Na_{0.07}O_{12}\ Si_{3.18}$ | 14.0 | 4.8 |
| Clinocrototile | $H_4Mg_3O_9Si_2$ | 9.8 | 0.4 |
| Low albite | $Al_{1.16}Ca_{0.16}Na_{0.84}\ O_8Si_{2.84}$ | 1.2 | 32.8 |
| Magnesium ferrite | $Fe_2Mg_1O_4$ | 0.9 | 0.8 |

On the other hand, Figure 9 shows the diffraction patterns obtained for the two selected samples.

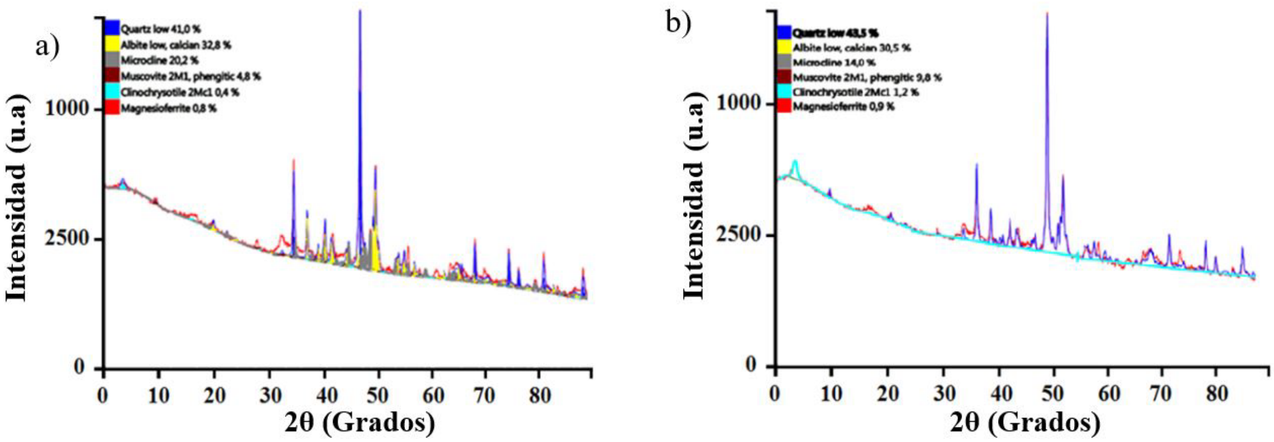


**Figure 9.** X-ray diffractogram. A) Sample P5M7 and b) Sample P5M8

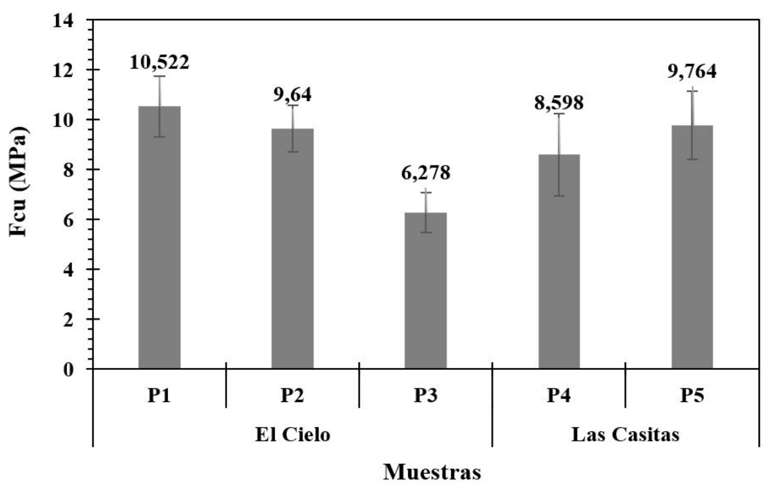


**Figure 10**. Resistance to compression of solid brick units.

Table 8 shows the results of the compressive strength of solid brick units with a value lower than 15 MPa, indicating that the samples should not be for structural use, only 5 units from the El Cielo brickyard and 4 from Las Casitas reach the limit to be classified as non-structural use [30][31]. Therefore, the samples of the selected companies must be mixed with others of better quality to optimize economic resources and achieve a product that meets the requirements of current Colombian regulations.

**Table 8.** Results of compressive strength (Fcu).

| Brickwork | Sample | Fcu | |
|---|---|---|---|
| | | Average | Standard deviation |
| El Cielo | P1 | 10.52 | 1.23 |
| | P2 | 9.64 | 0.93 |
| | P3 | 6.27 | 0.80 |
| Las Casitas | P4 | 8.59 | 1.64 |
| | P5 | 9.76 | 1.36 |

In the case of averaging the samples to 5 units, the analysis must take into account that the minimum required for structural masonry is 20 MPa, and for non-structural it is 14 MPa. It was evidenced that none of the average samples meets the minimum standards required for resistance to structural and non-structural compression.

## Conclusions

When analyzing the clay samples from the "El Cielo and Las Casitas" quarries, it can be deduced that these have characteristics that are dominant when obtaining more homogeneous and better quality products, even with excellent raw material performance; such as texture, plasticity, chemical and mineralogical composition, on which good practices and permanent control should be adopted. Taking into account the results obtained when performing the humidity tests, granulometry and Atterberg limits, from the total of selected samples it is evidenced that:

- All the samples presented high sand contents, higher than 25%, except the samples P4M5 and P5M7, whose indicators are between 10 and 20%, for which the extra addition of degreasing material is necessary to be able to increase and improve their characteristics. According to the unified soil classification system (SUCS) and the Casagrande diagram, most of the samples are classified as low comprehensibility inorganic clays, classified in the CL - ML zone, behaving as loamy-clay-silty soils, slightly plastic.

- The best clays for the production of bricks are the impure ones, with around 33% sand and silt, as these reduce shrinkage and cracking during drying and burning. The clays exploited and used in the production of bricks in the quarries El Cielo and Las Casitas do not meet the criteria for the distribution of particles (granulometry) for the production of common or solid bricks.

- The chemical components in the highest proportion were silica ($SiO_2$), alumina ($Al_2O_3$), iron oxide ($Fe_2O_3$) and titanium oxide ($TiO_2$); its presence is classified as favorable in the ceramic industry. The X-ray diffraction study confirms the presence of silica, due to the presence of quartz significantly in the samples. Furthermore, it was found that in the crystalline phases of clays P5M7 and P5M8, quartz existed in a large proportion as a common mineral in both. This mineral guarantees clay the character of being a degreasing material, which is related to the granulometry where these two have a higher percentage of sand in their component.

The highest strengths were obtained from the samples produced with the P1 clays from the El Cielo quarry, with an average strength of 10.52 MPa, of the 25 brick specimens tested, the L2 sample presented the highest value of resistance to simple compression (12.05 MPa) . The P1 and L2 clay samples from the El Cielo quarry present a sand content of 17.2%, silt of 61.64%, and clay of 21.16%. The lowest resistances were obtained from the samples produced with the P3 clays from the El Cielo quarry, with an average resistance of 6.28 MPa, of the 25 brick specimens tested, the L12 sample presented the lowest value of resistance to simple compression (6.64 MPa), and these clays have a sand content of 35.6%, silt of 39.55% and clay of 24.85%.

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