

# *Experimental design for the optimization of dry-pressed ceramic bricks (Dry-Press)*

## Diseño experimental en la optimización de ladrillos cerámicos prensados en seco (Dry-Press)

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### Abstract

One of the most common products in the construction industry is brick due to its advantages over other building materials such as blocks. In developing countries like Ecuador, brick is handcrafted and is the source of income for thousands of families. These bricks lack a systematized process that optimizes resources to make a brick that meets local regulations. This study evaluates the use of the different raw materials used in production through experimental designs to determine the amount of optimal components locally available ("White Clay," "Black Clay," "Water") and achieve the maximum compressive strength in dry-pressed ceramic bricks. This research also identifies the behavior of the compressive strength based on the compaction pressure in the molding stage, concluding that the model that best fits the behavior is a quadratic model, and from the trace plot, it was observed that compared with "White Clay," "Black Clay" contributes further to the brick strength. The optimal amount of components to meet the 6 MPa pressure standard required in the brick was 5% water, 85% "Black Clay" and 10% "White Clay." The pressure value required in the compaction stage was 4.9Mpa (712psi), and greater strength can be achieved by only adding "Black Clay" and water.

Keywords: Bricks; Pressed; Mixture Design; Optimization

### Resumen

Uno de los productos más comunes en la industria de la construcción es el ladrillo debido a las ventajas que presenta frente a otros materiales de construcción como los bloques. En los países en vías de desarrollo como Ecuador, el ladrillo es elaborado de manera artesanal siendo el sustento de miles de familias. Estos ladrillos carecen de un proceso sistematizado que permita optimizar los recursos para lograr un ladrillo que cumpla con las normativas locales. En el presente trabajo de investigación se evaluó el uso de las diferentes materias primas utilizadas en la fabricación mediante el uso de diseños de experimentos con el objetivo de determinar la cantidad de componentes óptimos disponibles localmente ("Arcilla Blanca", "Arcilla Negra", "Agua") para alcanzar la mayor resistencia a la compresión en ladrillos cerámicos prensados en seco, y a la vez, identificar el comportamiento de la resistencia a la compresión en función de la presión de compactación en la etapa de moldeo. De los resultados obtenidos se concluyó que el modelo que mejor se ajusta al comportamiento es un modelo cuadrático, y del gráfico de trazas de observo que en comparación a la "Arcilla Blanca", la "Arcilla Negra" contribuye más en la resistencia del ladrillo. Los valores óptimos alcanzados para cumplir con la norma de 6Mpa de presión requerida en el ladrillo fueron 5% de agua, 85% de "Arcilla Negra" y 10% de "Arcilla Blanca". Además, el valor de la presión requerida en la etapa de compactación fue de 4.9Mpa. Pudiéndose lograr mayores valores de resistencia con la adición únicamente de "Arcilla Negra".

Palabras clave: Ladrillos; prensados; diseño de mezclas; optimización

## 1. Introduction

In Ecuador, a large part of the artisanal sector is dedicated to the production of ceramic bricks by molding. The process consists of preparing a homogeneous mixture with different raw materials such as Water, Sawdust, "White" Clay and "Black" Clay and then moving on to the molding stage, followed by drying. Finally, the brick is fired in kilns at temperatures of over 900 °C (Manoharan et al., 2011).

As all artisans do not have the same systematized process to distribute the right amount of raw material or to follow a defined standard in the subsequent processes, the bricks do not meet the standards established in the INEN 297 Ecuadorian standard (NTE INEN 0297: Ladrillos Cerámicos. Requisitos, n.d.) (NTE INEN, 1977)

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An alternative to the conventional brick manufacturing process by molding is the production of bricks by pressing (Dry-Press) (Industry Association, 2006). Among the most important advantages of this process, compared to the conventional process, is the little or almost no use of water. The bricks obtain greater mechanical strength because of the better compaction of the raw material and have a better surface finish.

In order to implement a brick-making process by pressing, it is necessary to establish the most important guidelines in the brick-making process. These include determining the exact amount of components and compaction pressure that maximize the compressive strength of the bricks.

This research aims to carry out the necessary experimental designs to determine the optimum values of the different factors to maximize the mechanical strength of bricks by pressing.

## 2. Methodology

This research was conducted in the city of Chambo (Chimborazo, Ecuador), as the main economic activity of its inhabitants is the production of bricks.

To make an objective comparison with locally produced bricks and to establish a knowledge base, raw materials (clays) used by the artisans to make the bricks were characterized.

Among the clays used by the artisans, two types were found, commonly known as "White Clay" and "Black Clay" due to their colors. (Table 1) shows the chemical composition of each clay obtained by X-ray fluorescence (XRF).

Table 1. Chemical composition of clays

<b>Metal oxides</b>	<b>Black Clay</b>	<b>White Clay</b>
<b>SiO<sub>2</sub></b>	56.42%	56.17%
<b>Al<sub>2</sub>O<sub>3</sub></b>	14.99%	15.35%
<b>Fe<sub>2</sub>O<sub>3</sub></b>	6.35%	6.33%
<b>CaO</b>	4.84%	4.76%
<b>Na<sub>2</sub>O</b>	1.85%	1.80%
<b>K<sub>2</sub>O</b>	1.24%	1.28%
<b>MgO</b>	1.04%	1.42%
<b>TiO<sub>2</sub></b>	0.88%	0.84%
<b>P<sub>2</sub>O<sub>5</sub></b>	0.42%	0.21%
<b>BaO</b>	0.15%	0.11%
<b>SO<sub>3</sub></b>	0.11%	0.09%
<b>MnO</b>	0.10%	0.10%
<b>SrO</b>	0.09%	0.08%
<b>Cl</b>	0.03%	0.02%
<b>ZrO<sub>2</sub></b>	0.03%	0.03%
<b>CuO</b>	0.01%	0.01%
<b>Cr<sub>2</sub>O<sub>3</sub></b>	0.01%	94ppm
<b>ZnO</b>	0.01%	91ppm

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From the results shown in (Table 1), it can be observed that the metal oxides with the greatest presence in the clays are  $SiO_2$ ,  $Al_2O_3$ ,  $Fe_2O_3$ , and  $CaO$ , which are responsible for providing mechanical strength and the characteristic color to the brick. These metal oxides are within the admissible range for the production of these products, according to (Solis, 2014).

Although there is almost the same amount of oxides in the “White Clay” and “Black Clay” samples, the characteristic color of the “Black Clay” comes from the presence of organic matter. This allows better compaction in the pressing stage.

To determine the optimum composition of the different components (Water, “White Clay,” and “Black Clay”), a mixture design was performed considering previous research to determine the maximum and minimum limits of the raw material (Mahmoodi et al., 2020); (Tonduba et al., 2020); (Zhu et al., 2016). For water, the lower limit was established as 5% of the composition and a maximum of 15%.

The mixture design used was the Simplex augmented grid type. (Figure 1) shows the geometric representation of the established mixture design. The experimental region is the shaded area considering the restrictions of each component. Each red dot represents the treatments to be used in each sample according to (Table 2).

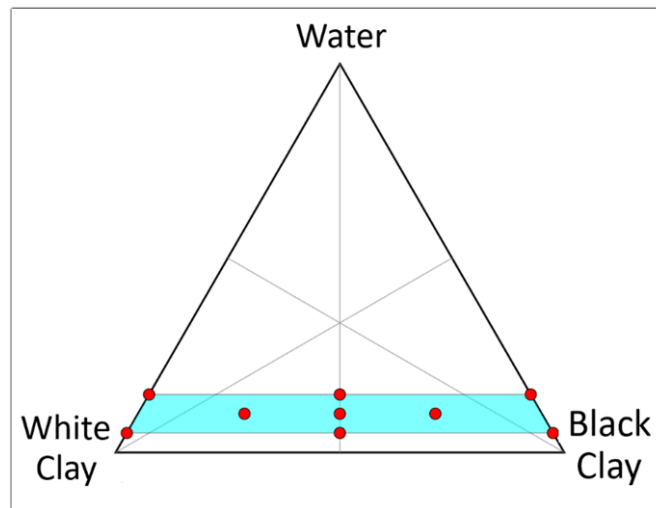


Figure 1. Geometrical representation of the Simplex Augmented Grid Mixture design

(Table 2) shows the points of the experimental region of the mixture design illustrated in (Figure 1), which summarizes the mixture design to be followed for the research.

Table 2. Mixture Design

Treatment	Black Clay %	Water %	White Clay %
1	0	0.15	0.85
2	0	0.05	0.95
3	0.85	0.15	0
4	0.95	0.05	0
5	0.42	0.15	0.42
6	0.47	0.05	0.47
7	0.22	0.11	0.67
8	0.67	0.11	0.22
9	0.45	0.1	0.45

Each mixture shown in (Table 2) was made in triplicate for better statistical analysis (two repetitions are recommended as a basis). A total of 27 samples were made from which mixture 3 could not be analyzed as, due to the higher water percentage, they presented greater plasticity and could not be compacted. This resulted in a total of 24 samples (experimental units).

To optimize resources, the mixture design was combined with a single-factor design considering the pressure with levels of 500, 1000, and 1500 psi applied to each repetition, respectively.

The 24 experimental samples prepared were fired at a temperature of 900°C for 24 hours, as shown in (Figure 2).



**Figure 2.** Fired and labeled samples

The compression test was performed according to the INEN 294 standard (NTE INEN 0294: Ladrillos Cerámicos. Determinación de La Resistencia a La Compresión, n.d.) on each sample as shown in (Figure 3).



**Figure 3.** Compression test for each simple

STATGRAPHICS Centurion XVI.I statistical software was used to perform the statistical analysis (ANOVA) of the experimental designs.

### 3. Results and discussion

(Table 3) shows the compression test results.

**Table 3.** Compression test results

<i>ID</i>	<i>Treatment</i>	<i>Repetition</i>	<i>Molding Pressure (psi)</i>	<i>Strenght (MPa)</i>
1	1	1	500	1.41
2	1	2	1000	1
3	1	3	1500	0.94
4	2	1	500	0.78
5	2	2	1000	1

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6	2	3	1500	1.11
7	3	1	500	-
8	3	2	1000	-
9	3	3	1500	-
10	4	1	500	4.65
11	4	2	1000	9.25
12	4	3	1500	9.14
13	5	1	500	3.54
14	5	2	1000	2.93
15	5	3	1500	5.47
16	6	1	500	1
17	6	2	1000	2.71
18	6	3	1500	1.99
19	7	1	500	2.38
20	7	2	1000	2.06
21	7	3	1500	1.54
22	8	1	500	6.55
23	8	2	1000	5.6
24	8	3	1500	7.95
25	9	1	500	3.93
26	9	2	1000	4.44
27	9	3	1500	3.54

(Table 4) shows the statistical results to determine the model that best fits the behavior of the data.

**Table 4.** Regression models adjusted to data

Model	p-value	R <sup>2</sup>
Linear	0.000	72.84
Quadratic	0.017	82.55
Special Cubic	0.907	81.40

The results in (Table 4) show that the model that best fits the behavior of the samples is the quadratic model, which has a significance of less than 0.05 ( $p < 0.05$ ), indicating that it can predict the model with a confidence of 95%. In addition, the R<sup>2</sup> value for this model is 82.55, which is higher than the Linear and Special Cube models. The R<sup>2</sup> value defines how well the experimental data fit the model.

(Equation 1) shows the mathematical model that determines the behavior of the strength based on the different components added to the mixture.

$$\text{Strength} = -182.965 * \text{Water} + 0.940024 * \text{White Clay} + 7.69089 * \text{Black Clay} + 206.808 * \text{Water} * \text{White Clay} + 251.211 * \text{Water} * \text{Black Clay} - 9.56371 * \text{White Clay} * \text{Black Clay} \quad (1)$$

(Table 5) shows the optimal values of the mixture percentage needed to satisfy the sample using the mathematical model presented in (Equation 1).

**Table 5.** Optimization at 6Mpa

Factor	Optimal Value
Water	0.0509122
White Clay	0.108932

Black Clay	0.840156
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(Figure 4) shows the behavior of each component in the trace plot.

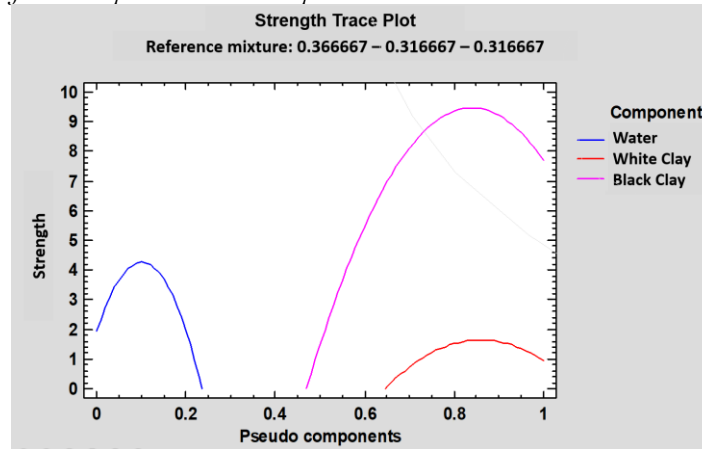


Figure 4. Compressive strength trace plots

The trace plot in (Figure 4) shows the contribution of the “Black Clay” to the strength of the brick compared to the other components.

From the statistical analysis made, it was observed that the *p*-value of the compressive strength factor did not reach a value less than 0.05 due to the lack of data necessary for the analysis. For this reason, it was decided to make a design of experiments only for the molding pressure (500, 1000 and 1500psi) with three repetitions using the optimal mixture values previously found. The compression test results for this design are shown (Table 6).

Table 6. Compression test results (molding pressure)

Repetition	Pressure	Strength (Mpa)
1	500	3.18
2	500	3.39
3	500	4.02
1	1000	7.54
2	1000	7.82
3	1000	7.96
1	1500	5.92
2	1500	6.65
3	1500	6.94

The results from the Analysis of Variance applied to the data in (Table 6) showed a *p*-value of 0.000, which means that there are statistical differences between the data. For this reason, a multiple range test was performed, as shown in (Figure 5).

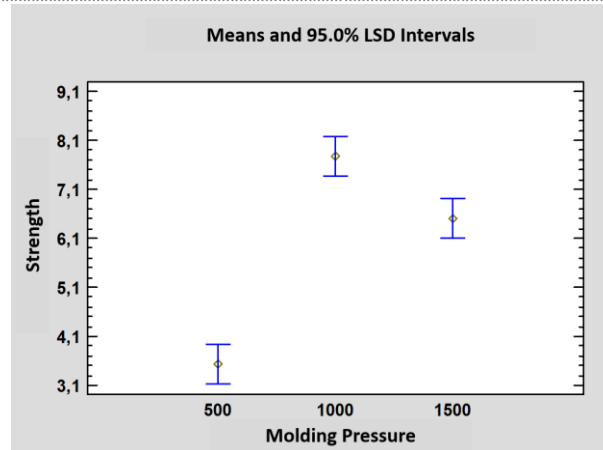


Figure 5. Multiple range test (LSD)

The LSD (Least Significance Difference) multiple range test shown in (Figure 5) determines that the highest compressive strength is achieved at a molding pressure of 1000psi. It is also observed that the higher the molding pressure, the lower the strength. Several authors (Indra et al., 2018) state that brick strength is directly proportional to the pressure in molding. This can be proven by the increase in strength reached from 500psi to 1000psi. However, not the same can be said from 1000psi to 1500psi as the strength of the ceramic brick decreases. This happens because the bricks, when compacted at very high pressures, present small failures in the form of cracks, causing an accumulation of mechanical stresses at these points and weakening the brick. This is consistent with Russel's description (Heuer, 1933)

Through regression analysis, it was possible to obtain a quadratic model that fits the points consistently with an  $R^2$  value of 96.5. (Figure 6) shows the fitted curve.

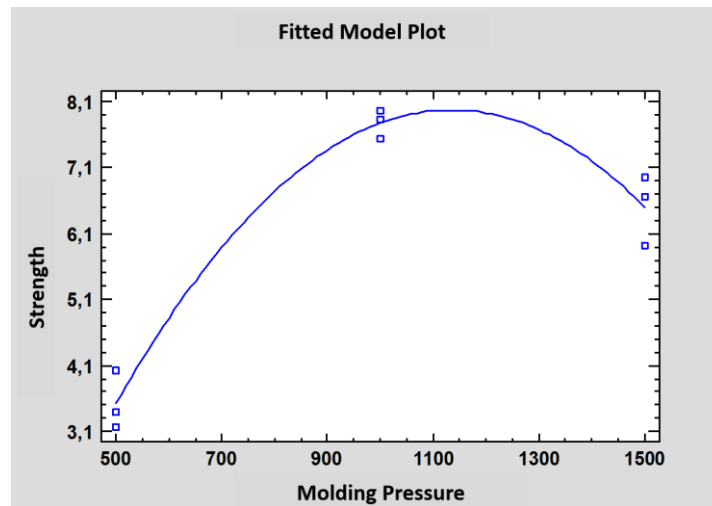


Figure 6. Curve fitted to the strength values based on molding pressure

The model obtained from the regression analysis showed that the molding pressure needed to reach the compressive strength limit value established by the standard is 712psi.

#### 4. Conclusions

The optimal mixture that reaches the highest compressive strength using the local raw material is 5% water, 10% "White Clay," and 85% "Black Clay."

The addition of white clay can be omitted, reaching compressive strength values higher than those established by the standard of 6Mpa.

The compaction pressure of 4.7Mpa in the molding was the optimum according to the regression analysis shown in (Figure 6). This pressure was determined using the optimal mixture to reach a compressive strength of 6Mpa.

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5. *The color of “Black Clay” compared to “White Clay” depends on the amount of organic matter present. From the preliminary results obtained, the “Black Clay” has higher strength because of its plasticity as it contains more organic matter, resulting in better cohesion between particles.*

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