

Article

Optimization via Taguchi of Artificial Lightweight Aggregates Obtained from Kaolinite Clay and Ceramic Waste: Development and Industrial Applications

José Anselmo da Silva Neto ^{1,*} , Marcos Alyssandro Soares dos Anjos ^{1,2}, Ricardo Peixoto Suassuna Dutra ¹, Maelson Mendonça de Souza ³ and Cinthia Maia Pederneiras ⁴ 

¹ Department of Materials Science and Engineering, Federal University of Paraíba (UFPB), João Pessoa 58051-900, Brazil; marcos.anjos@ifpb.edu.br (M.A.S.d.A.); ricardodutra@ct.ufpb.br (R.P.S.D.)

² Department of Civil Engineering, Federal Institute of Paraíba (IFPB), João Pessoa 58015-430, Brazil

³ Department of Civil Engineering, Federal Institute of Rio Grande do Norte (IFRN), Natal 59010-180, Brazil; maelson.mendonca@ifrn.edu.br

⁴ Buildings Department, National Laboratory for Civil Engineering (LNEC), 1700-066 Lisbon, Portugal; cpederneiras@lnec.pt

* Correspondence: jasn@academico.ufpb.br

Abstract: Lightweight artificial aggregates (LWAs) are widely used in civil construction, but their conventional production depends on pure clays, a finite natural resource that negatively impacts the environment. This study aims to contribute to minimizing this issue by exploring the use of sustainable ternary mixtures of kaolinitic clay (KC), chamotte residues (CHT), and eucalyptus firewood ash (EFA), promoting a more environmentally friendly approach to the manufacture of LWAs. Thus, the aim was to develop and optimize LWAs using different replacements of industrial waste. Furthermore, the Taguchi method is employed to identify the optimal manufacturing parameters, such as waste content, sintering temperature, and heating time. The research involved the production of 32 distinct mixtures with different proportions of KC, CHT, and EFA, processed through grinding and sintering at temperatures ranging from 1075 °C to 1180 °C. The samples were evaluated for density, water absorption, mechanical strength, and expansion index. Statistical analysis was conducted using ANOVA to validate the most significant factors. The results revealed that mixtures with 80% of waste presented an aggregate expansion index of up to 60%, a minimum bulk density of 1.20 g/cm³ (which aligns with requirements for structural applications but exceeds the maximum bulk density for some lightweight aggregates), and crushing strength higher than 5 MPa, satisfying the normative criteria for commercial LWAs. In addition, 63 industrial applications were identified for the developed materials, ranging from structural lightweight concretes to thermal and acoustic insulation with varied microstructures. Therefore, the partial replacement of clay by CHT and EFA waste represents a promising alternative for producing sustainable LWAs, helping to reduce environmental impacts while providing quality materials for various applications in the most diverse industrial sectors.

Keywords: artificial lightweight aggregate; kaolinite clay; chamotte; eucalyptus firewood ash; Taguchi method



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1. Key Points

- (1) Production of commercial LWAs with ternary mixtures of KC, CHT, and EFA.
- (2) Development of pyro-expansive additive with 80% CHT and EFA residue.

- (3) The expansive prediction based on Riley and $\text{SiO}_2/\Sigma\text{OF}$ is inadequate for LWAs with ternary KC, CHT, and EFA mixtures.
- (4) Of the 32 mixtures, 18 showed properties equivalent to a commercial LWA, enabling 63 industrial applications.
- (5) The Taguchi method identified critical parameters for optimizing LWAs.

2. Introduction

Lightweight aggregates (LWAs) are porous materials with an apparent density of up to 1200 kg/m^3 , formed by releasing gases during the decomposition of organic matter within a viscous matrix [1–4]. LWAs are widely used in thermal and acoustic insulation, landscaping, civil construction, and water treatment, showcasing their versatility and industrial relevance [5–7].

Clay is the primary raw material for LWAs due to the release of gases during combustion, promoting expansion [8,9]. For effective expansion, clays must contain SiO_2 (48–70%), Al_2O_3 (8–25%), and fluxing oxides ($\text{CaO} + \text{MgO} + \text{Na}_2\text{O} + \text{K}_2\text{O}$ in concentrations ranging from 4.5% to 31%) [10]. However, not all clays possess this capability, necessitating compositional modifications through the use of waste or pyro-expansive additives for LWA production. The evaluation of the expandability of LWAs takes into account factors such as fineness [11], $\text{SiO}_2/\Sigma\text{OF}$ ratio [12,13], and physicochemical and mineralogical properties [10]. Although well established for natural clays, these correlations may present inconsistencies with the use of waste, such as expansions outside the ideal range [14–23]. Therefore, the development of LWAs with waste remains a challenge, reinforcing the limitations of the models proposed in the literature for these applications.

The previous study [8] investigated the incorporation of waste materials, specifically chamotte (CHT) and eucalyptus firewood ash (EFA), into binary mixtures for the production of LWAs. To advance the understanding of this field, this study expands the scope by examining ternary mixtures containing the same waste materials, thereby introducing a greater level of formulation complexity. An effective strategy for optimizing such mixtures could involve leveraging machine learning techniques. However, developing reliable data-driven models requires a substantially larger dataset with numerous mixture formulations. The generation of such a dataset would necessitate the use of significantly greater quantities of raw materials and resources, an aspect this study sought to minimize [24,25].

To optimize these mixtures, the Taguchi method was employed as an alternative, ensuring a systematic and efficient reduced experimental approach. Furthermore, this study contributes by incorporating additional characterizations, including scanning electron microscopy (SEM) and X-ray diffraction (XRD) of the samples, providing valuable insights into the properties of the developed materials.

The waste materials used in both studies are residues from the ceramic industry in the northeast of Brazil, João Pessoa, Paraíba. Chamotte (CHT) can be considered an adequate waste for LWA production due to its composition, which presents a high content of SiO_2 and Al_2O_3 , forming aluminosilicates during the sintering process, reducing porosity, and increasing the mechanical strength of LWAs [26–28]. The other waste, namely, eucalyptus firewood ash (EFA), also has strong potential to be used in LWA production, since it is a carbonate material that decomposes into calcium oxide (CaO) and carbon dioxide (CO_2), contributing to the expansion and increasing the porosity [8,10,14,15,17,29].

Therefore, this study examines 32 ternary LWA mixtures using KC, EFA, and CHT to develop regional-grade aggregates from ceramic industry waste in João Pessoa, PB, addressing the current concentration of national production in the southeast region. The research aims to expand accessible areas in the north and northeast regions, promoting sustainable solutions, including the reduction of CO_2 emissions, transportation costs, clay

use, and delivery times, by incorporating up to 80% industrial waste. Furthermore, given the non-expansive nature of KC, the study evaluates EFA and CHT as potential pyro-expansive additives, leveraging their chemical and mineralogical properties to increase porosity during sintering. Additionally, it fills a critical gap by providing experimental data from 32 samples, creating a reliable catalog for engineering applications with statistical estimation support.

3. Experimental Program

Figure 1 presents a comprehensive overview of the entire experimental program, encompassing the selection of raw materials for the production of lightweight aggregates (LWAs) and the various sintering conditions explored (using the Taguchi method), visually represented through different color schemes. The study initially considered 32 potential mixtures [8]; however, those containing 40%, 60%, and 80% waste content experienced complete melting at the sintering temperature of 1180 °C (mixtures 7, 10, 13, 23, 26, and 29). This prevented their characterization and ruled out their suitability for industrial application. As a result, only 26 viable mixtures were successfully analyzed and are presented in this manuscript.

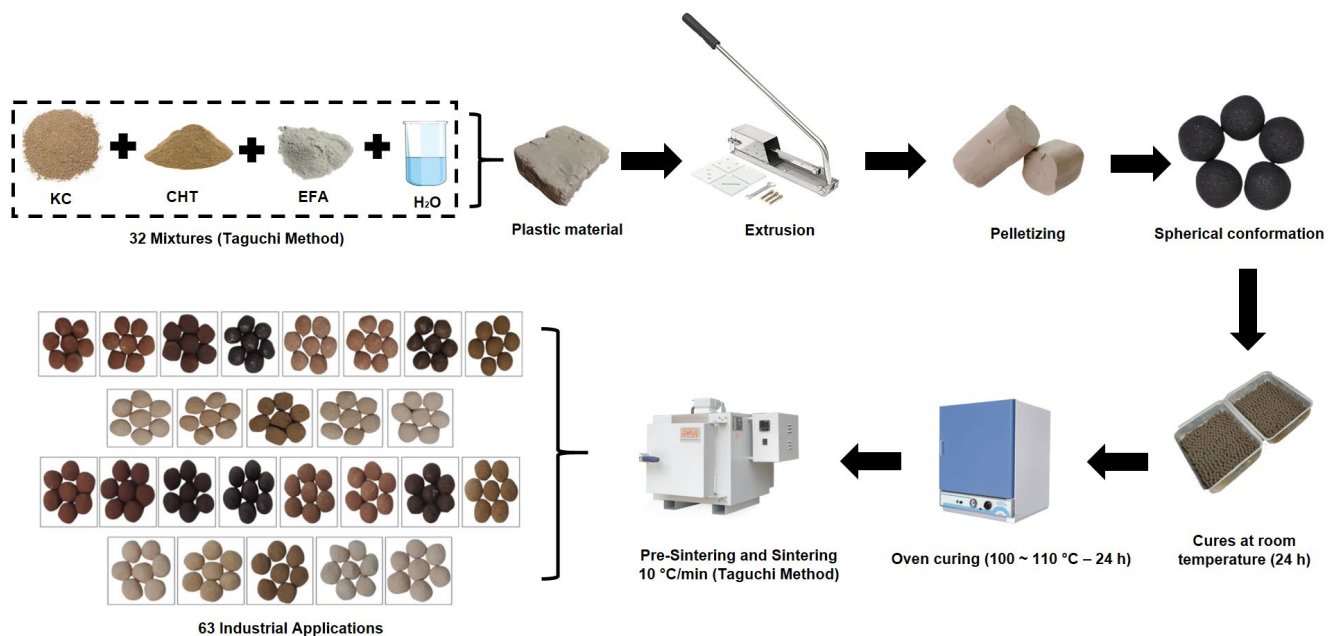


Figure 1. Graphical summary of the experimental program.

The materials utilized for LWA production in this study are identical to those previously characterized in [8]. The methods performed in this study are the same as those utilized in the previous investigation and are summarized in Table 1.

For the production of LWAs, the materials were dried and ground to achieve a particle size fraction of approximately 0.15 mm, in accordance with the specifications of Cougny [11] and previous studies [10,13,29,30]. The experimental design was based on LWA parameters such as waste replacement percentage, water content, aggregate size, pre-heating, and sintering conditions [8,9,22,31–33]. Thirty-two mixtures were awarded, as described in Table 2, following the same methodology as Silva Neto et al. [8].

Table 1. Experimental testing of raw materials and lightweight aggregates (LWAs).

	Tests	Objective
Materials (KC, CHT, EFA)	Particle size distribution	Grain size distribution
	X-ray fluorescence (XRF)	Chemical composition (oxides)
	Loss on ignition (LOI)	Mass loss from volatiles
	X-ray diffraction (XRD)	Crystalline phase identification (mineralogy)
	Thermogravimetry analysis (TGA)	Thermal stability and decomposition
	Scanning electron microscope (SEM)	Surface morphology and particle structure
LWAs	Bloating index	Expansion capacity during sintering
	Mass loss	Material loss during firing
	Density	Bulk density of aggregates
	Water absorption	Porosity
	Unit mass	Loose bulk density
	Crush resistance	Mechanical strength under load
	Modulus of deformation	Elastic behavior under stress
	Visual analyses	Macroscopic features (shape, cracks, color)
	Scanning electron microscope (SEM)	Surface morphology and particle structure
	X-ray diffraction (XRD)	Crystalline phase identification (mineralogy)
	Classification	Industrial applications (standards)

Table 2. Experimental program of LAW mixtures (Adapted from [8]).

Taguchi Method—Matrix L32 ($2^1 \times 4^6$)							
Nº	Aggregate Size (mm)	Residue Content (%)	Pre-Heating (°C)	Pre-Heating Time (min)	Sintering (°C)	Sintering Time (min)	Water Content (%)
1	6.25	20	750	10	1075	10	32
2	6.25	20	800	15	1110	15	33
3	6.25	20	850	20	1145	20	34
4	6.25	20	900	25	1180	25	35
5	6.25	40	750	10	1110	15	34
6	6.25	40	800	15	1075	10	35
7	6.25	40	850	20	1180	25	32
8	6.25	40	900	25	1145	20	33
9	6.25	60	750	15	1145	25	32
10	6.25	60	800	10	1180	20	33
11	6.25	60	850	25	1075	15	34
12	6.25	60	900	20	1110	10	35
13	6.25	80	750	15	1180	20	34
14	6.25	80	800	10	1145	25	35
15	6.25	80	850	25	1110	10	32
16	6.25	80	900	20	1075	15	33
17	12.5	20	750	25	1075	25	33
18	12.5	20	800	20	1110	20	32
19	12.5	20	850	15	1145	15	35
20	12.5	20	900	10	1180	10	34
21	12.5	40	750	25	1110	20	35
22	12.5	40	800	20	1145	25	34
23	12.5	40	850	15	1180	10	33
24	12.5	40	900	10	1145	15	32
25	12.5	60	750	20	1145	10	33
26	12.5	60	800	25	1180	15	32
27	12.5	60	850	10	1075	20	35
28	12.5	60	900	15	1110	25	34
29	12.5	80	750	20	1180	15	35
30	12.5	80	800	25	1145	10	34
31	12.5	80	850	10	1110	25	33
32	12.5	80	900	15	1075	20	32

The volume replacements were set at 20%, 40%, 60%, and 80%, with each percentage comprising equal proportions of the two waste materials. For example, a mixture with 20% waste content consists of 10% chamotte (CHT) and 10% eucalyptus firewood ash (EFA), combined with 80% kaolinite clay (KC). Similarly, a 40% replacement corresponds to 20% CHT, 20% EFA, and 60% KC; the 60% replacement consisted of 30% CHT and 30% EFA with 40% KC; and the 80% replacement contained 40% CHT and 40% EFA with 20% KC. This systematic variation made it possible to assess the influence of increasing waste content on the properties of the resulting mixtures. This proportional approach was consistently applied across all replacement levels, ensuring a balanced contribution of each waste material while varying the total waste content.

For each developed mixture, 150 spherical pellets were prepared and tested. The distribution of samples among the tests followed the protocol established in the previous study by Silva Neto et al. [8]. Figure 2a shows the chemical compositions of the ternary mixtures with 20%, 40%, 60%, and 80%, according to the experimental program, represented and classified as 1, 2, 3, and 4, respectively, in the Riley ternary diagram [8,10]. The $\text{SiO}_2/\Sigma\text{OF}$ ratio is shown in Figure 2b; the ideal range for commercial LWAs is between 1.00 and 3.85 [8,14,32].

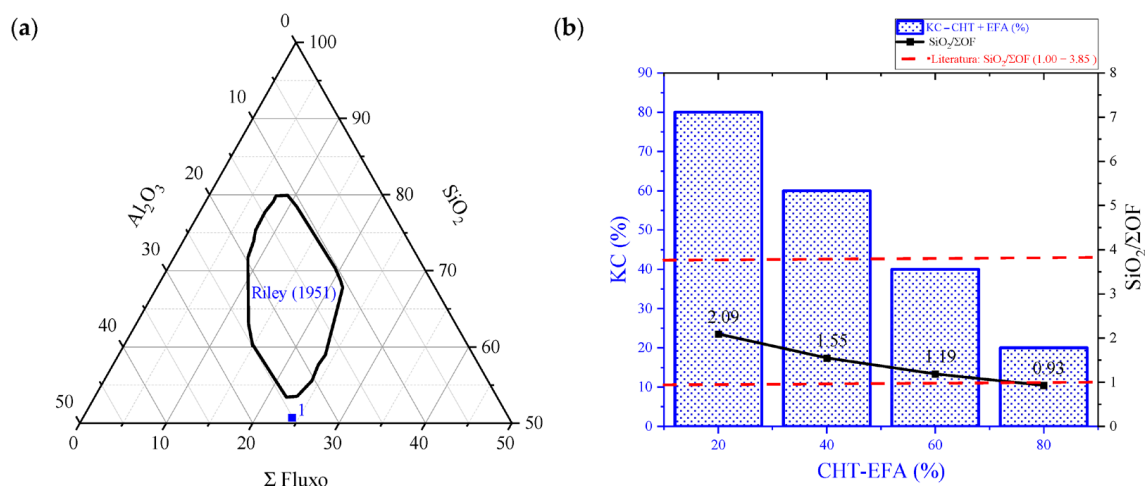


Figure 2. (a) Chemical composition of ternary mixtures in the Riley ternary diagram [10]; and (b) Values of the $\text{SiO}_2/\Sigma\text{OF}$ ratio.

Blends containing 40%, 60%, and 80% waste are not shown in the diagram (Figure 2a) because their SiO_2 content is less than 50%, although it is possible to produce LWAs in these proportions [8,10,14,30]. These blends may have insufficient viscosity to capture gases at high temperatures, compromising pore formation [13,27,31]. The $\text{SiO}_2/\Sigma\text{OF}$ ratio of the mixtures varies between 0.93 and 2.09, with most of the mixtures being within the optimal range [14], except for the one with 80% waste, which presents a slight deviation (Figure 2b). Increasing the waste content reduces the $\text{SiO}_2/\Sigma\text{OF}$ ratio, impacting the expansion mechanism and controlling the pyroclastic deformation temperature [33].

Furthermore, a statistical analysis was conducted following the methodology described in [8], employing the signal-to-noise (S/N) ratio to identify the key parameters influencing the evaluated response and ANOVA to validate this relationship. Subsequently, response surface plots were adjusted based on the two variables with the greatest influence on the response.

4. Results and Discussions

The discussion of the results focuses on the percentage of waste replacement and the sintering temperature, since these two factors were the ones that had the greatest influence on the results statistically.

4.1. Expansion and Mass Loss Rate

Figure 3 shows the behavior of ternary mixtures, highlighting the variations in the expansion index and mass loss of LWAs. The analysis in Figure 3 reveals that the mixtures present an expansion ranging from low to moderately high (5–60%), accompanied by a low mass loss (7.5–15%). According to the literature [4,8,29,34], this low expansion does not compromise the production of LWAs.

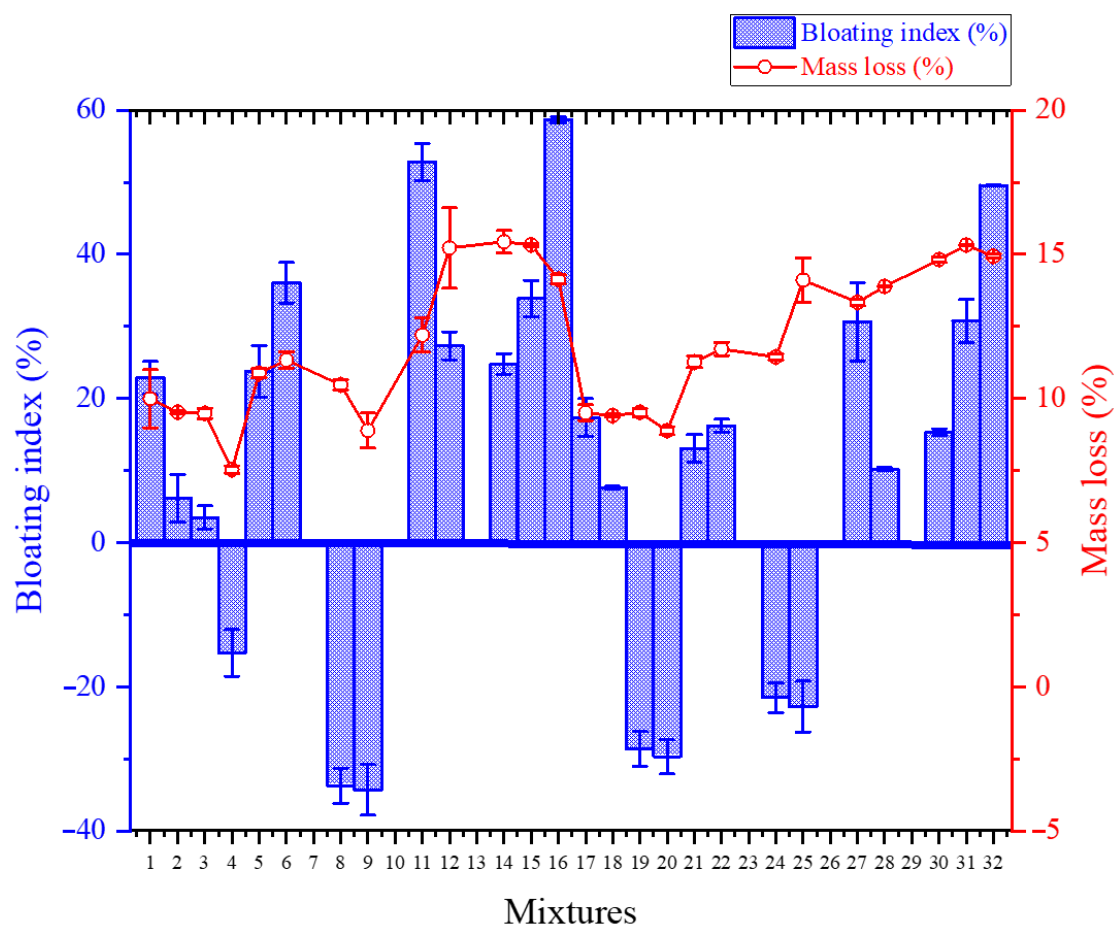


Figure 3. Expansion and mass loss index of LWAs.

The presence of volatile or reactive compounds, such as carbonates, sulfates, and organic matter, promotes the release of gases during heating, forming microcavities that significantly reduce the density of the material [8,9,27,35,36]. During sintering, precise temperature control is crucial to ensure partial fusion of the particle surface, trapping the released gases and preserving the internal cavities without collapsing.

Mixtures 7, 10, 13, 23, 26, and 29 showed total fusion (melting) at 1180 °C, especially with 40% and 80% residue content. Mixtures 4, 8, 9, 19, 20, 24, and 25 suffered contraction with 20% residue at 1180 °C and 40% and 60% at 1145 °C. This behavior results from total fusion (melting) or partial fusion (shrinkage), reducing the porosity and increasing the density of the ceramic matrix without sufficient release of expansive gases to generate additional porosity [19,37].

Increasing the waste replacement up to 80% at a temperature of 1145 °C (mixture 14) promoted the release of expansive gases during heating. This resulted in the formation of bubbles or internal cavities within the ceramic matrix, leading to the expansion of the material. This behavior was further enhanced by the chemical alterations in the mixture, which produced expansive compounds, as well as by the reactivity of the minerals present in the residues, as observed in the thermal and XRD analyses [8].

The most significant expansion was observed in mixture 16, with 80% residue and a sintering temperature of 1075 °C, reaching approximately 60%. This expansive behavior results from decomposing volatile compounds, such as carbonates and organic matter in the residues and KC, which release gases during heating [29,38–40]. In contrast, the most significant mass loss of 15% occurred in mixture 14, also with 80% residue, but at the higher temperature of 1145 °C. The partial melting of the material at this temperature facilitates the evaporation of water and other compounds, in addition to intensifying chemical reactions, thereby explaining the greater mass loss.

The results are relevant because they show the feasibility of developing pyro-expansion from CHT and EFA waste. When heated, these materials expand by releasing gases, which is advantageous in producing lower-density materials [7–9]. Furthermore, by reusing waste from the ceramic industry, the process contributes to the circular economy and sustainability in construction, reducing waste and adding value to discarded materials.

4.2. Specific Gravity and Water Absorption

Figure 4 shows the behavior of the ternary mixtures regarding specific mass and water absorption. Most of the mixtures presented water absorption below 20%, a value considered adequate for application in high-strength lightweight concrete, as described in commercial LWA catalogs [41–43]. Furthermore, most of the mixtures presented specific mass below 1.90 g/cm³, meeting the classification criteria for LWA as established by the standards [2].

Mixtures 5, 6, 11, 12, 15, and 16, with a higher residue content (40–80%) and sintered at lower temperatures (1075–1110 °C), exhibited water absorptions higher than 20%. In contrast, mixtures 1 to 4, with a lower residue content (20%) and a higher proportion of KC (80%), promoted the formation of denser microstructures with reduced porosity and increased specific mass, especially at higher sintering temperatures. Studies [41–45] reported similar results, associating residues with pore formation due to thermal decomposition and the release of volatile compounds.

The lowest water absorption, which was less than 1.0%, was observed in mixture 8 (40% residue) and sintered at 1145 °C, which also presented the highest specific mass (~2.20 g/cm³). The high temperature promoted partial fusion of the materials, filling the pores and densifying the structure. However, at 1180 °C, the sample melted completely, compromising its classification as an LWA. Similar results were recorded by [46–48], who highlighted the structural densification promoted by high temperatures during sintering.

Among the 32 mixtures evaluated, only four (8, 9, 19, and 25) presented specific masses greater than 2.00 g/cm³, thereby excluding them from the LWA classification according to the standards [1,2,49]. The lower sintering temperature (1075 °C) led to higher porosity due to incomplete fusion of the particles, particularly in mixes containing more than 20% waste. As a result, these mixes had lower specific gravities, typically below 2.00 g/cm³, as observed, for example, in mixture 4. According to the standard criteria for lightweight aggregates, materials with specific gravities below 2.00 g/cm³ can be classified as lightweight [1,2,41–43]. Therefore, these porous aggregates are potentially suitable for non-structural applications such as lightweight mortars, garden substrates, and landscaping elements, where thermal and acoustic insulation are beneficial.

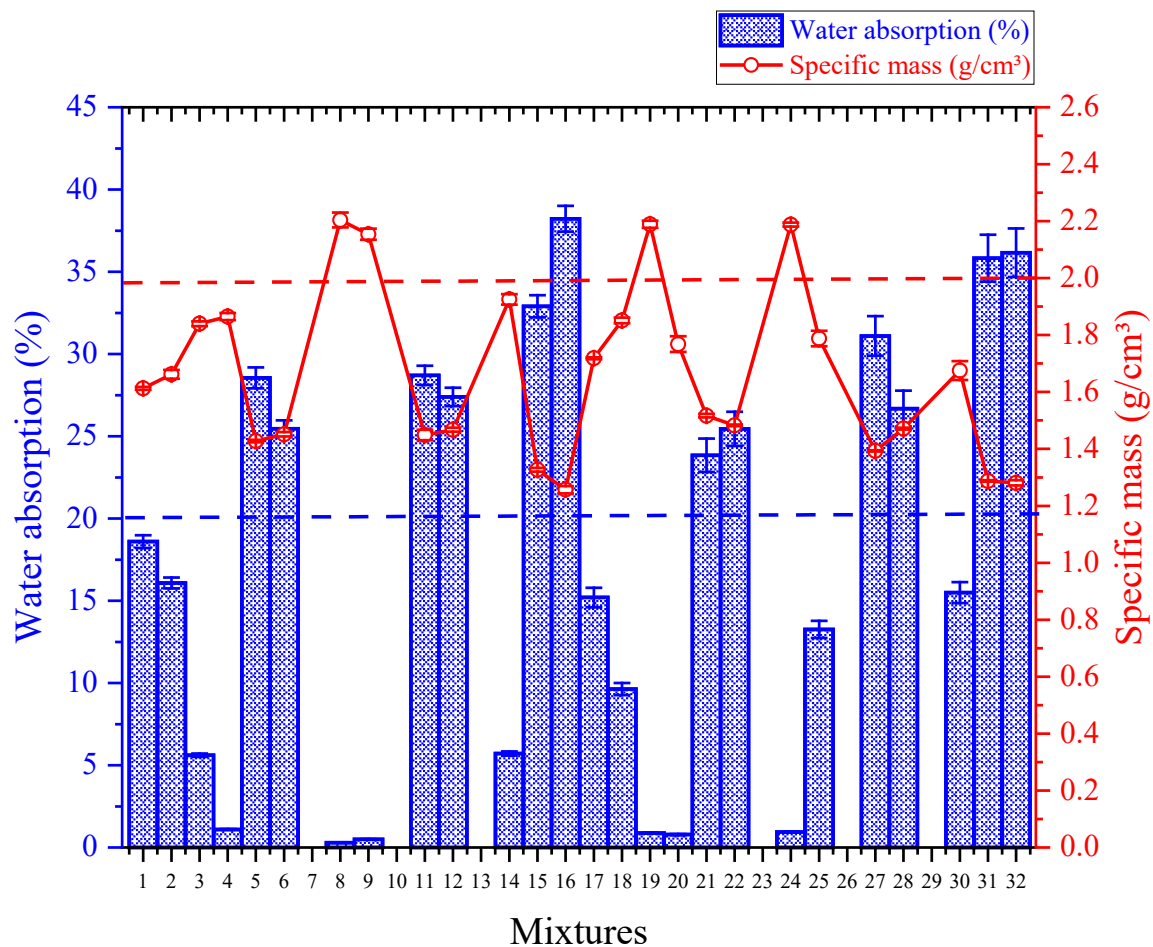


Figure 4. Water absorption and specific mass of LWAs.

The increase in residue content intensified the release of gases, contributing to the formation of interconnected pores, increased water absorption, and reduced density [50,51]. This variation in absorption makes it possible to adjust the LWAs to meet different demands, such as better retention or lower absorption, as needed.

4.3. Unit Mass and Expansion Index

Figure 5 shows the ternary mixtures according to the unit mass and the expansion index. The samples, in general, are mainly classified as light. Mixtures 4, 8, 9, 19, 20, 24, and 25 presented contractions when subjected to residue contents of 20% at 1180 °C and 40–60% at 1145 °C. It was observed that these mixtures, in particular, were close to the density limit that would disqualify them as light, indicating a direct relationship between the increase in density and contraction under these specific conditions.

The mixtures that presented shrinkage could balance the amount of water and the reactivity of the components during the sintering process. This balance minimizes the generation of excess gases or vapors, reducing the space available for expansion in the material, as described by [26,31,52]. In addition, the sintering temperature plays a fundamental role in the partial fusion of the components and the formation of liquid phases, which contributes to the rearrangement of the particles and the cohesion of the microstructure, resulting in a denser and more stable matrix [53–55].

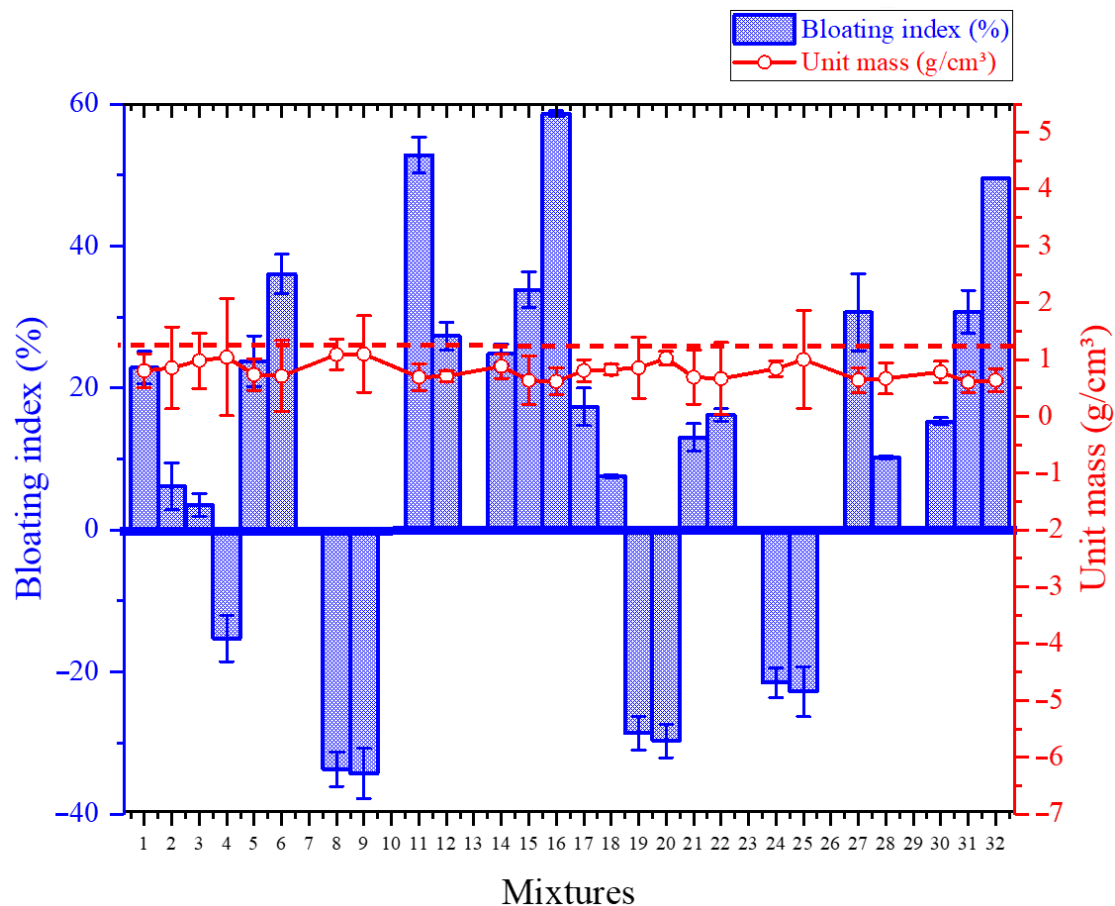


Figure 5. Unit mass and expansion index of LWAs.

As the residue content increases above 40%, unit masses decrease, especially at temperatures below 1145 °C. In the composition with 80% residue (mixture 32), the unit mass drops to 0.60 g/cm³. On the other hand, by reducing the residue content to 20% and maintaining the temperature at 1075 °C (mixture 1), the unit mass increases to 0.80 g/cm³. Previous research [10,33,34,40,56] found that, at high temperatures, densification of the clay matrix occurs, resulting in a reduction in porosity and high unit masses. With increasing waste content, this densification is increasingly affected by the residues [3,40,41]. Upon decomposition, calcium carbonate and calcium sulfate, present in the EFA residue, generate reactions that result in more significant expansion and pore formation, which significantly reduces the unit masses, corroborating the findings of [8,57,58].

4.4. Crush Resistance and Specific Mass

Figure 6 presents the ternary mixtures in terms of crushing resistance and specific mass. It is observed that crushing resistances are directly proportional to masses. In general, it was found that most mixtures meet the mechanical strength criteria established for commercial LWAs, with values above 1.00 MPa [41–43].

Mixtures 31 and 32 were the only ones that did not meet the criteria for commercial LWAs, presenting strengths below the established limit. Mixtures 7, 10, 13, 23, 26, and 29 suffered melting, attributed to the 40% and 80% residue content at a temperature of 1180 °C. On the other hand, mixtures 31 and 32, with 80% residue, presented insufficient sintering at lower temperatures (1075 °C and 1110 °C). However, when increasing the temperature to 1145 °C, mixture 30 demonstrated strength close to 5.00 MPa, meeting the requirements for commercial applications.

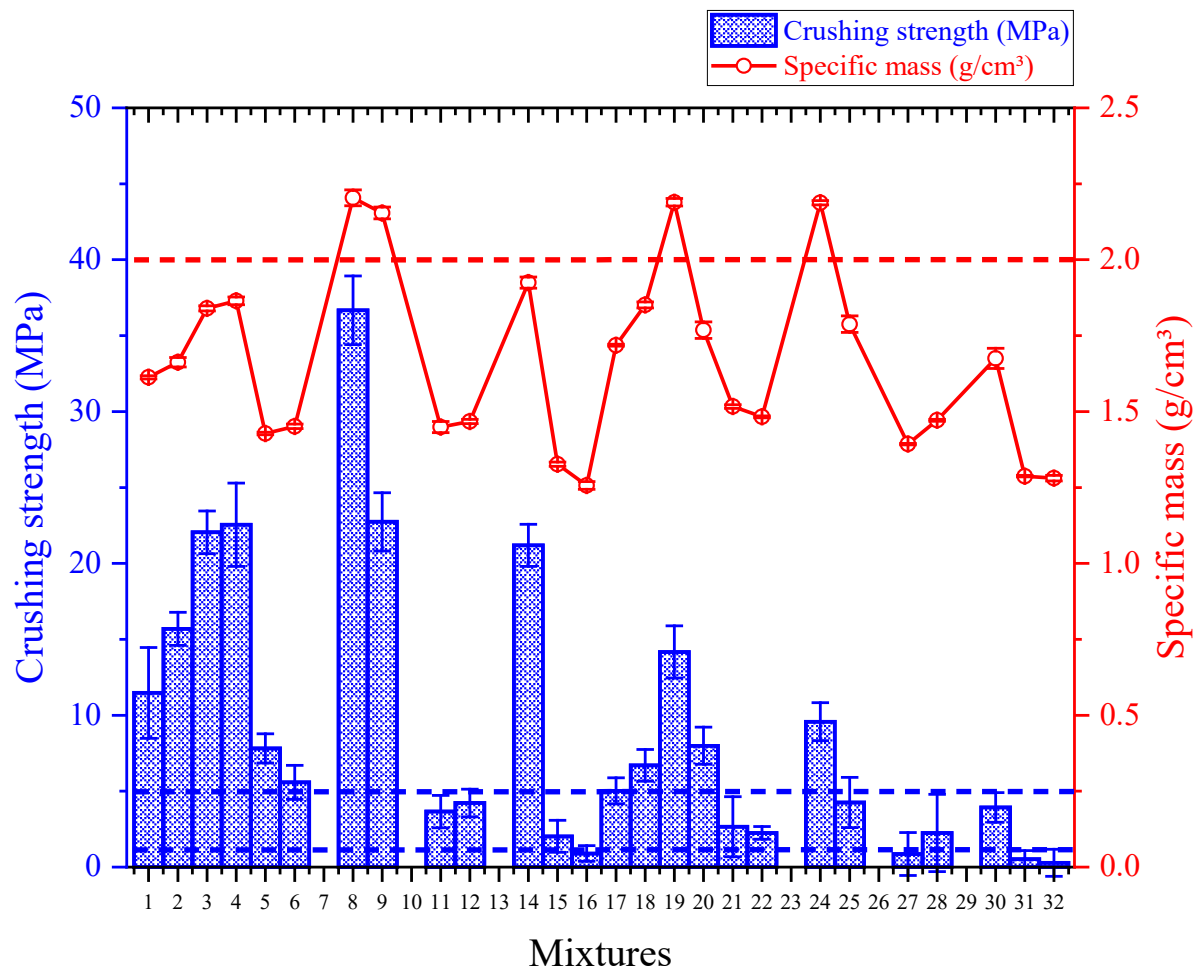


Figure 6. Crush resistance and specific mass of LWAs.

According to Silva Neto et al. [8], the mineralogical and chemical composition (CHT and EFA) of the mixtures containing 20% waste (Mixes 1 to 4) promoted the development of denser microstructures and higher mechanical strength, regardless of the sintering temperature. In contrast, mixtures with 80% waste content (Mixes 13 to 16) exhibited lower strength and density due to the excessive presence of fluxing agents and amorphous phases, which can hinder the formation of strong crystalline frameworks during sintering. This is due to the composition's excellent crystallinity and stability, which promotes a more cohesive and dense structure, with the formation of stable crystalline phases that improve the microstructure and mechanical properties [8,29,33,59–61].

The decomposition of organic material in the EFA, observed in the thermogravimeter [8], generates pores that reduce structural cohesion, explaining the decrease in mechanical strength with increasing residue content. Studies by [47,56,57,62,63] show that, during sintering, compounds rich in carbonates form amorphous and porous phases, resulting in lower density and lower mechanical strength.

Formulations 8, 9, 19, and 24, with 40% and 60% of residue sintered at 1145 °C, obtained the highest mechanical strengths and densities. The partial replacement of KC by CHT and EFA proved effective in optimizing sintering at high temperatures and improving gas retention. This performance is attributed to the more uniform particle size due to the differences in the average particle diameters [8], which favors the mechanical properties [8,11,13,29,30,64].

4.5. Modulus of Deformation and Specific Mass

The deformation modulus and specific mass are presented in Figure 7. The results indicate that the deformation modulus is proportional to the specific mass of the mixtures. Almost all the mixtures exhibited a deformation modulus higher than 15 GPa, exceeding the minimum limit of 3 GPa required for commercial LWAs. In some cases, such as mixtures 8, 9, 19, 24, and 25, the values exceeded 35 GPa.

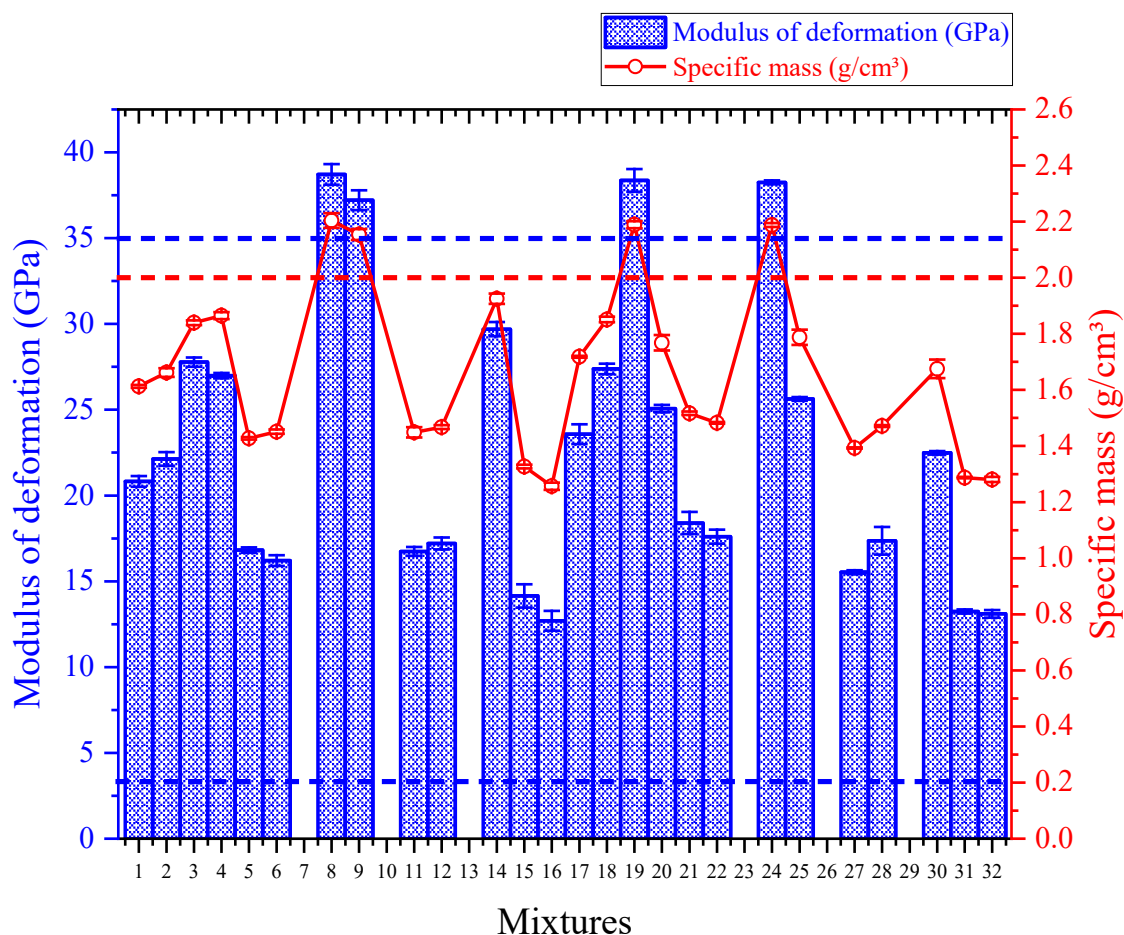


Figure 7. Deformation modulus and specific mass of LWAs.

The highest deformations and specific masses were recorded in the mixtures with 40% and 60% of residues, sintered at 1145 °C, emphasizing formulations 8, 9, 19, and 24. The replacement of KC by CHT and EFA residues demonstrated the potential to improve the sintering process at high temperatures and gas retention. This occurs due to the differences in the average particle diameters, which favor better particle size distribution, improve the mechanical properties, and increase the specific masses of the samples [13,29,64–68].

4.6. Visual Analysis

Figure 8 shows the 32 LWAs produced with CHT and EFA residues in particle sizes of 6.25 mm (mixtures 1–16) and 12.5 mm (mixtures 17–32), as described in the experimental program (Table 2). The colors range from light reddish-brown to white, including cream, black, and golden yellow shades. Mixtures 1 to 3 stand out, presenting a color identical to the expanded clay from Cinexpan, São Paulo/PB, Brazil [41,42].

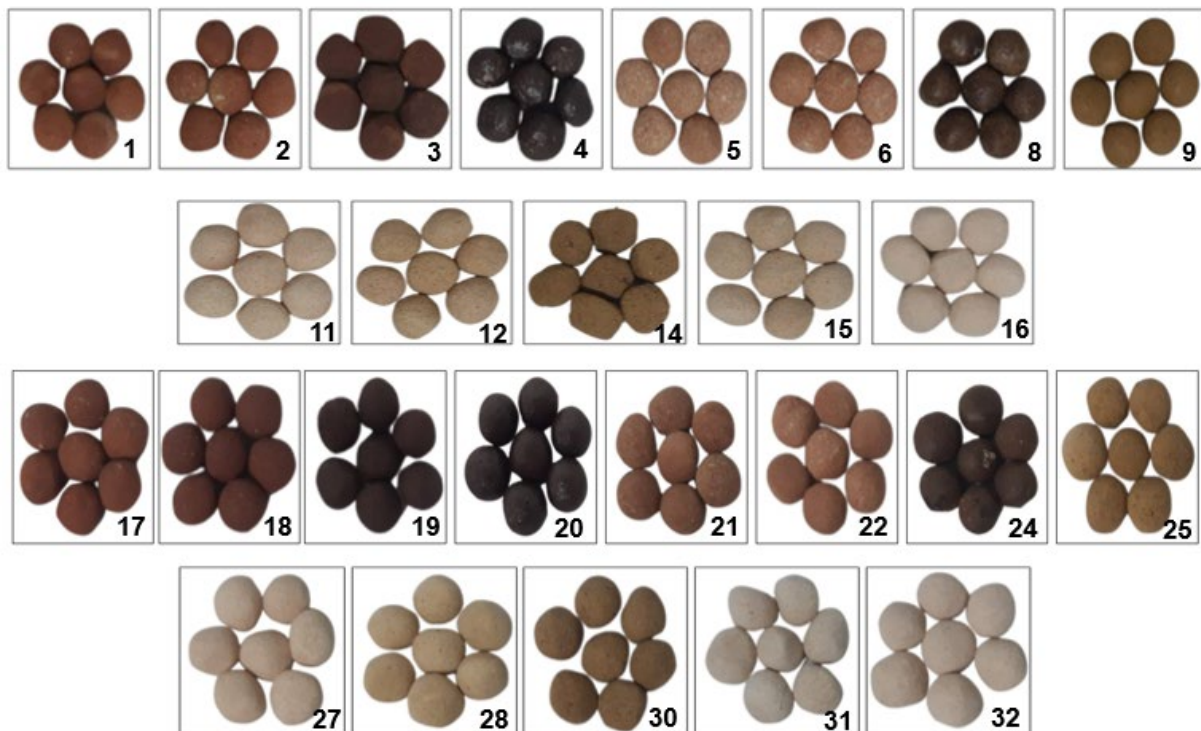


Figure 8. Visual analysis of the LWAs produced.

As illustrated in Figure 8, mixtures containing 20% waste present a reddish coloration, attributed to the oxidation of iron oxide (Fe_2O_3) present in the clay, whose concentration is higher in this proportion. This reddish hue intensifies as the temperature increases, culminating in a black coloration due to progressive sintering and changes in the oxidation states of the metal oxides, as also seen in the work of [8].

By increasing the waste content to 40%, a decrease in the intensity of the reddish hue is observed. Subsequently, it intensifies with increasing temperature, reflecting changes in the interaction between the oxides and the ceramic matrix.

In turn, high waste content (60% and 80%) results in lighter colors due to the melting effect of the waste (CHT and EFA), which reduces the melting point of the matrix and intensifies the viscous flow, influencing the final hue. However, at 1180 °C, samples with 40%, 60%, and 80% residues (mixtures 7, 10, 13, 23, 26, and 29) underwent complete melting, compromising the expected shape and properties. This result highlights the complex interaction between the chemical composition of the residues and the thermal conditions, highlighting the importance of optimizing processing parameters to avoid structural damage in formulations with high residue contents.

4.7. Microstructure

Figure 9 shows the microstructure of LWAs produced with different waste contents (20%, 40%, and 80%) and sintered at 1075 °C and 1145 °C. It highlights the influence of composition and sintering temperature on the internal structure of the aggregates. Mixtures 1, 3, 8, 14, and 16 were selected for this analysis, based on the experimental program (Table 2). These mixtures were chosen because, according to the statistical modeling, the sintering temperature and waste content were identified as the most significant factors influencing the properties of the LWAs. Selected samples were oven-dried at 110 °C for 24 h, mounted on aluminum stubs using carbon adhesive tape, and coated with a thin layer of gold via sputtering. Scanning electron microscopy (SEM) images were acquired at an accelerating voltage of 20 kV [8].

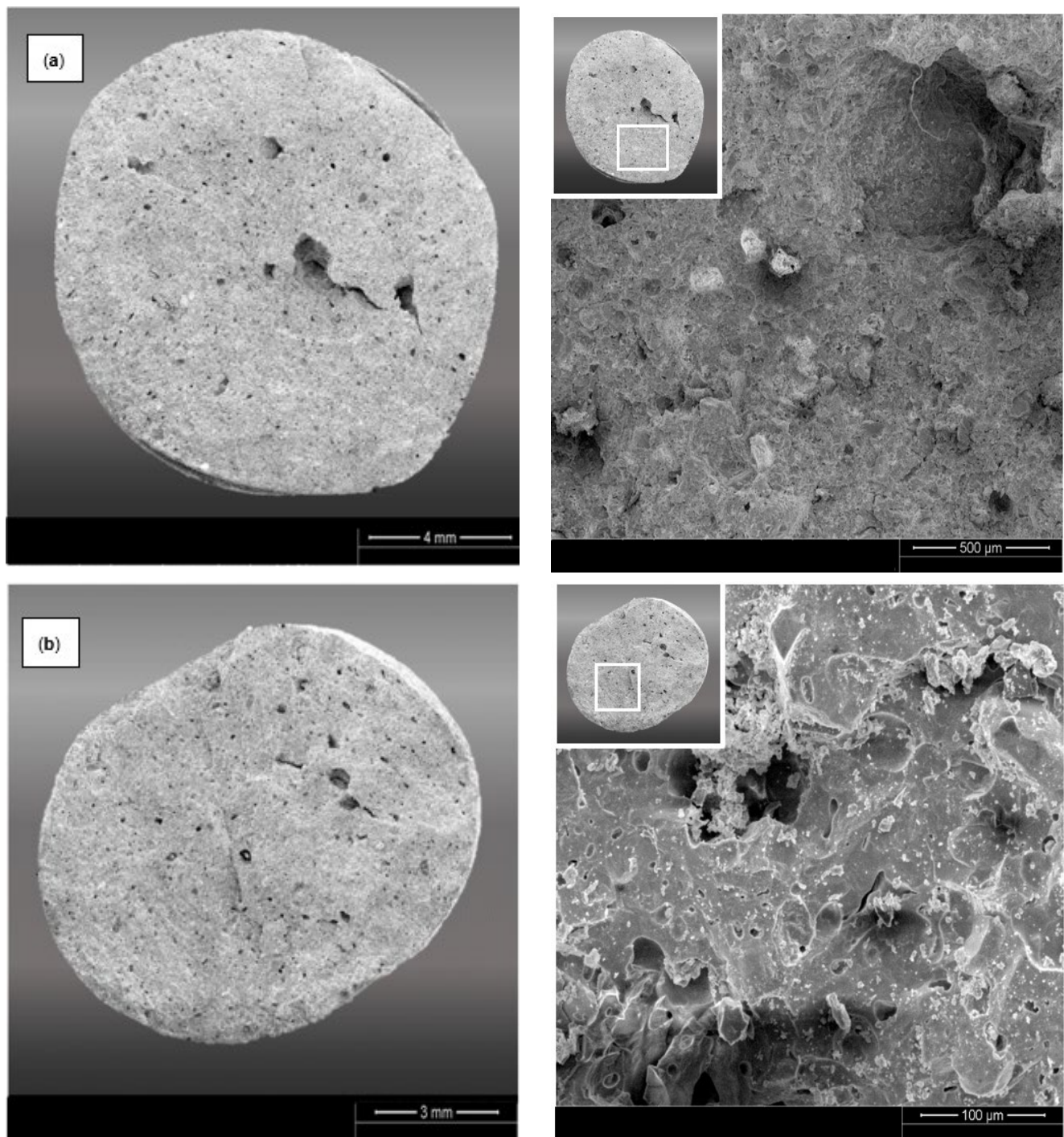


Figure 9. Cont.

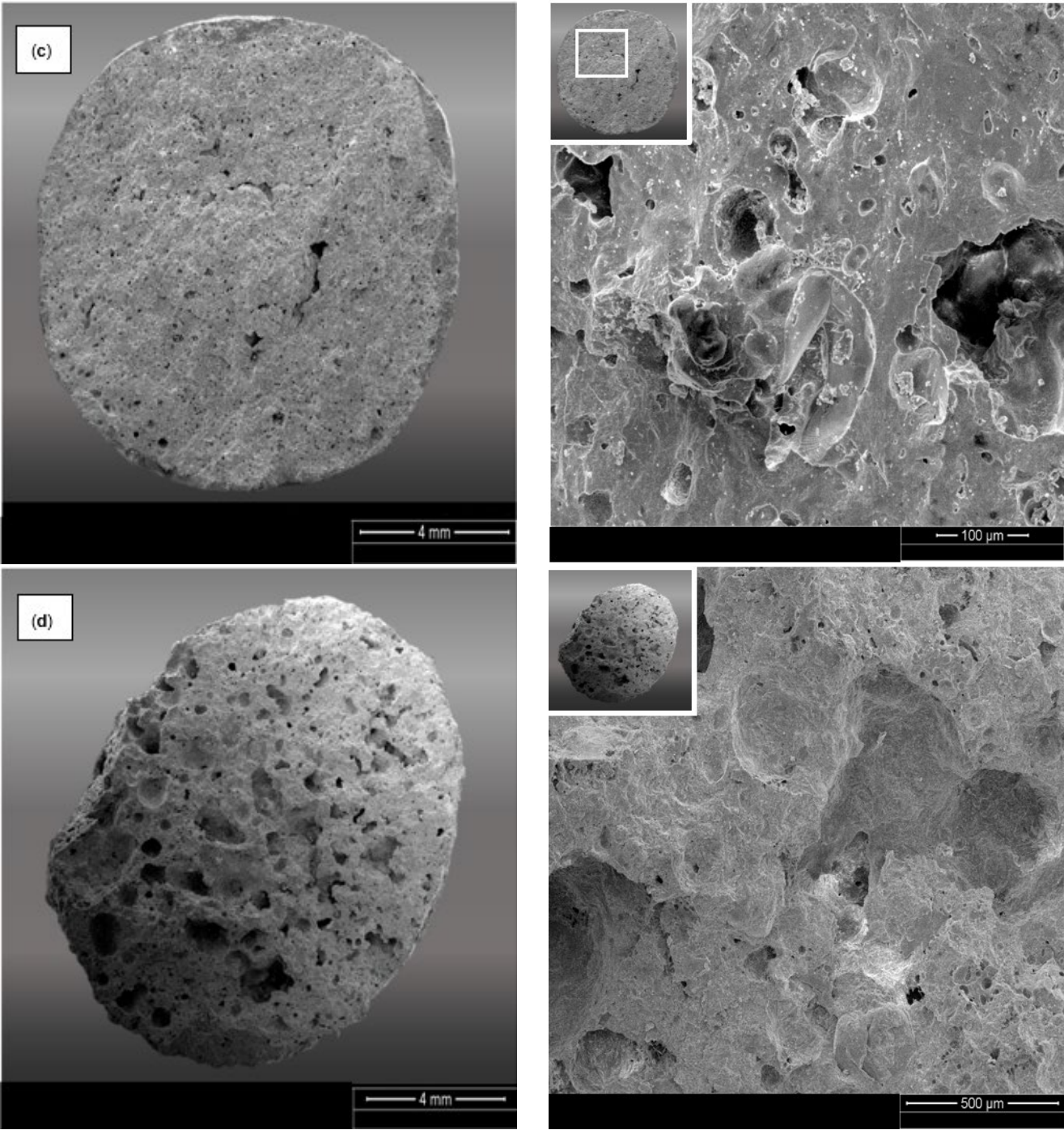


Figure 9. Cont.

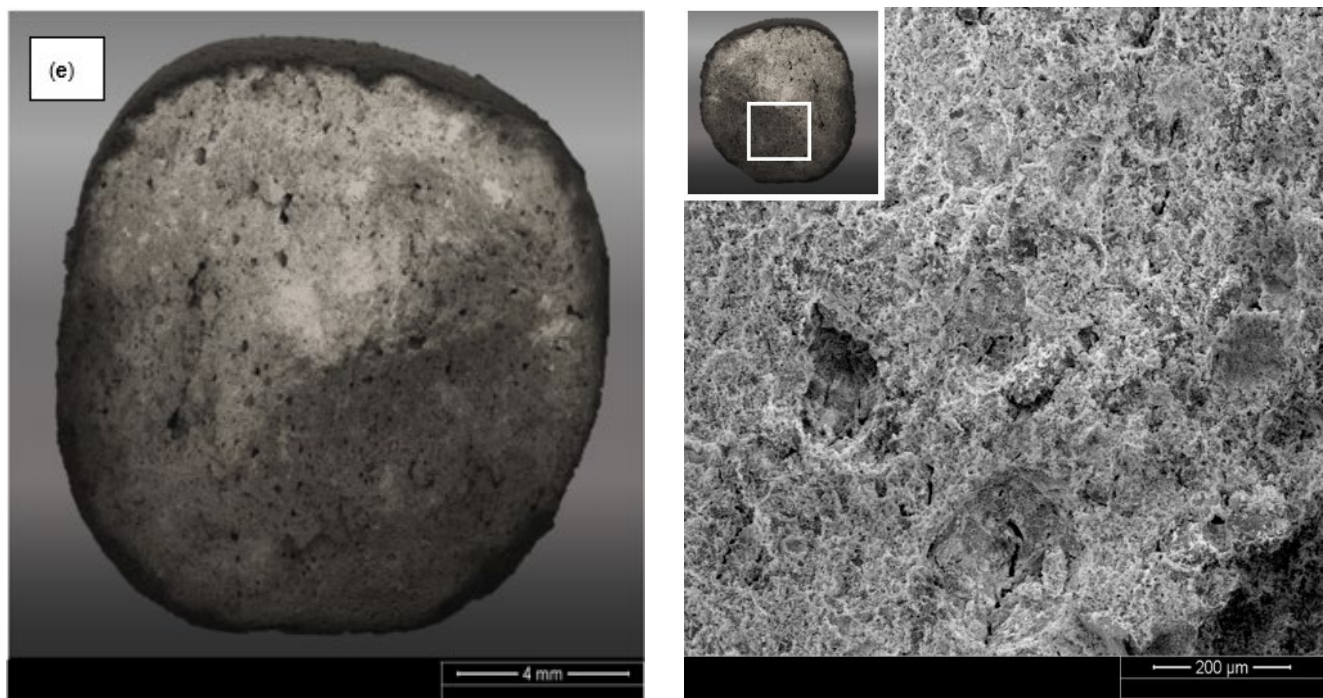


Figure 9. Scanning electron micrographs of LWAs: (a) Mixture 1; (b) Mixture 3; (c) Mixture 8; (d) Mixture 14; (e) Mixture 16.

Mixture 1, sintered at 1075 °C, and mixture 3, sintered at 1145 °C, both using 20% of the residue, demonstrate that increasing the temperature improves the sintering and mechanical properties of the LWAs. Mixture 1 reveals a porous and irregular structure with more significant voids, resulting in lower density and better water absorption. In contrast, mixture 3 exhibits a compact and homogeneous microstructure with lower porosity and greater mechanical strength.

Increasing the residue content from 20% to 40% while maintaining sintering at 1145 °C shows that mixture 8 presents denser grain packing and a more cohesive surface than mixture 3. This more significant densification reduced porosity and water absorption, significantly improving mechanical strength.

Comparing mixtures 14 and 16, the same pattern as in mixtures 1 and 3 is observed, where the increase in temperature favored sintering, resulting in a spongy microstructure in mixture 16 and a denser one in 14. When reducing the residue from 80% to 20% at 1145 °C, mixture 14 has a more open and interconnected structure than mixture 3 due to the release of gases in the decomposition of organic materials, resulting in more significant expansion, while vitrification contributes to maintaining the same mechanical strength. All this behavior is also seen in the work of [8,14,17,29,34,67,68].

Mineralogical analysis revealed the presence of gismondine in all the sintered mixtures, as shown in Figure 10. This zeolitic mineral, composed of hydrated calcium aluminosilicates, has a porous structure suitable for ion exchange, selective adsorption, and catalysis, with potential application in several industrial processes.

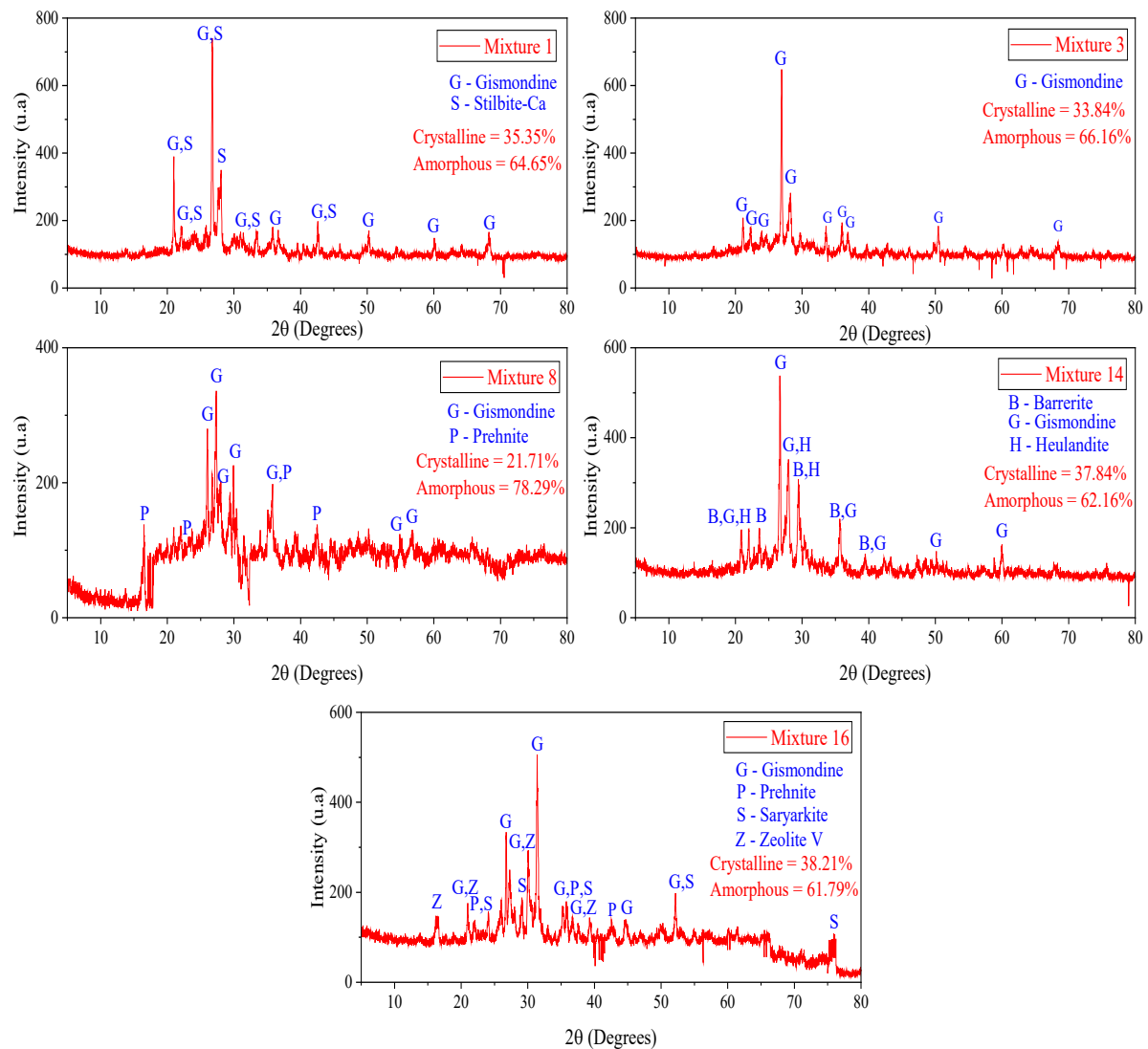


Figure 10. Mineralogical analysis of the sintered mixtures.

Mixture 1, composed of 20% residue sintered at 1075 °C, presented 35.35% crystallinity and 64.65% amorphousness, with phases such as gismondine (G) and Calcium Stilbite (S). In mixture 3, processed at 1145 °C, the crystallinity of gismondine (G) was slightly reduced, reflecting thermal transitions that increased amorphization. This effect results from the partial fusion of silica and calcium, forming an amorphous matrix rich in glassy phases, characteristic of calcium aluminosilicate systems, which favor densification and improve properties such as mechanical strength and reduced water absorption.

Mixture 8 presented 78.29% amorphousness, representing its predominantly glassy matrix with 40% residue and phases such as gismondine (G) and prehnite (P). The high amorphousness, resulting from intense partial melting at 1145 °C, contributed to better mechanical properties and lower water absorption due to reduced pore size and greater particle cohesion, as seen in the physical indices. Prehnite was formed as a stable secondary phase but in smaller quantities, as indicated by the low intensity of the peaks.

In mixture 14, with 37.84% crystallinity and 62.16% amorphousness, phases such as Barrerite (B), gismondine (G), and heulandite (H) were identified, indicating recrystallization during cooling. Rich in glassy phases, this matrix provides intermediate properties, such as good mechanical strength, but with higher water absorption than more amorphous mixtures, such as mixture 8.

Finally, mixture 16, composed of 80% residue and sintered at 1075 °C, presented the highest degree of crystallinity (38.21%) among the samples analyzed, as evidenced by the diffractogram. A greater diversity of crystalline phases indicates a heterogeneous structure with more excellent mineralogical organization. However, this more excellent crystallinity is directly associated with forming highly porous microstructures, characteristic of materials with zeolitic phases and related minerals.

4.8. LWA Application Catalog

Table 3 presents the catalog of industrial applications of LWAs produced with CHT and EFA ceramic waste. Of the 32 mixtures developed, 63 distinct applications were identified, demonstrating the viability of at least one industrial application for waste combinations between 20% and 80% and sintering temperatures of 1075 °C to 1180 °C, also considering the other parameters specified in the experimental program in Table 2.

Table 3. Catalog of industrial applications of LWAs.

Mixture	Does It Meet the Criteria?	Industrial Applications				
		(Δ) High-Strength Concrete	(□) Structural Lightweight Concrete	(◇) Non-Structural Lightweight Concrete, Lightweight Mortars	(○) Geotechnical Applications	(●) Gardening and Landscaping, Thermal and Acoustic Insulation
1	Yes	Δ	□	◇	○	●
2	Yes	Δ	□	◇	○	●
3	Yes	Δ	□	◇	×	×
4	Yes	Δ	□	◇	×	×
5	Yes	×	×	◇	○	●
6	Yes	×	×	◇	○	●
7	No	×	×	×	×	×
8	No	×	×	×	×	×
9	No	×	×	×	×	×
10	No	×	×	×	×	×
11	Yes	×	×	◇	○	●
12	Yes	×	×	◇	○	●
13	No	×	×	×	×	×
14	Yes	Δ	□	◇	×	×
15	Yes	×	×	◇	○	●
16	No	×	×	×	×	×
17	Yes	Δ	□	◇	○	●
18	Yes	Δ	□	◇	×	×
19	No	×	×	×	×	×
20	Yes	Δ	□	◇	×	×
21	Yes	×	×	◇	○	●
22	Yes	×	×	◇	○	●
23	No	×	×	×	×	×
24	No	×	×	×	×	×
25	Yes	Δ	□	◇	○	●
26	No	×	×	×	×	×
27	No	×	×	×	×	×
28	Yes	×	×	◇	○	●
29	No	×	×	×	×	×
30	Yes	×	□	◇	○	●
31	No	×	×	×	×	×
32	No	×	×	×	×	×

Legend: (X) Does not meet pre-established criteria.

It was possible to achieve a specific mass of less than 2.00 g/cm^3 , with water absorption ranging from 0% to 38% and crushing strength between 1.00 MPa and values greater than 5.00 MPa when 20% of waste was incorporated. These conditions met all the pre-established criteria for the analysis, allowing for diverse applications, from non-porous aggregates for high-strength lightweight concretes to porous LWAs for gardening and landscaping, as shown in Figure 11. Additionally, thermal and acoustic insulation applications stood out, especially for temperatures below 1145°C .



Figure 11. Non-porous and porous LWAs for possible industrial applications.

Increasing the waste content from 20% to 60%, combined with temperatures below 1145°C , increased water absorption, making it suitable for lightweight mortars, geotechnical applications, and thermal and acoustic insulation. However, increasing the temperature to 1145°C resulted in a density greater than 2.00 g/cm^3 , while raising it to 1180°C led to the fusion of the samples, causing them to melt, making them unviable for applications.

Finally, incorporating 80% waste demonstrated viability for producing high-strength lightweight concretes with sintering temperatures of 1145°C . However, by reducing the temperature to 1110°C , the applications were expanded to lightweight mortars, gardening, landscaping, and thermal and acoustic insulation. It was observed that, at the analyzed temperature extremes, 1075°C and 1180°C , the materials did not meet the criteria established by the technical standard [1] and the commercial lightweight aggregate catalogs [41–43].

5. Statistical Modeling

5.1. Specific Mass

Figure 12 analyzes the influence of seven experimental factors on the signal-to-noise (S/N) ratio using the Taguchi methodology on particle density. The most impactful factor was the sintering temperature, showing a large variation in S/N ($\Delta = 34.661 \text{ dB}$), followed by the residue content ($\Delta = 12.754 \text{ dB}$) and the pre-sintering temperature ($\Delta = 11.496 \text{ dB}$). These factors exhibited a strong influence on the process performance. In contrast, the aggregate size had a virtually negligible impact ($\Delta = 0.001 \text{ dB}$), making it the least relevant factor regarding the particle density of the LWAs.

From the results of ANOVA, as shown in Table 4, the model achieved a coefficient of determination (R^2) of 93.10% for the training set and an adjusted R^2 of 82.18%, demonstrating strong predictive performance and robustness in estimating particle density during material development.

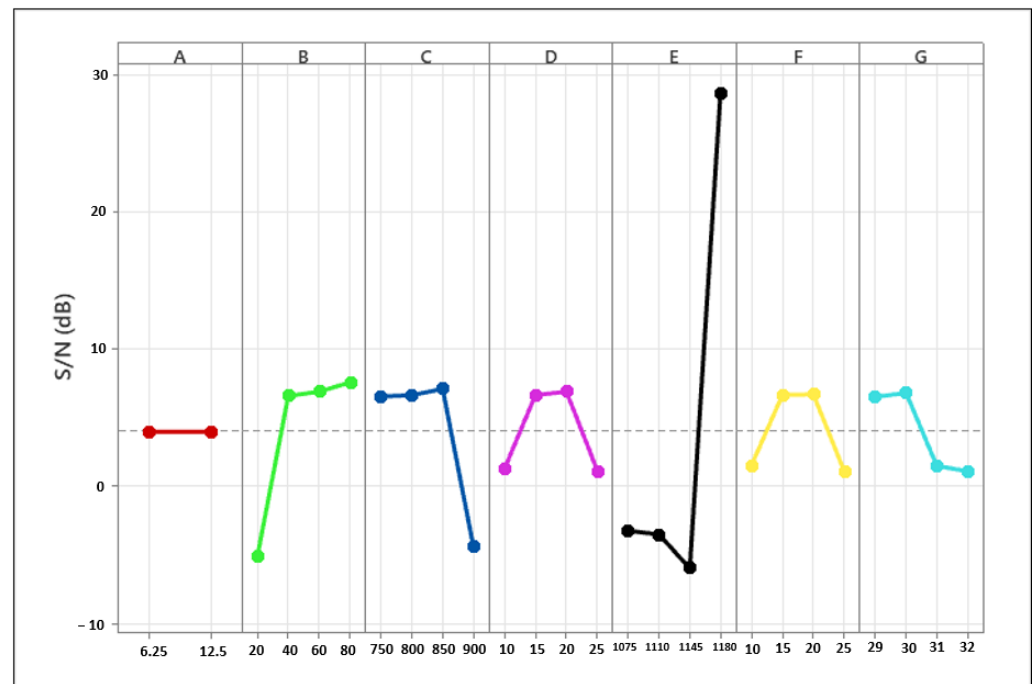


Figure 12. Signal-to-noise (S/N) responses to particle density in mixtures. (A) Aggregate size (mm); (B) Residue content (%); (C) Pre-sintering (°C); (D) Pre-sintering time (min); (E) Sintering (°C); (F) Sintering time (°C); (G) Water content (%).

Table 4. Analysis of variance of specific mass.

Experimental Control Factor	GL	SQ Seq	Contribution	SQ (Adj.)	QM (Adj.)	F-Value	p-Value
Aggregate size (mm)	1	0.0000	0.00%	0.00000	0.00000	0.00	0.995
Residue content (%)	3	2.3939	15.42%	2.39388	0.79796	8.95	0.002
Pre-sintering (°C)	3	1.2265	7.90%	1.22653	0.40884	4.58	0.023
Pre-sintering time (min)	3	0.3898	2.51%	0.38981	0.12994	1.46	0.276
Sintering (°C)	3	9.9211	63.92%	9.92108	3.30703	37.08	0.000
Sintering time (min)	3	0.2691	1.73%	0.26913	0.08971	1.01	0.424
Water content (%)	3	0.2503	1.61%	0.25031	0.08344	0.94	0.454
Error	12	1.0704	6.90%	1.07036	0.08920	-	-
Total	31	15.5211	100.00%	-	-	-	-

The factor with the greatest contribution was the sintering temperature, which was responsible for 63.92% of the total variation, with a p -value < 0.001 , confirming its statistical significance. Next, the residue content (15.42%) and the pre-sintering temperature (7.90%) were also statistically significant, with p -values of 0.002 and 0.023, respectively. On the other hand, factors such as pre-sintering and sintering times, water content, and aggregate size showed very small contributions ($\leq 2.51\%$) and p -values > 0.05 , indicating that their effects were not statistically significant within the tested range. The experimental error accounted for 6.90% of the total variance, which is acceptable, suggesting good experimental precision.

5.2. Water Absorption

Figure 13 analyzes the influence of the signal-to-noise (S/N) ratio on the water absorption of LWAs. The most impactful factor was the sintering temperature, presenting a large variation in the S/N ratio ($\Delta = 58.538$ dB), followed by the residue content ($\Delta = 11.453$ dB) and the sintering time ($\Delta = 10.552$ dB). In contrast, the aggregate size had a practically insignificant impact ($\Delta = 1.316$ dB), becoming the least relevant factor in relation to water absorption.

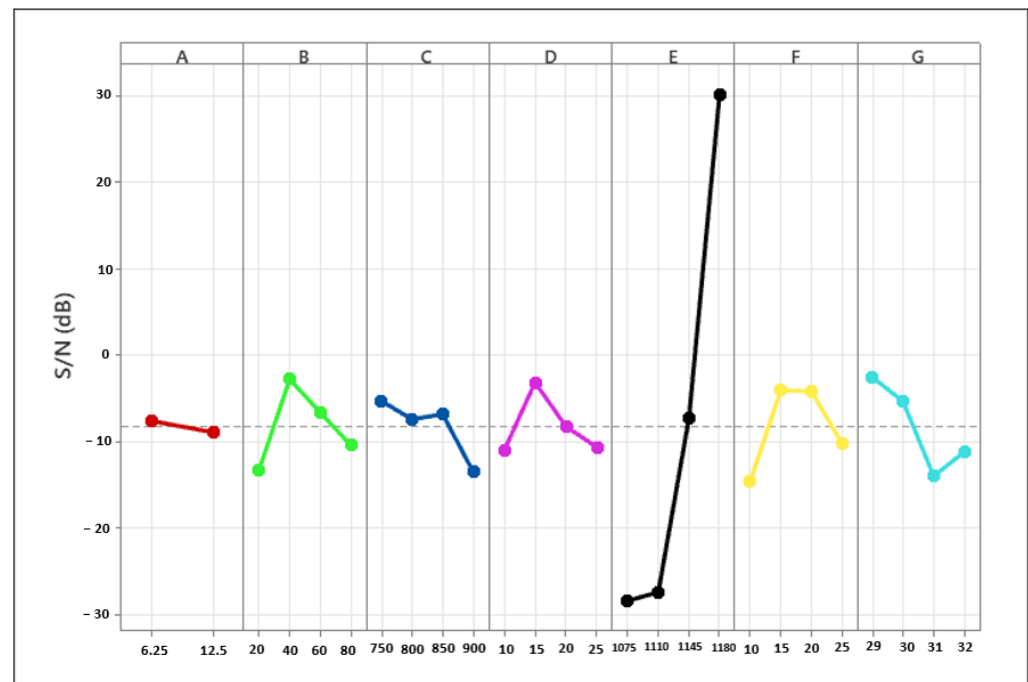


Figure 13. Signal-to-noise (S/N) responses for water absorption in mixtures. (A) Aggregate size (mm); (B) Residue content (%); (C) Pre-sintering ($^{\circ}\text{C}$); (D) Pre-sintering time (min); (E) Sintering ($^{\circ}\text{C}$); (F) Sintering time ($^{\circ}\text{C}$); (G) Water content (%).

From the ANOVA results, presented in Table 5, there was an R^2 of 94.60% and an adjusted R^2 of 86.05% in predicting water absorption during material development.

Table 5. Analysis of variance for water absorption.

Experimental Control Factor	GL	SQ Seq	Contribution	SQ (Adj.)	QM (Adj.)	F-Value	p-Value
Aggregate size (mm)	1	1.17	0.02%	1.17	1.17	0.05	0.835
Residue content (%)	3	614.71	10.71%	614.71	204.90	7.94	0.004
Pre-sintering ($^{\circ}\text{C}$)	3	148.91	2.60%	148.91	49.64	1.92	0.180
Pre-sintering time (min)	3	18.80	0.33%	18.80	6.27	0.24	0.865
Sintering ($^{\circ}\text{C}$)	3	4521.61	78.80%	4521.61	1507.20	58.39	0.000
Sintering time (min)	3	55.67	0.97%	55.67	18.56	0.72	0.560
Water content (%)	3	67.47	1.18%	67.47	22.49	0.87	0.483
Error	12	309.75	5.40%	309.75	25.81	-	-
Total	31	5738.09	100.00%	-	-	-	-

The analysis of variance shows that the sintering temperature is the most relevant factor for water absorption, contributing to 78.80% of the variation, followed by the residue content, with 10.71%. Both factors presented significant p -values, highlighting their importance. Other factors, such as aggregate size and water content, had little influence and non-significant p -values. The experimental model is well-adjusted, with only 5.40% of the variation attributed to error, reinforcing that sintering temperature and residue content are the main controllers of the process.

5.3. Mechanical Resistance

In Figure 14, the most influential factor on the mechanical strength of the LWAs is the sintering temperature ($\Delta = 46.7263$), followed by the residue content ($\Delta = 27.9464$) and the pre-sintering temperature ($\Delta = 15.3837$). These results indicate that these factors have the greatest impact on the response analyzed. Aggregate size showed the least influence

($\Delta = 7.8380$). The S/N values suggest that the optimal levels of each factor may vary depending on the desired application.

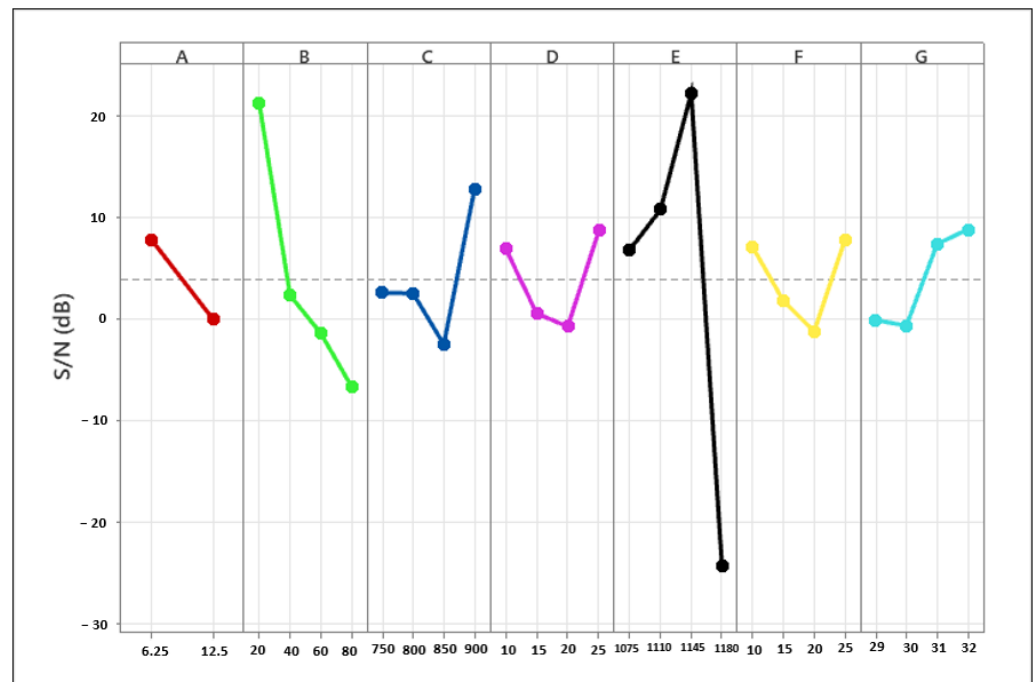


Figure 14. Signal-to-noise (S/N) ratio responses for crushing strength in mixtures. (A) Aggregate size (mm); (B) Residue content (%); (C) Pre-sintering ($^{\circ}\text{C}$); (D) Pre-sintering time (min); (E) Sintering ($^{\circ}\text{C}$); (F) Sintering time ($^{\circ}\text{C}$); (G) Water content (%).

From the ANOVA results, presented in Table 6, there was an R^2 of 97.43% and an adjusted R^2 of 88.96% in predicting mechanical strength during material development.

Table 6. ANOVA analysis for mechanical strength in mixtures.

Experimental Control Factor	GL	SQ Seq	Contribution	SQ (Adj.)	QM (Adj.)	F-Value	p-Value
Aggregate size (mm)	1	421.08	16.63%	421.08	421.08	13.61	0.003
Residue content (%)	3	444.20	17.54%	444.20	148.07	4.78	0.020
Pre-sintering ($^{\circ}\text{C}$)	3	116.23	4.59%	116.23	38.74	1.25	0.335
Pre-sintering time (min)	3	81.98	3.24%	81.98	27.33	0.88	0.477
Sintering ($^{\circ}\text{C}$)	3	956.33	37.76%	956.33	318.78	10.30	0.001
Sintering time (min)	3	105.47	4.16%	105.47	35.16	1.14	0.374
Water content (%)	3	35.77	1.41%	35.77	11.92	0.39	0.766
Error	12	371.38	14.66%	371.38	30.95	-	-
Total	31	2532.43	100.00%	-	-	-	-

Among these, the sintering temperature contributed the most (37.76%) to the variability, showing a highly significant effect with an F-value of 10.30 and a p -value of 0.001. Residue content followed, contributing 17.54%, with a significant p -value of 0.020. Aggregate size also had a notable influence, contributing 16.63%, with a significant p -value of 0.003. Other factors, such as pre-sintering temperature, pre-sintering time, sintering time, and water content, showed lower contributions (ranging from 1.41% to 4.59%) and were not statistically significant based on their p -values (all > 0.05). The error accounted for 14.66% of the total variability, indicating good overall model performance.

According to the results of the S/N ratio and ANOVA analyses, the optimal parameter configuration to achieve the highest particle strength in the mixtures was identified as $A_1B_1C_4D_4E_3F_4G_4$, which are the maximum points in Figure 14. This corresponds to using

an aggregate size of 6.25 mm, 20% waste content, pre-sintering at 900 °C for 25 min, sintering at 1145 °C for 25 min, and a water content of 32%, based on Table 2.

5.4. Response Surface

Contour plots were then generated, as shown in Figures 15–17, for the two leading independent variables, sintering temperature and residue content, obtained from the analysis of the signal-to-noise ratio (S/N). The objective was to evaluate each response based on the essential properties of the lightweight aggregates: specific mass, water absorption, and crushing resistance.

In Figures 15–17, a clear relationship is observed between the sintering temperature, residue content, and the resulting physical and mechanical properties of the sintered materials. At lower sintering temperatures (1075–1110 °C) combined with high residue contents (60–80%), the material exhibits reduced densification. This behavior is evidenced by a specific mass below 1.50 g/cm³, which correlates with a highly porous microstructure. Such porosity is further confirmed by elevated water absorption values, exceeding 35%, and very low crushing strength values, generally below 5 MPa. These conditions suggest incomplete sintering, limited particle bonding, and a loose internal structure.

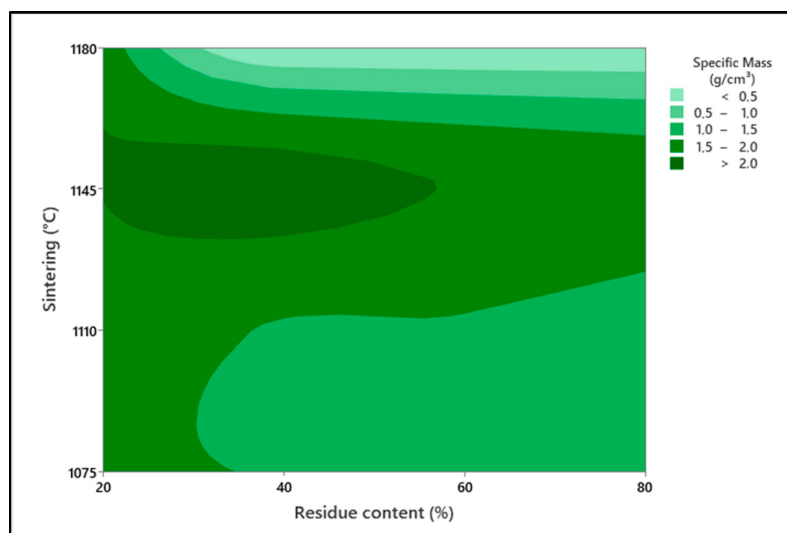


Figure 15. Contour graph of the specific density of the mixtures.

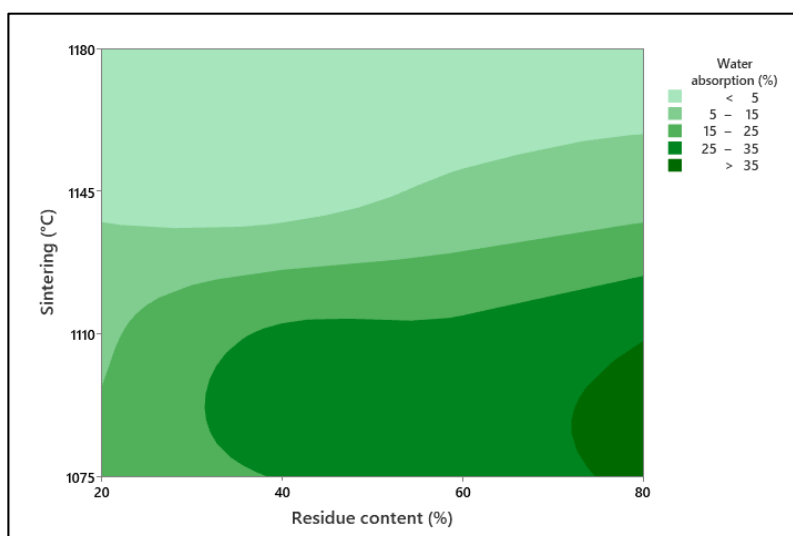


Figure 16. Contour diagram showing the water absorption behavior of the mixtures.

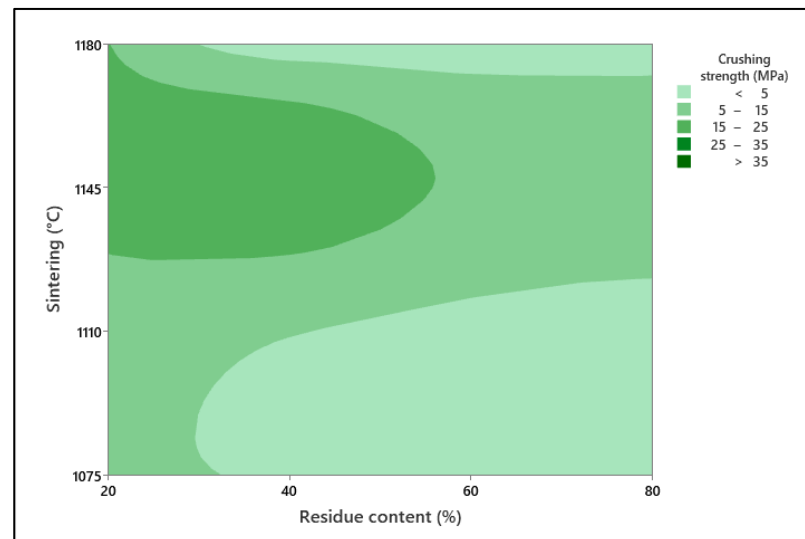


Figure 17. Contour graph of the crushing strength of the mixtures.

On the other hand, at higher sintering temperatures (1145–1180 °C) and lower residue contents (20–40%), the sintering process is significantly more effective, leading to enhanced densification. Under these conditions, the specific mass surpasses 2.00 g/cm³, water absorption drops below 15%, and crushing strength values exceed 35 MPa. This behavior can be attributed to improved diffusion and viscous flow during sintering, which reduce the volume of open pores and promote the formation of a more cohesive and compact microstructure.

Intermediate conditions, such as moderate residue contents (40–60%) and sintering temperatures around 1110–1145 °C, result in transitional behavior, where partial densification occurs. In these regions, specific mass values range between 1.50 and 2.00 g/cm³, water absorption typically varies between 15% and 25%, and compressive strength remains between 15 and 25 MPa. These results emphasize the combined influence of both processing parameters. The residue content likely influences the formation of low-melting phases or inhibits sintering by introducing refractory components, while temperature directly affects the extent of liquid-phase sintering and the overall microstructural development.

Therefore, optimizing both residue content and sintering temperature is critical to achieving lightweight materials with desirable properties, balancing low density and porosity with sufficient mechanical performance.

5.5. Significance of Models

Table 7 presents predictions of optimal values, which are used to validate LWAs' essential properties for different commercial purposes. These purposes include use in structural lightweight concrete and applications related to thermal and acoustic insulation, such as gardening and landscaping.

Table 7. Prediction of optimal values of LWAs for commercial applications.

Application	Property	Mixture	Predictive Value	Observed Value	Error
Structural Lightweight Concrete	Specific Mass (g/cm ³)	A ₁ B ₁ C ₁ D ₁ E ₁ F ₁ G ₁	1.92 g/cm ³	1.81 g/cm ³	−0.11 g/cm ³
	Water Absorption (%)		19.85%	18.67%	−1.18%
	Strength (MPa)		9.25 MPa	10.61 MPa	1.36 MPa
Thermal and acoustic insulation	Specific Mass (g/cm ³)	A ₁ B ₂ C ₁ D ₁ E ₁ F ₂ G ₃	1.37 g/cm ³	1.45 g/cm ³	0.08 g/cm ³
	Water Absorption (%)		25.94%	28.72%	2.78%
	Strength (MPa)		5.30 MPa	7.25 MPa	1.95 MPa

For structural lightweight concrete, the predictive specific mass (1.92 g/cm^3) showed an error of -0.11 g/cm^3 compared to the observed value (1.81 g/cm^3), while water absorption (-1.18%) and compressive strength (1.36 MPa) also showed good agreement. In thermal and acoustic insulation, the predictive specific mass (1.37 g/cm^3) exhibited an error of 0.08 g/cm^3 compared to the observed value (1.45 g/cm^3), with errors of 2.78% in water absorption and 1.95 MPa in compressive strength. These results highlight the model's accuracy and consistency, with errors within acceptable margins.

6. Conclusions

This study evaluated the technical feasibility and environmental potential of producing lightweight aggregates (LWAs) from regional ceramic industrial waste—specifically chamotte (CHT) and eucalyptus firewood ash (EFA)—combined with kaolinite clay (KC). The development of ternary mixtures using these by-products responds directly to the growing demand for sustainable building materials aligned with circular economy principles. The experimental results confirmed that the LWAs produced meet the requirements of the European standard EN 13055-1:2002 [1] and demonstrate physical and mechanical behavior comparable to commercially available aggregates [41–43]. The findings validate an alternative production route for lightweight aggregates based on abundant regional waste, promoting low-carbon, resource-efficient materials for use in construction.

The pyro-expansive behavior of the residues was evident in mixtures with 80% waste content, which reached up to 60% linear expansion at 1075°C and 15% mass loss at 1145°C . Although high residue contents increased porosity and water absorption, compromising mechanical strength, they favored properties desirable in non-structural applications, such as low thermal conductivity and lightweight performance. Aggregates sintered at lower temperatures ($1075\text{--}1110^\circ\text{C}$) with 40–80% waste content are particularly promising for use in insulating mortars, horticultural substrates, and acoustic or thermal barriers. In contrast, mixtures with lower waste content (20%) exhibited higher strength and density, attributed to improved sintering behavior, crystallinity, and compositional stability, making them more suitable for structural and semi-structural uses.

Optimal performance was observed in mixtures containing 40–60% waste sintered at 1145°C , which achieved a favorable balance between porosity, mechanical performance, and volumetric stability. These conditions enabled controlled expansion, sufficient strength, and desirable morphological features such as consolidated particle interfaces and open pore distribution. In addition to their technical performance, the aggregates presented aesthetic versatility: residue content and sintering temperature significantly influenced coloration, ranging from reddish tones at low waste levels to lighter hues at higher ones. This chromatic diversity increases the appeal of these aggregates for architectural and landscaping applications, adding commercial value to their use.

The experimental planning using the Taguchi method demonstrated high predictive accuracy for key response variables, reinforcing its value in process optimization and scale-up. Its robustness contributed to consistent results and efficient exploration of complex interactions among material variables. Furthermore, a comprehensive review of commercial applications identified 63 distinct use scenarios for LWAs across the construction sector, including high-performance and structural lightweight concretes, geotechnical fills, prefabricated components, and specialty mortars. Overall, this study offers a technically sound and environmentally viable pathway for producing sustainable LWAs with broad applicability in modern construction practices.

Author Contributions: J.A.d.S.N.: Writing—review and editing, Investigation, Formal analysis, Data curation. M.A.S.d.A.: Writing—review and editing, Visualization, Validation, Formal analysis, Data curation, Conceptualization. R.P.S.D.: Writing—review and editing, Validation, Supervision, Formal analysis, Data curation. M.M.d.S.: Writing—review and editing, Validation, Supervision, Formal analysis, Data curation. C.M.P.: Writing—review and editing, Validation, Supervision, Formal analysis, Data curation. All authors have read and agreed to the published version of the manuscript.

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