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Optimization of Bio-Brick Composition Using Agricultural Waste: Mechanical Properties and Sustainable Applications

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Abstract: The construction industry is a major contributor to environmental pollution, with cement production only accounting for nearly 8% of global CO_2 emissions. Sustainable alternatives, such as bio-bricks incorporating agricultural waste, offer a promising solution to reduce emissions. This study investigates the development and optimization of bio-bricks using lignin as reinforcement in cementitious composites. A mixture design approach was applied to determine optimal proportions of cement, lignin, and bovine excreta, enhancing mechanical properties such as compressive and flexural strength while promoting sustainability. Response Surface Methodology (RSM) was used to model the effects of mixture components, revealing that a blend of 959 g of cement, 224 g of lignin, and 314 g of bovine excreta resulted in the best performance. Compressive strength reached ~1.7 MPa, demonstrating the composition viability for eco-friendly construction. The study highlights the bio-brick's potential to mitigate the environmental impact by reducing reliance on traditional cement while integrating renewable materials.

Keywords: bio-bricks; agricultural waste; coffee husks; sustainable construction; response surface methodology; mechanical properties

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1. Introduction

Organic waste disposal remains one of developing countries' most pressing environmental challenges. In many cases, waste is disposed of in landfills, which damages the environment and promotes health risks due to insect infestation and the spread of diseases. This issue highlights the critical need for innovative strategies to manage organic waste effectively. One promising solution is waste integration into the construction industry as a low-cost, sustainable building material. Note that cement is one of the most widely used materials in civil engineering and is essential for building construction and infrastructure. Its history reflects its evolution into an indispensable material today, improving its chemical composition for practical civil constructions and producing high CO_2 in the environment [1].

Agricultural waste has been widely used as a component in producing earth bricks, offering benefits such as improved dimensional stability, enhanced strength, and greater resistance to erosion. Additionally, incorporating agricultural products into bricks can

reduce their thermal conductivity and increase durability, making structures more comfortable in colder climates (see Figure 1). In comparison, previous studies have focused on oil palm shells and coconut waste in concrete [2]. Considering the above, bio-brick is a sustainable building material developed through organic waste incorporation into the brick-making process, such as agricultural residues. These bricks are typically produced using environmentally friendly methods, like microbial-induced calcite precipitation (MICP), which binds materials together to form a solid structure. Bio-bricks offer a lower carbon footprint than traditional bricks by utilizing waste products and reducing their reliance on conventional cement and other energy-intensive materials, presenting a novel alternative for sustainable construction promoting resource efficiency and waste recycling [3].

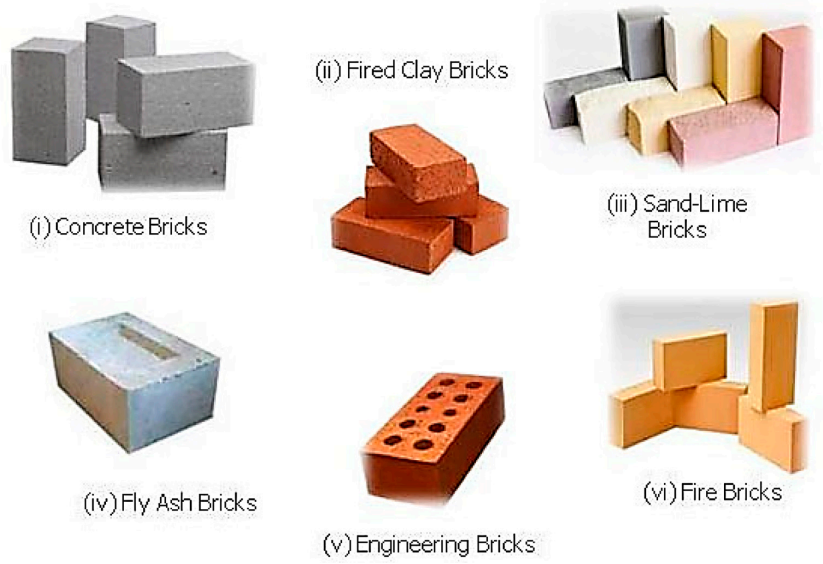


Figure 1. Different types of bricks based on raw materials. Source: Obtained from [3].

In recent years, sustainable materials have been used in construction, with biomass emerging as a promising alternative to develop new products. Biomass refers to the biodegradable portion of waste and residues from agricultural, forestry, livestock, and related activities. Biomass has been increasingly utilized in applications such as bio-composites and engineering materials, and it is recognized for its versatility and sustainability [4].

Agricultural residues like sugarcane bagasse, corn stalks, bamboo, coconut fibers, and coffee husks represent valuable sources of cellulose fibers, characterized by their low density, renewable nature, and competitive mechanical properties compared to synthetic materials like glass and carbon fibers [5–7]. The use of agricultural waste as reinforcement in concrete and mortar has shown promising results. Natural fibers, such as banana, bamboo, rice husk, and coffee husks, have been employed to enhance the mechanical performance of cementitious composites while aligning with environmental sustainability goals [8,9]. Coffee husks, a byproduct of coffee production, are highlighted for their renewable nature and significant cellulose content. However, improper disposal of coffee byproducts has led to environmental issues such as soil and water pollution, emphasizing the need for innovative applications of these materials in construction [10].

Due to the urban growth that cities are currently experiencing and the climatic consequences associated with this phenomenon, research interest has been generated in incorporating environmentally sustainable construction materials that present mechanical properties similar to the blocks currently used in the masonry construction sector. These materials seek to be a practical option for housing projects that promote environmental sustainability [11]. In recent years, researchers have recognized the potential of plant fibers for their cost-effective and efficient application in high-quality fiber-reinforced polymeric

materials in construction project production. Also, agricultural waste use in the construction industry is feasible due to the similar bricks elaborated from cement with mortar only. As a result, the use of cellulose fiber materials as reinforcement for concrete and mortar has been investigated, using different natural fibers such as banana [12], bamboo [13], rice husk [14], wheat and barley straw [15], coconut, sisal, jute, palm, and linseed [16]. Note that the recovery of this plant waste in the construction sector has several objectives: economic, technical, and environmental [17].

Related Research

Nakkeeran et al., 2023 [18] evaluated the use of raw rice husk, fly ash, and hydrated lime as replacements for fine aggregate and cement in the production of raw rice husk-concrete bricks using Response Surface Methodology (RSM). The model's accuracy was validated with Artificial Neural Networks (ANN), which demonstrated superior performance, capturing over 99.9% of the variability, compared to Multiple Linear Regression (MLR~R^2 > 91%). In the next year, the same author, Nakkeeran et al., 2024 [19], optimized the bio-brick properties made from agricultural waste, combining cement, fly ash, hydrated lime, and groundnut shell. Machine learning techniques (MLT) contributed to more efficient predictions of concrete's mechanical properties, saving time, labor, and resources in civil engineering. The authors employed MLT, including RSM and ANN, to predict the mechanical properties of the bio-bricks. The RSM models demonstrated high accuracy, with R^2 > 0.88, while the ANN models further improved prediction precision, achieving R^2 > 0.99. Also, Nakkeeran et al., 2024 [20] investigated the use of raw groundnut shell (RGGNS) as a fine aggregate in cement bricks, aiming to reuse agricultural waste in construction materials. The study applied RSM to optimize compressive strength, water absorption, and dry density. Results exposed that 40%, 50%, and 60% RGGNS improved the fine aggregate performance, with a maximum 28-day compressive strength of 6.1 N/mm^2, meeting technical standards with high (significant R^2).

Bernardi et al., 2014 [21] explored sand cementation into sandstone through microbial activity, a novel technology for creating sustainable building materials called bio-bricks, which were tested for compression wave velocity, unconfined compression strength, and calcite concentration. The results showed that bio-bricks exhibited higher P-wave velocity, stiffness, strength, and calcite content with increased bacterial treatment, with compressive strength values up to 1.0 MPa. Similarly, Yang et al., 2020 [22] investigated the use of microbially induced calcium carbonate precipitation (MICP) for cementing sand into bio-bricks, which was enhanced with additives like fibers, coarse sand, and clay. The results exposed that treatment time increased compressive strength, with fiber addition improving strength by 50–70%. The optimal clay content for strengthening was 2%, and coarse sand had a minimal impact on water absorption values from 10.16% to 17.89%, similar to traditional red bricks. Poornima et al., 2019 [23] explored the use of fly ash and bacterial solutions in manufacturing mud and fly ash bio-bricks, aiming for sustainability. The experiment was conducted at various pH levels (basic, acidic, neutral) and temperatures (4, 31, 60 °C), using both lab-grade and groundwater to determine the optimal mix proportion. The bio-bricks were tested for physical and mechanical properties such as compressive strength, water absorption, hardness, and shape. Results showed that the bio-bricks exhibited better properties than conventional bricks, making them suitable for temporary construction.

Shihui et al., 2021 [24] improved the bio-bricks resistance against accelerated water erosion using fiber reinforcement and multiple MICP treatments. The effects of synthetic fibers, coconut coir fibers, and multiple MICP treatments on bio-brick properties were investigated. Experimental results showed significant performance improvements, with

bio-bricks reinforced with 0.3% synthetic fibers showing an increase in flexural strength from 1185 kPa to 1210 kPa and strain from 1.0% to 2.3%. The optimal coconut coir fiber content was found to be 0.6–0.9%. Multiple MICP treatments significantly enhanced the resistance of bio-bricks to water erosion.

Considering the above research, the construction industry faces increasing environmental challenges, particularly due to the disposal of organic waste and the high carbon footprint associated with cement production. Also, almost all of the studies focus on bio-brick preparation and general mechanical testing without a structured methodology to optimize the mixture composition based on statistical analysis and optimization. Then, this study addresses these issues by exploring the potential of coffee husks, an agricultural byproduct, as reinforcement in bio-bricks. While prior research has highlighted the use of natural fibers like rice husks and coconut in construction, there remains a gap in understanding how coffee husks can be integrated into cementitious composites to achieve optimal mechanical properties. The innovation of this research lies in its application of RSM to optimize the proportions of cement, lignin, and bovine excreta in bio-bricks. By combining statistical modeling with practical experimentation, the study goes beyond conventional investigations of mechanical properties to propose a systematic approach for designing high-performance bio-bricks, also contributing to reducing carbon emissions and promoting sustainable practices in the built environment.

2. Materials and Methods

Sustainable materials in construction are an innovative line, allowing advances in the development of new elements applied to architecture and construction. For this research, biomaterial materials such as lignin and bovine excreta were characterized. Thus, the optimal mixture of mechanical properties for the elaboration of the biomaterial was evaluated. Figure 2 shows the flowchart step-by-step methodology employed in this study to evaluate bio-bricks' performance statistically considering different dosages used in experimental work.

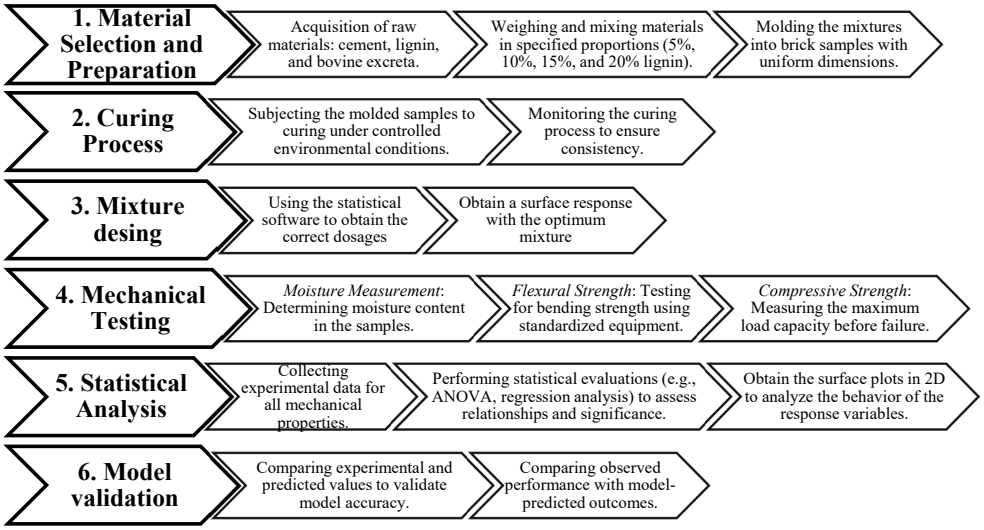


Figure 2. Step-by-step methodology employed.

2.1. Biomaterials Selection and Preparation

The bio-bricks in this study were formulated using cement, lignin (from coffee husks), and bovine excreta, and their physical, chemical, and morphological properties were thoroughly characterized. Cement exhibited a density of ~3.15 g/cm^3, ensuring adequate binding properties in the mixture. Lignin, obtained from coffee husks and sieved us-

ing mesh No 200 (see Figure 3a) [10], displayed a high carbon content (53.22 wt%) and potassium concentration (36.85 wt%), which enhance durability, microbial resistance, and binding capability within the bio-brick matrix. Bovine excreta, collected from a farm in Ocaña, Norte de Santander (Colombia), was treated with lime (CaO) to eliminate pathogens and reduce organic degradation (see Figure 3b). It exhibited a moisture content of 0.43–0.45% and a high oxygen content (35.21 wt%), providing lightweight properties, improved thermal insulation, and enhanced workability in the bio-brick formulation. The integration of these materials contributes to a sustainable and high-performance composite, balancing mechanical strength with environmental benefits.

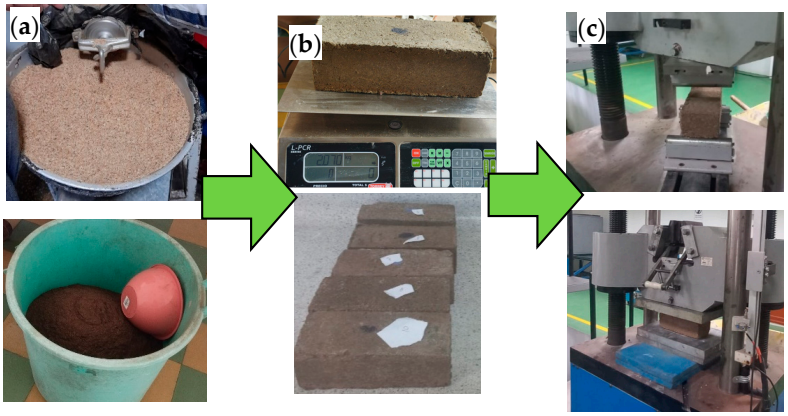


Figure 3. (**a**) Biomaterials preparation (lignin and excreta), (**b**) development of bio-bricks, and (**c**) experimental tests.

Sustainable architecture has demonstrated that alternative eco-materials, such as those used in this study, provide excellent mechanical properties while maintaining environmental and health safety standards. The integration of lignin and bovine excreta aligns with green construction principles, reducing reliance on conventional cement and promoting the adoption of bio-based composites in engineering, construction, and architecture [25].

The variables for the design mixture were selected based on the proportions of each material relative to the desired response, such as the mechanical strength of the biomaterial. These proportions include the establishment of minimum parameters accepted by the current Colombian standards (NTC). Different methods and techniques for biomaterial optimization were developed through practical experiences that enabled a step-by-step evaluation of the procedures adopted for the mixing experiments [26]. It is important to note that lignin added to concrete significantly improves its compressive strength due to its pozzolanic properties when used as an additive. However, this project utilized lignin in its natural state, avoiding calcination in industrial furnaces, as such processes increase greenhouse gas emissions and contribute to global warming [27].

Additionally, reducing the cement content in the mixture was considered, considering the current excessive use of bricks. This behavior contradicts sustainability principles due to the high environmental impact of CO_2 emissions during production. Both lignin and bovine excreta were incorporated to mitigate CO_2 emissions because, thanks to their cohesive and plastic properties, they have been historically used in traditional construction techniques, such as baroque.

2.2. Procedure for Mixture Design

The mix design aims to determine the influence of each component on the final product while providing analytical tools to model these relationships and optimize component proportions, ensuring the desired material properties. The process begins with the selection of key variables, including general-purpose cement (binder), bovine excreta, and lignin

(fillers), with their proportions adjusted according to the results of physical and mechanical tests established by Colombian technical standards (NTC-4205) [28].

The experimental preparation of the biomaterial mixture and homogenization procedure shown in Figure 4 ensures uniformity and consistency across all samples. It begins with sample collection (Figure 4a) and material preparation through crushing and sieving (Figure 4b), followed by thorough homogenization to ensure an even distribution of components (Figure 4c). The optimized mixture was combined with 390 g of water to achieve the required consistency (Figure 4d) and then compacted in successive layers to enhance stability and uniformity during demolding, minimizing excessive porosity in the final biomaterial (Figure 4e). Note that the drying process was developed by being exposed to the sun's rays.

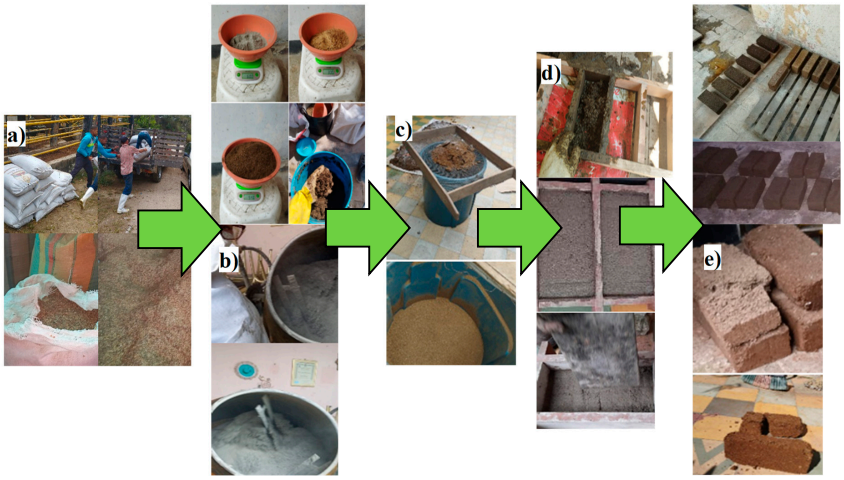


Figure 4. Biomaterial fabrication process. (**a**) Collection, (**b**) material processing, (**c**) homogenization, (**d**) molding, (**e**) natural drying.

For this research, the mixture design process was carried out considering the following three main steps: (1) Identification of Influences: One of the fundamental purposes of mix design is to determine which components or their interactions significantly impact one or more responses of interest. This analysis identifies key factors affecting the product's properties. According to Abadou et al., 2020 [29], statistical studies enable the adjustment of mathematical models to express response variables as functions of the mixture component proportions. Additionally, statistical modeling of mixtures ensures material optimization within the design process. (2) Response Modeling: It is essential to model the responses of interest concerning the mixture of components' proportions based on the mechanical property values. This analysis involves the development of mathematical models that describe the relationship between input variables (cement proportions, lignin, and bovine excreta) and observed responses or output variables (compressive strength, flexural strength, and moisture absorption) to optimize and obtain the appropriate mixture [30]. And (3) Optimal Proportion Determination: Using some models, the mix design enables the optimal proportions determination for each component to achieve the desired mechanical properties in the final formulation and ensure the product meets quality standards and requirements for biomaterial production in the construction sector.

Furthermore, Hwang et al., 2015 [31] emphasized the importance of studying the feasibility of using an appropriate mix design method to achieve optimal physical and mechanical performance. According to Colombian regulations, such as NTC-4205, clay ceramic bricks, widely used in the construction and engineering industries, must meet these performance standards. This procedure involves carefully planned experiments in which the proportions of the components vary systematically.

2.3. Optimization of Composite Biomaterials Using Statistics

Statistical determination of the optimal biomaterial mixture is essential to define specific technical data for a material or product (bio-brick). In this context, Statgraphics-15 software is a valuable tool for probabilistic design and statistical data analysis through the execution of Analysis of Variance (ANOVA) to identify significant differences between various biomaterial formulations.

The bio-brick composition, made from lignin, bovine excreta, and cement, was evaluated using a Design of Experiments (DOE) approach and a Response Surface Methodology (RSM) model to analyze the material's compression, flexural strength, and moisture properties. DOE is particularly suitable because it allows determining how the proportions of the three ingredients/variables/components (lignin, bovine excreta, and cement) influence the final bio-brick mechanical properties. In this design, the input variables are the proportions of each material, while the response variables (mechanical properties) such as compression, flexion, and moisture absorption. It is important to note that biological compounds exhibit a wide range of distinct and significant mechanical properties. However, biomaterial architectures cannot always be considered optimal designs for engineering applications, as biomaterials continuously evolve for multiple functions beyond supporting external loads [32].

2.3.1. Optimal Mix Design

Initially, mix designs were conducted to evaluate variations in cement, lignin, and bovine excreta proportions, ranging from 0% to 100% for each material. Different mixing methodologies were applied, assessing each component's physical properties and their specific interactions. During the initial designs, it was observed that the amount of organic material, particularly lignin and bovine excreta, and the mesh size used in bio-brick preparation significantly influenced the compound cohesion. High proportions of organic material reduced the cohesion and strength of the bio-brick due to its lower density and fibrous nature compared to cement. The mesh size also affected the components' distribution and uniformity, impacting the bio-brick's structural integrity after drying [33].

According to Goos et al., 2016 [34], mix design involves the study of the mixture's components' proportions. Depending on the nature of these factors, specific regression models and experimental designs are required. Experimental mix designs aim to predict the response or optimal dosage for all possible formulations, enabling analysis from an initial dosage and creating variations to find the optimal response through probabilistic design to improve the biomaterial. An allowable range for each variable was established based on trial-and-error testing with replicates to analyze biomaterial mixture response variables. The collected data were used to approximate parameters relating the strength to the proportion of each component in the mixture. For example, strength was a linear function of cement, lignin, and bovine excreta proportion. Goel et al. (2017) [35] recommend varying the mix proportions by 5%, 10%, 15%, and 20%. In this study, the lignin and bovine excreta content were modified to evaluate the biomaterial's compressive strength, flexural strength, water absorption, and apparent density, adhering to Colombian standards such as NTC-4205 [36] and ASTM-C76 [37].

Mix analysis began with lignin dosages of 5%, 10%, 15%, and 20%. Cement content remained constant at 59–64% of the total mixture, with bovine excreta completing 100%. The variations aimed to determine the optimal compressive strength for the bio-brick before observing a significant decrease in performance. Mechanical properties were analyzed, focusing on cohesion and the strength of different material combinations. This evaluation process identified the most efficient and resistant mixture by carefully adjusting the proportions of lignin, cement, and bovine excreta to achieve an ideal balance between

sustainability and structural performance. Through experimentation, precise adjustments were made to material proportions and mixing parameters. Compacting and strength tests were conducted to evaluate the mix's consistency and stability under different conditions. These tests included variations in compaction pressure and material dosages, resulting in a mixture with the appropriate consistency.

Table 1 shows the mixed design based on the considerations of workability and handling for the different materials. This design incorporates three main components: cement, lignin, and bovine excreta, for a total constant weight of 1500 g. The results obtained are summarized in Table 1, and the results agreed with those reported by different authors who have incorporated biomaterials into the composition of structural bricks [3,38,39].

Table 1. Mixture design and response variable results.

%	Experimental Variables			Response Variables		
	Cement (g)	Lignin (g)	Bovine Excreta (g)	Moisture (%)	Flexure (MPa)	Compression (MPa)
5	959	75	465	0.375 ± 0.1129	0.510 ± 0.0576	1.544 ± 0.0530
10		150	390	0.435 ± 0.0362	0.550 ± 0.1117	1.619 ± 0.0443
15		224	314	0.430 ± 0.0669	0.568 ± 0.0662	1.700 ± 0.0769
20		300	240	0.445 ± 0.0596	0.610 ± 0.0358	1.670 ± 0.0554

Although the analyzed factors (components) are continuous by nature, four primary proportions were defined for lignin and bovine excreta, while the cement amount was kept constant at 959 g. Note that six replicates were performed for each proportion, resulting in a total of 24 experimental points. This experimental design identified how variations in the component proportions affect the physical and mechanical properties of the composite material, aiming to develop a sustainable bio-brick.

In mixture design experiments, a specific variant is established where the factors (components) represent mixture proportions, and their sum must remain constant. The following statistical considerations were considered:

- **Factors:** Cement, lignin, and bovine excreta (proportions);
- **Constraint:** The sum of the factors is constant (1500 g);
- **Response variables:** Moisture content, flexural strength, and compressive strength. **Note:** Density and morphological aspects were not considered due to the low dispersion observed in the experimental results.

According to Oluwatoyin et al., 2023 [39], global cement productions contribute to 5% of carbon emissions and propose the use of agricultural waste as a sustainable alternative to cement in building materials. Materials such as Sugarcane Bagasse Ash (SCBA), palm leaf ash, and Rice Husk Ash (RHA) are identified as partial cement substitutions. Incorporating these agricultural wastes into materials like bricks, concrete, and pavements can enhance their strength by up to 65%, reduce unit weight by 25%, and improve compressive strength, water resistance, and bulk density. Also, 50% of cement can be replaced with SCBA, and hybrid combinations of rice husk ash, agricultural olive waste, and other agricultural wastes can completely replace cement. This approach could aid in reducing global emissions while providing cost-effective, sustainable building materials.

2.3.2. ANOVA Analysis

The analysis of variance (ANOVA) was performed using Statgraphics-15 software with the aim of determining the statistical significance of the effect of the studied factors, such as the amount of lignin and bovine excreta, on the composite material mechanical properties, specifically the flexural and compressive strength. This analysis allowed for the evaluation of the main effects of each factor and the interaction between them, iden-

tifying which ones significantly contribute to the variability of the responses. ANOVA provides key information to either reject or confirm hypotheses related to the independent variables' influence on the analyzed properties, facilitating an accurate interpretation of the experimental results [40].

The results obtained from these experiments are used to adjust the model and make predictions about the bio-brick properties based on the material proportions. This approach allows for bio-bricks properties analysis and optimizes its composition to achieve the best performance in terms of the response variables obtained from testing. Additionally, for bio-brick with appropriate dosages, the response surface analysis facilitates the identification of the most suitable mixing ranges, which can improve material quality, reduce costs, or increase the efficiency of the manufacturing process. Finally, the statistical aspects described below are considered:

- The Variance Inflation Factor (V.I.F.): A VIF value equal to 1.0 confirms that the experimental design is orthogonal, meaning there are no issues with confounding effects, which strengthens the validity and estimates accuracy. If the standard error of the effects is low, it reflects good control over the experimental error. In conclusion, lignin is the key factor for improving the compressive strength of the bio-brick, while the other factors have lesser contributions, and no significant interactions are observed between them. Furthermore, the largest variance inflation factor (V.I.F.), being equal to 1.0, indicates a perfect orthogonal design, where all factors would have a V.I.F. of 1.0. Factors of 10 or more are usually interpreted as indicators of severe confounding between effects;
- Analysis of Variance (ANOVA): It is a statistical method used to compare the means of three or more groups and determine whether significant differences exist between them. ANOVA is based on the ratio calculation called the F-statistic, which assesses whether the differences between the means are greater than what would be expected due to chance. If the F value is significant, it is concluded that at least one of the means is different;
- R^2 (Coefficient of Determination): It is a measure that indicates the proportion of total variability in the data explained by the model. Its value ranges from 0 to 1. If $R^2 = 1$, the model explains all the variability in the data, but if R^2 is close to 0, this explains that the model presents a little variability. In simple terms, R^2 reflects how well the model fits the data;
- Adjusted R^2: It is a modified version of R^2 that adjusts the value for the number of independent variables in the model. It is useful because R^2 can increase simply by adding more variables, even if they are not relevant. Adjusted R^2 penalizes models that include unnecessary variables and only increases if an additional variable significantly improves the model;
- Standard Error of Estimation (SEE): It is a measure that quantifies the actual data points dispersion around the regression line (or predictive model). It indicates, on average, how far the observed values are from the values predicted by the model;
- Mean Absolute Error (MAE): A metric measures the absolute error average between the observed values and the predicted values by the model. It helps evaluate a model's performance, as it only considers the error's magnitude, ignoring its direction (positive or negative);
- Durbin–Watson Statistic (DW): This test is used in regression analysis to detect the autocorrelation presence in the residuals of a model. Autocorrelation occurs when the residuals are not independent of each other but are correlated based on time or the order of the observations.

2.3.3. Response Surface Methodology (RSM)

RSM was employed as a robust tool to model and analyze the relationships between the studied factors and the mechanical properties of the composite material (bio-brick). This statistical approach facilitates the mathematical model's development, which describes the behavior of response variables based on factor levels. RSM provides valuable insights into system behavior by enabling the visualization of trends through two-dimensional (2D) or three-dimensional (3D) contour plots. Specifically, it was used to identify the optimal proportions of lignin and coiled excreta that maximize the flexural and compressive strength of the bio-brick [41]. As contour curves illustrate the evolution of specific wear rates, color gradations—green (low), yellow (medium), and dark red (high)—are used to represent wear severity. This visualization aids in identifying optimal conditions and understanding the relationship between wear behavior and contributing factors. These models offer insights into key values, such as R^2 and adjusted R^2, describing the behavior of response variables to minimize or maximize experimental outcomes [40].

3. Results and Discussions

The bio-brick mixture optimization was based on the careful selection of three primary components: cement, lignin, and bovine excreta. The influence of these materials on mechanical properties such as compressive strength, flexural strength, and moisture absorption was assessed. The mixture design followed Colombian technical standards (NTC-4205), ensuring compliance with structural performance requirements.

The RSM approach was employed to evaluate the varying proportions effects of the three components systematically. A full factorial experimental design was implemented, where cement content varied from 59% to 64% of the total mixture, and lignin content ranged between 5% and 20%. Bovine excreta made up the remaining percentage to maintain a total of 100%. Notice that the experimental matrix consisted of 24 different mix combinations, with six replicates per proportion, to guarantee statistical accuracy.

3.1. Optimum Mixture Design

The optimal bio-brick composition was determined to be 959 g cement, 224 g lignin, and 314 g bovine excreta (follows a ratio of 4.3:1:1.4, which consists of cement (64%), lignin (15%), and bovine excreta (21%)). Note that this formulation achieved a compressive strength of ~1.7 MPa, meeting the minimum requirements for sustainable masonry applications. The mechanical properties were validated through experimental tests, confirming that this mixture offers an optimal balance between strength, workability, and environmental sustainability. This mixture has a density of 0.893 g/cm^3, a base of 10 cm, a height of 24 cm, and a width of 7 cm (See Figure 1).

Figure 5 shows cement, lignin, and bovine excreta compositions, which were used to obtain the bio-bricks studied from the optimal mixture (15%). The micrographs reveal a heterogeneous but well-integrated distribution of mineral particles and organic fibers. The cement and lignin particles exhibit surface roughness and close contact with the fibrous components, which favor cohesion and stress transfer within the matrix. The fibers from the coffee husk (lignin) and excreta act as reinforcement, providing dimensional stability and improving the material's flexural strength. This structural balance suggests a synergistic interaction between the components that contributes to the mechanical properties of the bio-brick.Note that the optimal mixture selection is based on a ternary diagram shown in Figure 6, which displays the estimated response surface contours for the compressive strength of the bio-brick.

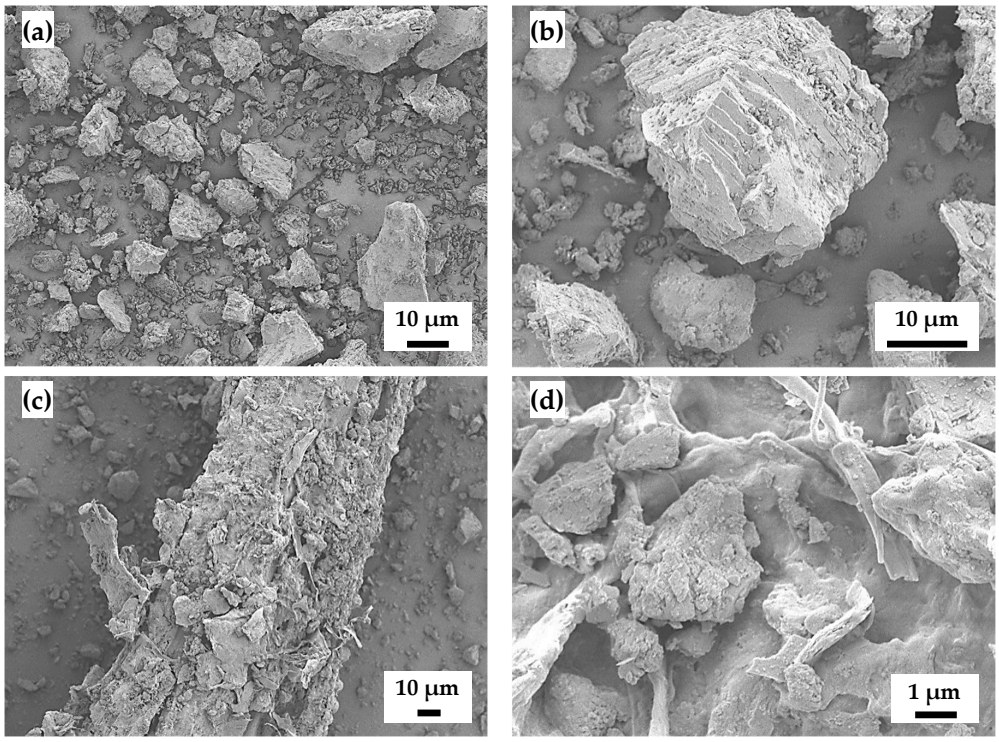


Figure 5. SEM-EDS micrographs obtained for (**a**) optimal mixture of the bio-brick, (**b**) cement, (**c**) lignin, and (**d**) excreta with lime. Note: SEM-EDS were performed using the JEOL JCM-6000 equipment at 15 kV.

Likewise, the EDS spectra are shown in Table 2, revealing different compositional differences among cement, lignin, and excreta, which highlight their varied contributions to bio-brick functionality. Cement is dominated by calcium (Ca, 79.39 Wt%), emphasizing its role in providing structural strength and durability through the formation of calcium silicate hydrates during curing. Lignin exhibits high potassium (K, 36.85 Wt%), consistent with its plant-based origin; potassium can improve resistance to microbial degradation and enhance chemical stability. Excreta is rich in carbon (C, 53.22 Wt%) and oxygen (O, 35.21 Wt%), indicative of organic matter, contributing to lightweight bio-bricks with better thermal insulation. Trace elements such as magnesium (Mg) and silicon (Si) in all materials reinforce the optimal mixture, improving compressive strength and thermal stability. Phosphorus (P) and sulfur (S), found in lignin and excreta, can promote microbial activity, potentially enabling self-healing properties in bio-bricks by facilitating the repair of microcracks. Copper (Cu) and zinc (Zn), present in excreta, could add antibacterial properties, enhancing resistance to biofouling in humid environments.

These compositional differences align with theoretical expectations of inorganic-dominant materials (cement) versus organic and nutrient-rich materials (lignin and excreta). Bio-bricks can be optimized for sustainability, improved mechanical performance, and enhanced environmental resilience, making them a viable eco-friendly alternative in construction [42].

On the other hand, Figure 6 shows the ternary diagram of the elements that make up the optimal mixture (cement, lignin, and bovine excreta), considering the colors in the graph (Blue: Low compressive values (~1.5 MPa) and Red: High compressive values (~1.74 MPa)). Since the component's proportions are distributed along each axis, always summing to 100%, the following observations were made: (1) The highest values (in yellow, orange, and red) are concentrated in regions with a high cement proportion and a low husk proportion. This behavior suggests that cement is the main contributor to mechanical strength. (2) The lowest values (in blue) are found in the regions where

husk and excreta predominate, reflecting lower structural cohesion. (3) The green zones represent an intermediate balance in strength (~1.6 MPa), where the proportions of all three components are more balanced. This mix may suggest an optimal combination of lignin, excreta, and cement to achieve good strength without relying only on cement. (4) Finally, increasing the cement proportion directly improves compressive strength. At the same time, lignin and excreta contribute to the material sustainability, but their excess significantly reduces strength. Therefore, it should be limited to proportions that do not compromise the structural and sustainable functionality of the material dosages used in the experimental design.

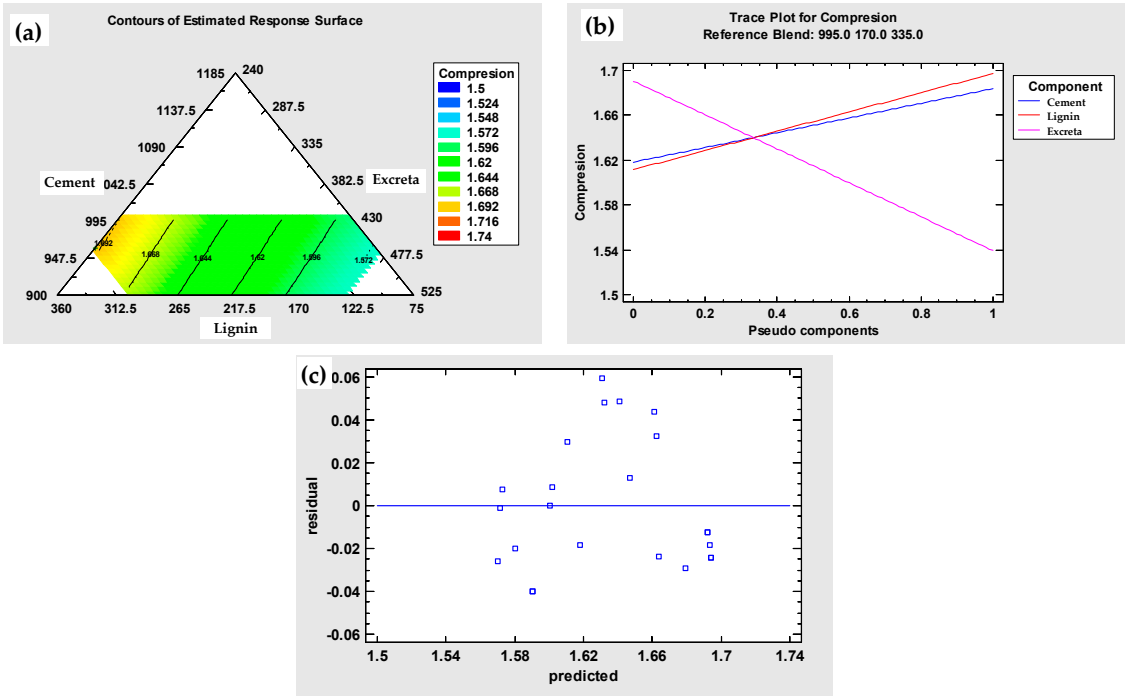


Figure 6. Optimal mixture design, (**a**) response surface, (**b**) trace plot, and (**c**) residuals.

Table 2. EDS results of the optimal mixture for the bio-brick.

Element	Cement		Lignin		Excreta	
	Wt %	At %	Wt %	At %	Wt %	At %
C K	---	---	---	---	53.22	63.60
O K	---	---	---	---	35.21	31.59
Na K	---	---	0.71	1.23	0.73	0.46
Mg K	0.61	0.95	3.86	6.28	0.29	0.17
Al K	3.28	4.61	1.85	2.71	0.17	0.09
Si K	10.09	13.65	3.76	5.30	3.93	2.01
P K	---	---	4.11	5.24	0.37	0.17
S K	1.51	1.78	4.01	4.94	0.26	0.12
Cl K	---	---	0.99	1.10	0.38	0.15
K K	1.02	0.99	36.85	37.25	2.08	0.76
Ca K	79.39	75.30	23.42	23.09	0.90	0.32
Mn K	---	---	---	---	0.05	0.01
Fe K	2.98	2.03	1.83	1.29	0.15	0.04
Ni K	---	---	---	---	0.20	0.05
Cu K	1.14	0.68	18.60	11.57	1.14	0.26
Zn K	---	---	---	---	0.91	0.20
Total	100	100	100	100	100	100

Furthermore, the statistical analysis established that the model providing the best statistical fit was the linear model. The adjusted R^2 statistic, which is more suitable for comparing models with different numbers of independent variables, is 65.4208%. The estimate's standard error shows that the residuals' standard deviation is 0.0317772. The mean absolute error (MAE) of 0.0252615 is the average residual value, with an F-Ratio equal to 21.81. The Durbin–Watson (DW) statistics evaluate the residuals to determine if there is any significant correlation based on the order in which they appear in the data file. Since the *p*-value is greater than 5.0%, there is no evidence of autocorrelation in the residuals at the 5.0% significance level. Considering the above, the response surface for compressive strength (Figure 7) in the ternary diagram is dominated by Equation (1) at a 95% confidence level.

$$\text{Compression} = (1.68369 \times \text{Cement}) + (1.69721 \times \text{Lignin}) + (1.53938 \times \text{Bovine Excreta}) \quad (1)$$

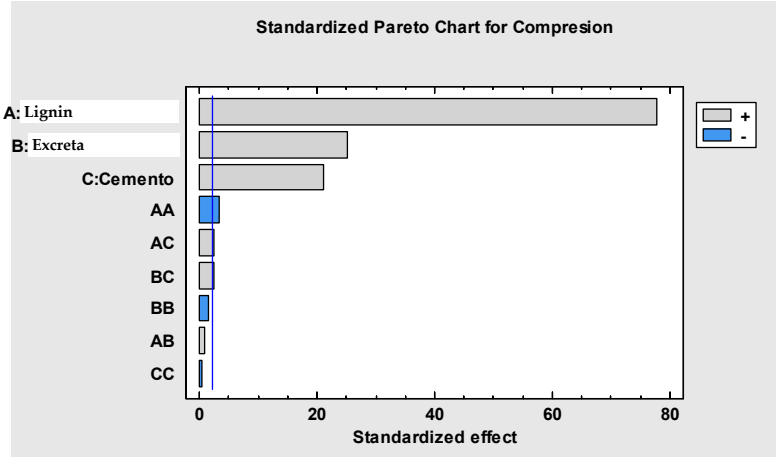


Figure 7. Pareto chart for compressive strength.

3.2. Statistical Analysis for the Response Variables

3.2.1. Compression Resistance Statistical Analysis

Estimated Effects on Compression Resistance

According to the estimated effects for compressive strength (MPa) in Table 3 and Figure 7, it is observed that lignin (Factor A) has the greatest positive impact, with a value of 0.1272 MPa, suggesting that this component is the most influential in increasing the bio-brick compressive strength. On the other hand, bovine excreta (Factor B) and cement (Factor C) also show positive effects, although of smaller magnitude, with values of 0.0411 MPa and 0.0344 MPa, respectively. This behavior indicates that, while they contribute to the increase in strength, their influence is significantly lower than lignin. Regarding the quadratic effects (AA, BB, CC) and the interactions between factors (AB, AC, BC), it can be noted that the values are small and not very relevant, suggesting that there are no significant synergistic effects between the components and that the individual effects mainly dominate the strength behavior.

Lignin plays a crucial role in bio-bricks development by enhancing their mechanical properties and durability while maintaining sustainability. Similar to its application in lignin-based polyurethane foams (LPUFs), because it improves fire resistance, moisture resistance, and structural integrity, lignin can also be incorporated into bio-bricks to increase compressive strength and water absorption reduction. The lignin use in bio-bricks helps mitigate brittleness, a common issue in eco-friendly construction materials, by acting as a natural binder that enhances cohesion between particles [43]. Additionally, the improved

processability observed in LPUFs with co-solvents and plasticizers suggests similar strategies that could optimize lignin's performance in bio-bricks and make them more flexible.

Table 3. Estimated effects by compression (MPa).

Effect	Estimate	Stnd. Error	V.I.F.
Average	1.6218	0.00176655	---
A: Lignin	0.1272	0.00163551	1.0
B: Bovine Excreta	0.0411	0.00163551	
C: Cement	0.0344	0.00163551	
AA	−0.0094	0.00283279	
AB	0.0017	0.00200308	
AC	0.0050	0.00200308	
BB	−0.0044	0.00283279	
BC	0.0050	0.00200308	
CC	−0.0011	0.00283279	

ANOVA Compression Resistance

According to the Analysis of Variance (ANOVA) for compressive strength (MPa), it can be argued that lignin (Factor A) has a highly significant effect on compressive strength, with an F-Ratio value of 6050.88 and a *p*-value of 0.0001, confirming that lignin is the most influential factor in the model. Bovine excreta (Factor B) and cement (Factor C) are also statistically significant, though with a lesser impact, with F-Ratios of 631.85 and 443.54, respectively, and both with *p*-values of 0.0001 (see Table 4). The effects of interactions are less relevant; for instance, the quadratic effect of AA (lignin) has an F-Ratio of 11.12 and a *p*-value of 0.0039, indicating statistical significance, although of lesser magnitude compared to the linear effects. The interactions between AC (lignin and cement) and BC (bovine excreta and cement) are also significant, with F-Ratios of 6.23 and *p*-values of 0.0231. However, other interactions, such as AB and BB and CC's quadratic effects, are insignificant, with *p*-values greater than 0.05 (see Table 4).

Table 4. Analysis of variance for compression.

Source	Sum of Squares	DOF	Mean Square	F-Ratio	*p*-Value
A: Lignin	0.0728347	1	0.0728347	6050.88	0.0001
B: Bovine Excreta	0.00760556	1	0.0076055	631.85	0.0001
C: Cement	0.00533889	1	0.0053389	443.54	0.0001
AA	0.000133796	1	0.0001338	11.12	0.0039
AB	0.00000833333	1	0.0000083	0.69	0.4169
AC	0.000075	1	0.000075	6.23	0.0231
BB	0.0000296296	1	0.0000296	2.46	0.1351
BC	0.000075	1	0.000075	6.23	0.0231
CC	0.00000185185	1	0.00000185	0.15	0.6998
Total error	0.00020463	17	0.0000120	---	---
Total (corr.)	0.0863074	26	---	---	---
R^2	99.76%				
R^2 ajusted	99.63%				
MAE	0.002091				
Durbin–Watson	1.8635 (p = 0.1328)				

Where DOF stands for Degrees of Freedom.

On the other hand, the coefficient of determination (R^2) is 99.76%, which implies that the model explains almost all the variability in compressive strength. The adjusted R^2 is 99.63%, confirming a good fit of the model. The standard error of the estimate is low (0.00346944), as is the mean absolute error (MAE) of 0.00209191, reflecting high precision and good fit to the data. The Durbin–Watson statistics have a value of 1.8635 with a *p*-value of 0.1328, indicating no significant autocorrelation in the residuals, validating the error's independence and the model robustness. It is concluded that lignin is the most important factor in compressive strength, followed by bovine excreta and cement. Although some interactions like AC and BC have significant influence, the model is highly precise and reliable for describing and predicting the compressive strength of the bio-brick.

Finally, in the case of compressive strength, six effects have *p*-values smaller than 0.05, indicating that they are significantly different from zero at the 95.0% confidence level. The Durbin–Watson (DW) statistic tests the residuals to determine if there is any significant correlation based on the order in which they appear in the data file. Since the *p*-value is greater than 5.0%, there is no evidence of serial autocorrelation in the residuals at the 5.0% significance level.

Regression Model Obtained to Compression Resistance

The regression model obtained for compressive strength (MPa) demonstrates how each factor and its interactions affect this property. The adjusted equation includes a constant term of 1.12107, which represents the reference strength when the factors are set to zero. In linear terms, cement (C) has the highest positive coefficient of 0.000526667, indicating that its increase in the mixture significantly boosts the compressive strength. Lignin (A) also has a positive effect (although smaller), with a coefficient of 0.000259918. In contrast, the bovine excreta (B) shows a negative effect with a value of -0.000128066, suggesting that its increase slightly decreases the compressive strength of the bio-bricks.

The quadratic effects of AA, BB, and CC are small and negative, with values of -3.73114×10^{-7}, -1.75583×10^{-7}, and -2.22222×10^{-7}, respectively, indicating that an excess of any of the components could reduce the strength due to non-linear behaviors that occur during the mixing of the biomaterials. The interactions AC and BC have a value of 4.44444×10^{-7}, showing a slight synergy between these materials that enhances strength, while the interaction AB is insignificant with a value of 6.58436×10^{-8} (see Table 5).

Table 5. Regression coefficients for compressive strength.

Coefficients	Estimate
Constant	1.12107
A: Lignin	0.000259918
B: Bovine Excreta	-0.000128066
C: Cement	0.000526667
AA	-3.73114×10^{-7}
AB	6.58436×10^{-8}
AC	4.44444×10^{-7}
BB	-1.75583×10^{-7}
BC	4.44444×10^{-7}
CC	-2.22222×10^{-7}

In conclusion, cement has the greatest positive impact on compressive strength, followed by lignin, while bovine excreta has a negative effect in linear terms. The quadratic

effects suggest that an excessive component dosage is counterproductive, and the interactions between AC and BC indicate combinations with slightly favorable effects.

Equation (2) presents the regression model adjusted to the experimental data statistically analyzed for compressive strength, where EV represents bovine excreta, C represents cement, and CC represents lignin. Additionally, Figure 8 shows the estimated response surface for compressive strength as a function of the experimental variables analyzed.

$$\begin{aligned}\text{Compression} = \; & (1.12107 + 0.000259918) \times (\text{CC} - 0.000128066) \times (\text{EV} + 0.000526667) \\ & \times (\text{C} - 3.73114 \times 10^{-7}) \times \left(\text{CC}^2 + 6.58436 \times 10^{-8}\right) \times \text{CC} \times (\text{EV} + 4.44444 \times 10^{-7}) \\ & \times \text{CC} \times (\text{C} - 1.75583 \times 10^{-7}) \times \left(\text{EV}^2 + 4.44444 \times 10^{-7}\right) \times \text{EV} \times \text{C} \\ & - \left(2.22222 \times 10^{-7} \times \text{C}^2\right)\end{aligned} \tag{2}$$

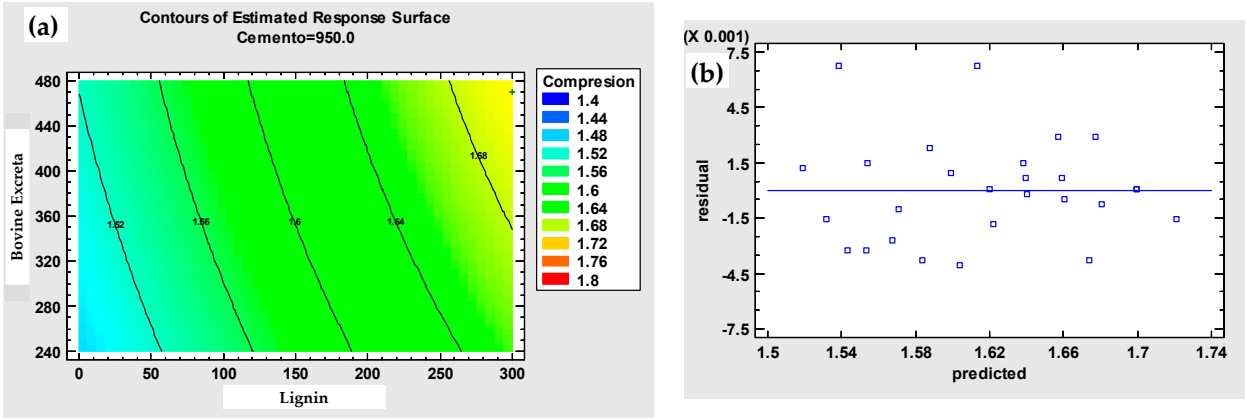


Figure 8. Compression strength, (**a**) response surface, and (**b**) residuals.

Steepest Ascent Path for Compression Strength

The steepest ascent path for compression strength, as observed in Table 6, describes the fastest change in the interest property (compression) when varying the experimental factors (lignin, bovine excreta, and cement) in a controlled manner. This path indicates how to adjust the factors to maximize the compression in the bio-brick and achieve optimal strength. In this case, the path was determined by modifying the lignin weight in increments of 1.0 g, while the other factors (bovine excreta and cement) remained relatively constant.

Table 6. Steepest ascent path for compression.

Lignin (gr)	Excreta (gr)	Cement (gr)	Estimated Compression (MPa)
187.5	352.500	950.00	1.62185
188.5	352.823	950.12	1.62242
189.5	353.147	950.241	1.62318
190.5	353.471	950.362	1.62385
191.5	353.796	950.484	1.62451
192.5	354.121	950.606	1.62518

From Table 6, it is possible to observe that as the lignin amount increases from 187.5 g to 192.5 g, the predicted compression also shows a slight increase (from 1.62185 MPa to 1.62518 MPa). This behavior suggests that adding more lignin gradually increases the compression strength within the current factor ranges. The steepest ascent path analysis indicates the most efficient path to improve the interest property without making drastic or

unnecessary changes to the other factors. In this case, the model shows that small variations in lignin content within the evaluated range positively impact compression. If the goal is to continue increasing compression, this path suggests that further increasing the amount of lignin could be an effective strategy. The steepest ascent (or descent) path was observed. This behavior is the path from the center of the current experimental region along which the estimated response changes progressively for the smallest change in the experimental factors. It indicates good locations to run additional experiments if the goal is to increase or decrease compression. Currently, six points are generated by changing the lignin in increments of 1.0 g.

3.2.2. Flexural Strength Statistical Analysis

Estimated Effects for Flexural Strength

According to the estimated effects for flexural strength (MPa), shown in Table 7 and Figure 9, it is observed that lignin (Factor A) has the greatest positive impact, with a value of 0.0817 MPa, suggesting that this factor is crucial for improving the flexural strength of the bio-brick. On the other hand, bovine excreta and cement have smaller effects, 0.025 MPa and 0.02 MPa, respectively. This behavior indicates that, although all three factors influence flexural strength, lignin is the most determining factor. Additionally, the interactions between factors, such as AA, AB, and AC, show small effects, and some are not statistically significant, implying that combinations between certain factors do not significantly influence flexural strength.

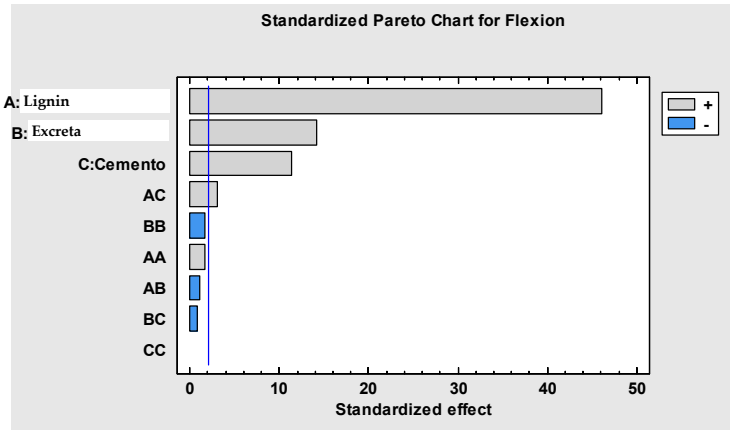


Figure 9. Standardized Pareto chart for flexural resistance.

Table 7. Estimated effects for flexural strength (MPa).

Effect	Estimate	Stnd. Error	V.I.F.
Average	0.5583	0.00191149	---
A: Lignin	0.0816	0.00176969	
B: Bovine Excreta	0.025	0.00176969	
C: Cement	0.020	0.00176969	
AA	0.005	0.00306520	
AB	−0.0025	0.00216742	1.0
AC	0.0066	0.00216742	
BB	−0.005	0.00306520	
BC	−0.0016	0.00216742	
CC	0.0000	0.00306520	

ANOVA Flexural Strength

According to the Analysis of Variance (ANOVA) for flexural strength (MPa), lignin has a significant impact, as its F-ratio value (2129.58) is high for a low *p*-value of 0.0001. On the other hand, some interactions, such as AB and BC, have *p*-values greater than 0.05, indicating that these interactions do not significantly impact the flexural strength of the bio-brick. Both bovine excreta and cement also have significant effects, with F-ratios of 199.57 and 127.72, respectively, although they are of smaller magnitude compared to lignin. Moreover, the CC interaction is insignificant, suggesting that quadratic interactions between these factors do not significantly affect flexural strength (See Table 8).

Table 8. Analysis of variance for flexural strength.

Source	Sum of Squares	DOF	Mean Square	F-Ratio	*p*-Value
A: Lignin	0.0300125	1	0.0300125	2129.58	0.0001
B: Bovine Excreta	0.0028125	1	0.0028125	199.57	0.0001
C: Cement	0.0018	1	0.0018	127.72	0.0001
AA	0.0000375	1	0.0000375	2.66	0.1212
AB	0.00001875	1	0.00001875	1.33	0.2647
AC	0.000133333	1	0.000133333	9.46	0.0069
BB	0.0000375	1	0.0000375	2.66	0.1212
BC	0.00000833333	1	0.000008333	0.59	0.4525
CC	0.0	1	0.0	0.00	1.0000
Total error	0.000239583	17	0.0000140931	---	---
Total (corr.)	0.0351	26	---	---	---
R^2	99.31%				
R^2 ajusted	98.95%				
MAE	0.00246914				
Durbin–Watson	0.897101 (p = 0.0001)				

Where DOF means Degrees of Freedom.

On the other hand, interactions between factors AB, BB, and BC are insignificant, as their *p*-values are greater than 0.05. Specifically, AB and AC are relevant but have smaller effects than the main factors. The CC interaction has a *p*-value of 1.0000, indicating no significant effect on flexural strength. This behavior suggests that, although interactions may influence, most of the significant effects come from the main factors, especially lignin, as observed in Table 8.

The coefficient of determination (R^2) of 99.31% indicates that the model explains almost all the variability observed in the flexural strength of the bio-brick. The adjusted R^2 is 98.95%, confirming a good fit of the model. The standard estimate error is low (0.00375408), and the mean absolute error (MAE) is 0.00246914, reflecting high precision and a good fit of the statistical model to the data. However, the Durbin–Watson statistics have a value of 0.8971 with a *p*-value of 0.0001, indicating significant autocorrelation in the residuals, meaning that the model errors are not completely independent and there might be unaccounted patterns in the data (see Table 8). As observed, lignin is the most important factor in flexural strength, followed by bovine excreta and cement, which also shows that the AC interaction provides the best fit to optimize the flexural strength of the bio-brick under the experimental conditions studied.

Finally, in the case of flexural strength, four effects have *p*-values less than 0.05, indicating that the effects are significantly different from zero at the 95.0% confidence level. The Durbin–Watson (DW) statistic tests the residuals to determine if there is any significant correlation based on the order in which they appear in the data file. Since the *p*-value is less than 5.0%, there is an indication of a possible serial correlation at the 5.0% significance level.

Regression Model Obtained for Flexural Strength

The regression model obtained for flexural strength (MPa) shows how each factor and its combinations affect this property. The adjusted equation includes a constant term of 0.292928, representing the reference strength when the factors are zero. In linear terms, bovine excreta (B) has the highest positive coefficient of 0.00040963, indicating that its increase in the mix significantly increases flexural strength. Cement (C) also has a positive effect with a coefficient of 0.000141111, although its impact is smaller than excreta. On the other hand, lignin (A) has a negative effect with a value of −0.000239259, suggesting that its increase in the mix decreases flexural strength.

Regarding quadratic effects, AA has a positive value of 1.97531×10^{-7}, suggesting that a moderate increase in cascarilla could have beneficial effects within certain ranges. Both BB and CC have values of -1.97531×10^{-7} and 0.0, respectively, indicating that excess excreta or cement does not significantly impact or could even be slightly detrimental regarding non-linear behavior. The AC interaction shows the highest positive value of 5.92593×10^{-7}, showing strong synergy between lignin and cement, which enhances flexural strength. In contrast, BC and AB have negative coefficients of -1.48148×10^{-7} and -9.87654×10^{-8}, respectively, indicating that these combinations could have an unfavorable effect, although their impact is minor (see Table 9).

Table 9. Regression coefficients for flexural strength.

Coefficients	Estimate
Constant	0.292928
A: Lignin	−0.000239259
B: Bovine Excreta	0.00040963
C: Cement	0.000141111
AA	1.97531×10^{-7}
AB	-9.87654×10^{-8}
AC	5.92593×10^{-7}
BB	-1.97531×10^{-7}
BC	-1.48148×10^{-7}
CC	0.0

In conclusion, bovine excreta has the greatest positive impact on flexural strength, followed by cement, while lignin has a negative effect in linear terms. The quadratic effects suggest that a moderate increase in lignin could be beneficial, but excessive excreta dosages could be counterproductive. The interactions highlight the favorable combination of lignin and cement (AC), while excreta combinations with other components show negative effects.

Equation (3) presents the regression model fitted to the experimental data statistically analyzed for flexural strength, where EV is bovine excreta, C is cement, and CC is lignin. Additionally, in Figure 10, the estimated response surface for compressive strength as a function of the experimental variables analyzed is shown.

$$\begin{aligned} \text{Flexure} = \; & 0.292928 - (0.000239259 \times \text{CC}) + (0.00040963 \times \text{EV}) + (0.000141111 \times \text{C}) \\ & + 1.97531 \times 10^{-7} \times \left(\text{CC}^2 - 9.87654 \times 10^{-8}\right) \times \text{CC} \times \left(\text{EV} + 5.92593 \times 10^{-7}\right) \times \text{CC} \\ & \times \left(\text{C} - 1.97531 \times 10^{-7}\right) \times \left(\text{EV}^2 - 1.48148 \times 10^{-7}\right) \times \text{EV} \times \text{C} + 0.0 \times \text{C}^2 \end{aligned} \quad (3)$$

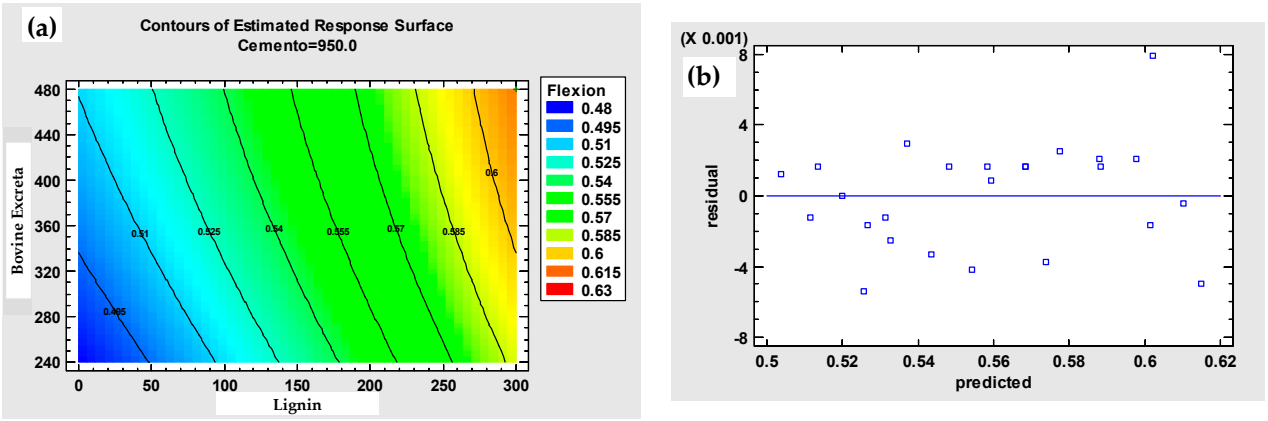


Figure 10. Flexural strength, (**a**) response surface, and (**b**) residuals.

Steepest Ascent Path for Flexural Strength

In Table 10, it is possible to observe a gradual increase as the lignin amounts are adjusted. In the first mixture, with 187.5 g of lignin, 352.5 g of bovine excreta, and 950 g of cement, the predicted flexural strength is 0.558333 MPa. When the lignin increases to 188.5 g, the predicted flexural strength increases to 0.558752 MPa, continuing to rise gradually to 0.560431 MPa at the last point, with 192.5 g of lignin, 354.018 g of bovine excreta, and 950.546 g of cement. This pattern indicates that the flexural strength improves slightly as the lignin amount increases. The steepest aids to identify the factor values that have the strongest effect on flexural strength, which is valuable for future experiments if the goal is to optimize the bio-brick flexural strength. Additionally, the path from the current experimental center region along which the estimated response changes rapidly for the smallest change in the experimental factors. It indicates good locations for further experiments if the goal is to increase or decrease the flexural strength. Currently, six points are generated by changing the lignin in increments of 1.0 g.

Table 10. Steepest ascent path for flexural strength.

Lignin (gr)	Excreta (gr)	Cement (gr)	Estimated Flexure (MPa)
187.5	352.5	950.0	0.558333
188.5	352.806	950.109	0.558752
189.5	353.11	950.218	0.559172
190.5	353.414	950.327	0.559591
191.5	353.716	950.437	0.560011
192.5	354.018	950.546	0.560431

3.2.3. Moisture Absorption Statistical Analysis

Estimated Effects for Moisture Absorption

Considering the estimated effects for moisture absorption (%), Table 11 and Figure 11 show the individual contributions of each factor and their interactions in the statistical model. The average moisture value is 0.425%, representing the moisture reference level when all factors are at their central values. The lignin factor (A) significantly positively affects moisture, with an estimated increase of 0.0688889%. This behavior suggests that as the lignin content increases, moisture also increases. The other factor effects, such as bovine excreta (B) and cement (C), are smaller but still important because they present a positive effect of 0.0222%, while cement contributes to a lesser extent, with a 0.0122% increase in moisture. These effects are consistent with the literature, as organic materials

such as bovine excreta can contribute to higher water absorption than inorganic additives like cement.

Table 11. Estimated effects for moisture (%).

Effect	Estimate	Stnd. Error	V.I.F.
Average	0.425556	0.00081683	---
A: Lignin	0.0688889	0.000756238	
B: Bovine excreta	0.0222222	0.000756238	
C: Cement	0.0122222	0.000756238	
AA	−0.02	0.00130984	
AB	−0.0075	0.000926198	1.0
AC	0.0025	0.000926198	
BB	−0.00333333	0.00130984	
BC	−0.0025	0.000926198	
CC	0.0	0.00130984	

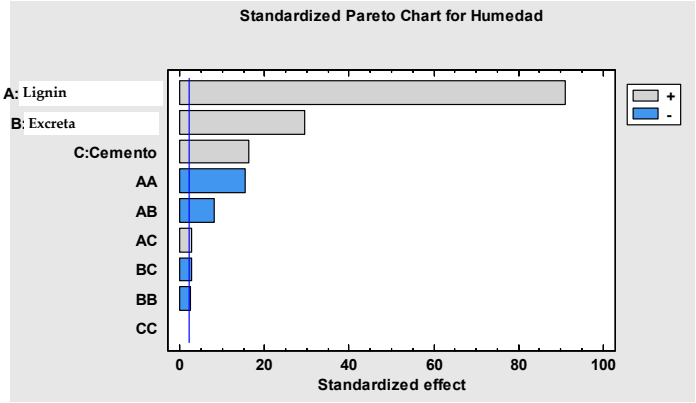


Figure 11. Standardized Pareto chart for moisture.

On the other hand, the factor interactions analysis is also relevant, as the interaction between lignin and bovine excreta (AB) shows a negative effect of −0.0075%, implying that when both factors increase simultaneously, their effect on moisture is slightly reduced. This interaction may indicate that the combined effects of these two materials are not completely additive but may have adverse effects on moisture absorption. The interaction AC (lignin and cement) has a positive effect of 0.0025, suggesting that the presence of both materials tends to increase the moisture in the bio-bricks slightly.

ANOVA for Moisture Absorption

It is evident that different factors and their interactions affect the moisture absorption content in bio-bricks. Firstly, the *p*-values for factors A (lignin), B (bovine excreta), and C (cement) are all below 0.05, indicating that these factors have a significant effect with a high correlation. The lignin factor (A), with a *p*-value of 0.0001 and an F-Ratio of 8298.16, has the most significant impact on moisture, confirming its relevance in moisture behavior as a non-absorptive agent. Bovine excreta (B) and cement (C) are also significant factors, though with a lesser impact compared to lignin, having F-Ratios of 863.49 and 261.21, respectively (see Table 12).

The effects of interactions between factors are highly significant, as interactions AA (lignin), AB (lignin with bovine excreta), AC (lignin with cement), BB (bovine excreta), and BC (bovine excreta with cement) all present *p*-values below 0.05. Among them, inter-

action AA (lignin with itself) has the highest F-Ratio of 233.14, indicating that the influence of lignin on moisture is not only linear but also depends on the amount of material in the mixture.

Table 12. Analysis of variance for moisture absorption.

Source	Sum of Squares	DOF	Mean Square	F-Ratio	*p*-Value
A: Lignin	0.0213556	1	0.0213556	8298.16	0.0001
B: Bovine excreta	0.00222222	1	0.00222222	863.49	0.0001
C: Cement	0.000672222	1	0.000672222	261.21	0.0001
AA	0.0006	1	0.0006	233.14	0.0001
AB	0.00016875	1	0.00016875	65.57	0.0001
AC	0.00001875	1	0.00001875	7.29	0.0152
BB	0.0000166667	1	0.0000166667	6.48	0.0209
BC	0.00001875	1	0.00001875	7.29	0.0152
CC	0.0	1	0.0	0.00	1.0000
Total error	0.00004375	17	0.00000257353	---	---
Total (corr.)	0.0251167	26	---	---	---
R^2	99.82%				
R^2 ajusted	99.73%				
MAE	0.000997942				
Durbin–Watson	1.89153 (p = 0.1476)				

Where: DOF, are Degrees of Freedom.

On the other hand, the coefficient of determination (R^2) equal to 99.8258% indicates that the model used to analyze moisture explains nearly 100% of the variability observed in the bio-bricks. The adjusted R^2, which corrects for the number of independent variables in the model, is 99.73%, reinforcing the fit quality. The standard error of estimation of 0.00160422 is low, suggesting that moisture estimates provide a good fit, too. Additionally, the mean absolute error (MAE) of 0.000997942 is very small, further supporting the precision of the model's predictions (see Table 12). The Durbin–Watson statistics have a value of 1.89153 with a *p*-value of 0.1476, suggesting no evidence of autocorrelation in the model's residuals. These results mean there are no systematic patterns in prediction errors, which indicates that the residuals are random and not influenced by the order in which data were collected [44].

Finally, eight effects have *p*-values less than 0.05 for moisture absorption, indicating that they are significantly different from zero at the 95.0% confidence level. The Durbin–Watson (DW) statistic tests the residuals to determine if there is any significant correlation based on the order in which they appear in the data file. Since the *p*-value is greater than 5.0%, there is no evidence of sequential autocorrelation in the residuals at the 5.0% significance level.

Regression Model Obtained for Moisture Absorption

The regression model obtained is useful for understanding how each factor and its interactions influence the moisture absorption of bio-bricks. The equation adjusted to the model includes a constant term of 0.118665, which represents the reference moisture when the factors are equal to zero. The coefficients for the linear factors (lignin, bovine excreta, and cement) indicate the direct relationship between each factor and moisture, and these coefficients are positive: 0.000495802 for lignin, 0.000458272 for bovine excreta, and 0.000158889 for cement suggesting that an increase in the amount of any of these materials will increase moisture absorption, albeit in small quantities due to the magnitude of the coefficients.

The quadratic effects of AA, BB, and CC are small and negative. For example, the coefficient for the interaction AB is -2.96296×10^{-7}, indicating that the combination of

these two factors decreases moisture, although of lesser magnitude. On the other hand, the interaction AC has a positive coefficient of 2.22222×10^{-7}, suggesting that this interaction slightly increases the moisture absorption of the bio-brick. Additionally, quadratic terms like AA and BB have negative coefficients, implying that increasing the amount of these materials beyond an optimal point could begin to reduce moisture. However, the quadratic term for CC showed a value of 0.0, indicating that cement does not have a significant quadratic effect on moisture in this model due to the physical and mechanical properties of the material (see Table 13).

Table 13. Regression coefficients for moisture.

Coefficients	Estimated
Constant	0.118665
A: Lignin	0.000495802
B: Bovine excreta	0.000458272
C: Cement	0.000158889
AA	-7.90123×10^{-7}
AB	-2.96296×10^{-7}
AC	2.22222×10^{-7}
BB	-1.31687×10^{-7}
BC	-2.22222×10^{-7}
CC	0.0

In conclusion, the combination and materials proportion, such as lignin, bovine excreta, and cement, affect moisture, and how their interactions and non-linear effects also play a crucial role in the final moisture behavior in bio-bricks. The small magnitudes of the coefficients suggest that changes in the factors may have relatively small but cumulative effects on moisture, indicating that precise adjustments to material proportions are important for controlling this property of the bio-bricks.

Equation (4) presents the regression model fitted to the experimentally analyzed data for moisture absorption. Where: EV is bovine excreta, C is cement, and CC is lignin. Additionally, Figure 12 shows the estimated response surface for moisture absorption as a function of the analyzed experimental variables.

$$\begin{aligned} \text{Moisture} = \; & 0.118665 + (0.000495802 \times \text{CC}) + (0.000458272 \times \text{EV}) + (0.000158889 \times \text{C}) \\ & - \left(7.90123 \times 10^{-7} \times \text{CC}^2\right) - (2.96296 \times 10^{-7} \times \text{CC} \times \text{EV}) + (2.22222 \times 10^{-7} \times \text{CC} \times \text{C}) \\ & - \left(1.31687 \times 10^{-7} \times \text{EV}^2\right) - (2.22222 \times 10^{-7} \times \text{EV} \times \text{C}) + \left(0.0 \times \text{C}^2\right) \end{aligned} \quad (4)$$

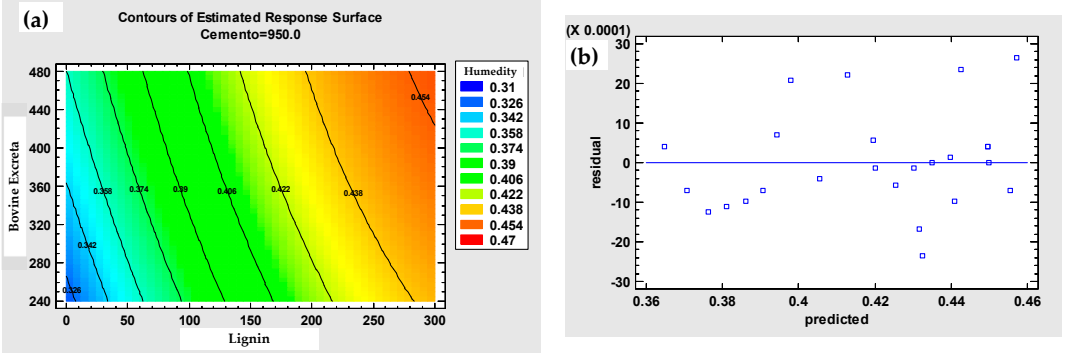


Figure 12. Moisture absorption, (**a**) response surface, and (**b**) residuals.

Steepest Ascent Path for Moisture Absorption

The steepest ascent path for moisture absorption, shown in Table 14, describes the fastest change in the property of interest (moisture absorption) as the experimental factors (lignin, bovine excreta, and cement) are varied in a controlled manner. This path indicates how the factors adjust to maximize moisture absorption in the bio-bricks to obtain the lowest possible moisture absorption. In this case, the path was determined by changing the weight of lignin in increments of 1.0 g while keeping the other factors (bovine excreta and cement) relatively constant.

Table 14. Steepest ascent path for moisture absorption.

Lignin (gr)	Excreta (gr)	Cement (gr)	Estimated Moisture (%)
187.5	352.5	950.0	0.425556
188.5	352.823	950.079	0.425902
189.5	353.146	950.159	0.426248
190.5	353.47	950.239	0.426591
191.5	353.794	950.32	0.426933
192.5	354.119	950.401	0.427273

From Table 14, it is possible to observe that as lignin increases in small amounts (from 187.5 g to 192.5 g) while the other factors remain constant, the moisture gradually increases. At the starting point, with 187.5 g of lignin, the predicted moisture is 0.425556%. With each 1.0 g increment in lignin, the predicted moisture value increases slightly, reaching 0.427273% for the final combination with 192.5 g of lignin. This pattern suggests that within this range of changes, the moisture increases steadily with the increase in lignin, considering a slight rise. If the goal is to maximize moisture, following this path can be an effective way to make small adjustments in the lignin. Since the response is more sensitive to changes in lignin along this path, smaller increments in this variable have the greatest impact on the predicted moisture. However, it is important to note that the variations in moisture are small throughout the analysis, suggesting that although the model predicts an increase, the magnitude of the changes may not be significant unless larger adjustments are made to the factors. Currently, 6 points were generated by changing the line in 1.0 g increments.

3.3. Experimental vs. Predicted Values for the Response Variables

Figure 13 compares experimental and predicted values for three response variables studied. The experimental values were obtained to modify cement, lignin, and bovine excreta compositions, as outlined in Table 1. Each property was evaluated using a regression model, with R^2 values indicating the prediction accuracy (<99%), similar results to those reported by [19,45].

The moisture predictions demonstrate a positive correlation with experimental values, as was evidenced in Figure 13a with $y = 0.6019x + 0.1688$. $R^2 = 0.8199$ shows that the model explains approximately 81.99% of the variability in the experimental data. However, the offset of 0.1688 suggests a systematic deviation that should be minimized. This variability may stem from slight inconsistencies in the material composition or moisture interaction with lignin and bovine excreta. Future iterations could enhance the accuracy by incorporating additional variables or considering a non-linear model.

The model performs good agreement in predicting flexural strength, with $R^2 = 0.9818$, indicating minimal discrepancy between predicted and experimental values. The $y = 0.5987x + 0.2249$ equation implies strong proportionality, while the intercept of 0.2249 highlights a small preference (see Figure 13b). Experimental results from Table 1,

such as flexural strength of 0.550 ± 0.1117 MPa at 10% composition, align closely with predicted values. This high precision underlines the robustness of the model for flexural performance across various material compositions.

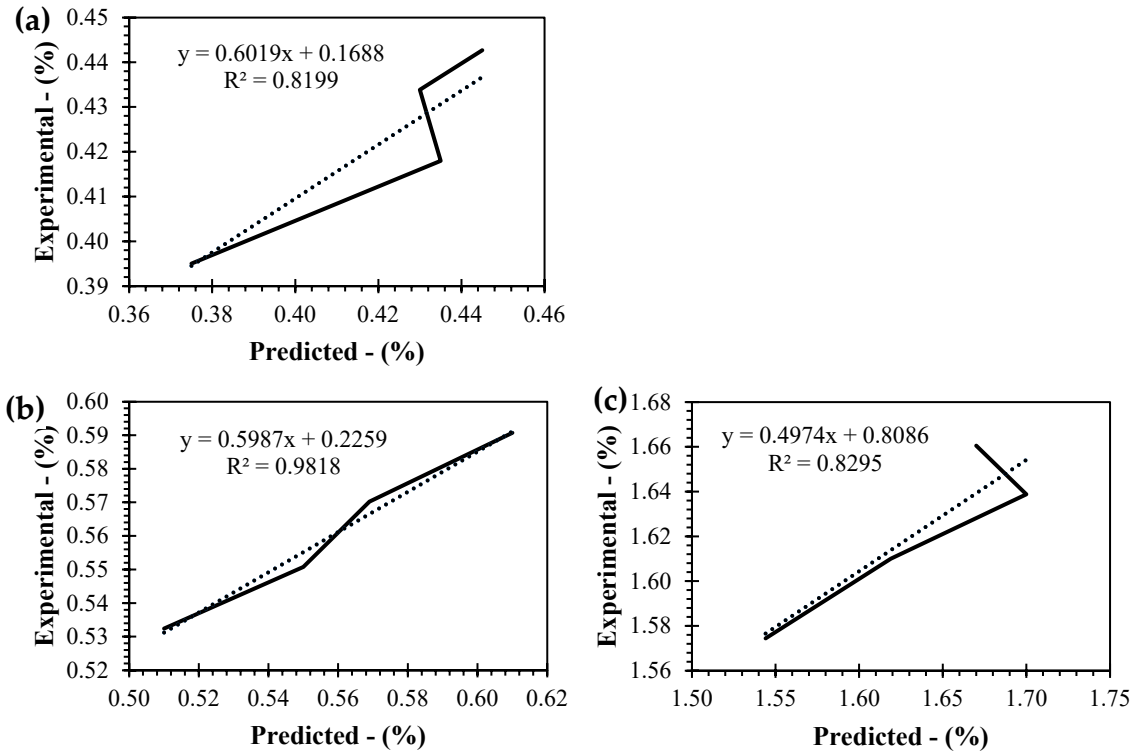


Figure 13. Comparison between experimental and predicted values for (**a**) moisture, (**b**) flexure, and (**c**) compression.

Finally, in Figure 13c, the compression predictions exhibit moderate accuracy, with R^2 = 0.8295, suggesting that the model captures the general trend but leaves room for improvement. The linear equation y = 0.4974x + 0.8086 indicates a weaker correlation than flexure, and the significant offset (0.8086) suggests a consistent overestimation. For instance, experimental compression values range from 1.544 ± 0.0530 MPa at 20%, with predicted values showing larger deviations [44]. Refining the predictive compression model may require input parameter adjustments, particularly interactions between cement and excreta. These results are similar to those reported by [46].

4. Conclusions

This study on bio-bricks offers significant contributions to enhancing sustainability in the construction industry, as described as follows:

- This study demonstrates that bio-bricks, formulated with 959 g cement, 224 g lignin, and 314 g bovine excreta (follows a ratio of 4.3:1:1.4, which consists of cement (64%), lignin (15%), and bovine excreta (21%)), offer a sustainable and viable alternative to traditional masonry materials while complying with Colombian technical standards (NTC-4205). The optimized composition achieved a compressive strength of ~1.7 MPa, making it suitable for lightweight construction and low-load-bearing applications;
- Response Surface Methodology (RSM) effectively optimized the mixture design, with an R^2 value of 99.76%, confirming the model's high accuracy in predicting compressive strength while reducing experimental costs. ANOVA analysis identified lignin as the most influential factor in mechanical performance, particularly in enhancing compressive and flexural strength (F-ratio = 6050.88, p = 0.0001);

- ➢ Although bio-bricks exhibit lower mechanical properties than conventional concrete (5–12 MPa vs. 30–40 MPa for compressive strength), they present a viable alternative for sustainable construction due to their reduced CO_2 emissions (up to 70%) and biodegradable nature, promoting a closed-loop lifecycle. The predictive model further validated these results, with a maximum error of ± 0.0769 MPa for compressive strength and high reliability in flexural strength predictions ($R^2 = 0.9818$);
- ➢ In terms of sustainability, bio-bricks exhibit a significantly lower environmental impact, with a reduction in CO_2 emissions by up to 70% compared to traditional concrete bricks. Additionally, their biodegradable nature allows for a closed-loop lifecycle, making them a viable option for green construction projects.

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