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Analysis of the properties of ceramic glazes with the substitution of granite ornamental rock waste from the Seridó Potiguar region

Análise das propriedades dos esmaltes cerâmicos com a substituição de resíduos de rocha ornamental granítica da região do Seridó Potiguar

Maria Luiza Lima da Silva[1]; Igor Henrique Lima da Silva[2]; Djalma Valério Ribeiro Neto[3] ; Flánelson Maciel Monteiro[4].

[1] Federal Institute of Education, Science, and Technology of Rio Grande do Norte, Natal Central/Diac Campus, Natal, Rio Grande do Norte, Brazil. Email: silva.luiza3@escolar.ifrn.edu.br
ORCID: https://orcid.org/0009-0005-8124-8298

[2] Federal Institute of Education, Science, and Technology of Rio Grande do Norte, Natal Central/Diac Campus, Natal, Rio Grande do Norte, Brazil. Email: h.igor@escolar.ifrn.edu.br
ORCID: https://orcid.org/0009-0001-8420-1066

[3] Federal Institute of Education, Science, and Technology of Rio Grande do Norte, Natal Central/Diac Campus, Natal, Rio Grande do Norte, Brazil. Email: djalma.neto@ifrn.edu.br
ORCID: https://orcid.org/0009-0001-8420-1066

[4] Federal Institute of Education, Science, and Technology of Rio Grande do Norte, Natal Central/Diac Campus, Natal, Rio Grande do Norte, Brazil. Email: flanelson.monteiro@escolar.ifrn.edu.br
ORCID: https://orcid.org/0000-0001-5674-4736

Abstract: Ceramic glazes are widely used in sectors such as artistic ceramics, wall and floor tiles, and sanitary ware. However, due to the increasing scarcity of mineral resources, the search for new alternatives has intensified. This study aims to investigate the replacement of sodium and potassium feldspars, commonly used in ceramic glazes, with ornamental stone waste from the Seridó Potiguar region in the State of Rio Grande do Norte. The stone waste will undergo processing (grinding and sieving) and characterization (XRD and XRF) and will be incorporated into glaze formulations in proportions of 5%, 10%, and 15%. Samples will be tested at temperatures of 1000°C, 1100°C, 1150°C, and 1200°C. The firing process was conducted at a heating rate of 10 °C/min, with a 60-minute dwell time (isothermal hold) at each peak temperature. Technological tests will include water absorption, apparent porosity, linear shrinkage, loss on ignition, and apparent density. The results will allow for the evaluation of the physicochemical performance of the formulations and their viability compared to traditional products, offering a sustainable alternative for the ceramic sector.

Keywords: Ceramic glazes; Rock waste; Seridó Potiguar; Sustainability.

Resumo: Os esmaltes cerâmicos são amplamente utilizados em setores como cerâmica artística, revestimentos e louça sanitária. No entanto, devido à crescente escassez de recursos minerais, a busca por novas alternativas tem se intensificado. Este estudo visa investigar a substituição dos feldspatos de sódio e potássio, comumente utilizados em esmaltes cerâmicos, por resíduos de rocha ornamental provenientes da região do Seridó Potiguar, no Estado do Rio Grande do Norte. O resíduo de rocha será submetido a processos de beneficiamento (moagem e peneiramento) e caracterização (DRX e FRX), e incorporado em proporções de 5%, 10% e 15% nas formulações de esmalte. As amostras serão testadas em temperaturas de 1000°C, 1100°C, 1150°C e 1200°C. O processo de queima foi conduzido sob uma taxa de aquecimento de 10 °C/min, com isoterma de 60 minutos em cada temperatura máxima.Os ensaios tecnológicos incluirão absorção de água, porosidade aparente, retração linear, perda ao fogo e massa específica aparente. Os resultados permitirão avaliar o desempenho físico-químico das formulações e sua viabilidade em comparação com produtos tradicionais, oferecendo uma alternativa sustentável para o setor cerâmico.

Palavras-chave: Esmaltes cerâmicos; Resíduos de rocha; Seridó Potiguar; Sustentabilidade.

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1. Introduction

The manufacture of ceramic materials represented a fundamental milestone in early materials engineering, as it demonstrated to humanity the feasibility of obtaining substances with properties remarkably distinct from those found in nature. Initially, with the creation of vessels and small domestic utensils over ten thousand years ago, this practice evolved throughout history, resulting in the production of a wide variety of ceramic materials (Silva, 2012).

The Northeast region is known for hosting a significant concentration of processing industries, which are responsible for releasing hundreds of tons of waste annually into the environment. This scenario is aggravated by the indicative growth in production, raising concerns among environmentalists and the community in general, due to the potential dangerous and harmful impacts on the environment and public health. In this context, the study of manufacturing ceramic materials with the addition of ornamental rock waste emerges as a promising strategy to mitigate environmental impact and promote sustainability in the ceramic industry (MENEZES, 2002).

According to Silva (2012, p. 1120), "the ceramics area may be the most appropriate technological solution for the recovery and definitive inertization of solid ornamental rock waste, without causing any other type of pollution. It is also highlighted that the incorporation of ornamental rock waste into ceramic bodies, besides contributing to solving the environmental problem, can also contribute to reducing the energy cost of the production process."

The demand for ornamental rocks has always been of a high economic level, such as marble and granite. According to Teixeira (2017), in Brazil, the extraction and processing of ornamental rocks face a significant challenge: the considerable amount of waste generated during the process, due to the low percentage of utilization of the rock block at the extraction site. This volume utilization of the rock, from extraction to obtaining the final product, varies according to the rock type and its intended application.

Almeida (2020) points out that the presence of quartz improves the mechanical strength and durability of the ceramic product, making it more resistant to breakage. The glassy phase formed in the firing process, in turn, improves properties such as corrosion resistance and impermeability, making the products more robust and suitable for various applications in civil construction.

In environmental terms, the reuse of industrial waste, such as that from granite production, is aligned with new environmental legislation and demands for sustainable solutions.

Almeida (2020, p. 2) highlights that:

> "[...] industries have been looking for ways to minimize the waste generated, as well as finding ways to adequately reuse it, mainly due to the new environmental laws in force. Incorporation into ceramic products is a form of treatment for industrial waste, reducing the volume of raw material and immobilizing it in order to avoid negative impacts on the environment. The red ceramic industry has shown interest in developing new materials incorporating waste, which can present better technological properties and thus seek the development of new viable technologies for reusing this waste."

This approach demonstrates the potential of ornamental rock waste as valuable inputs in ceramic production, promoting technical and environmental benefits, in addition to contributing to sustainability in the civil construction sector.

The objective of this work is to analyze the properties of ceramic glazes and how they behave with the substitution of rock waste; it is expected to obtain glazed ceramic pieces with physicochemical and technological properties similar to traditional ceramic products.

2. Methodology

The collection of ornamental rock waste (granite) took place in the municipality of Parelhas/RN, at the company Thor Granitos. As shown in the flowchart, Figure 1, the steps are listed and adopted to analyze the technological properties of the ceramic glazes.

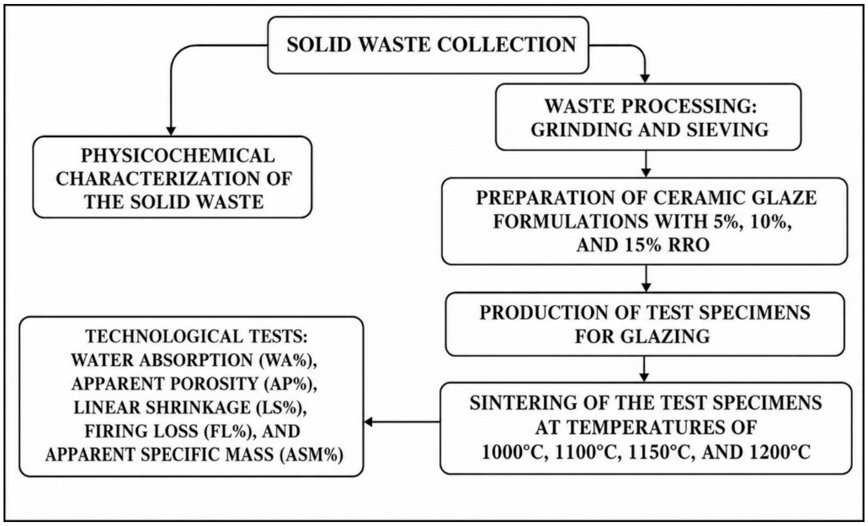

Figure 1 – Flowchart.
Source: Authors (2025).

One of the problems addressed in this research is that the waste generated comes from rock cutting activities, which mostly involve the use of water to lubricate the diamond wire. When this method is used, it results in the formation of a "sludge". In environmental terms, it becomes evident that inadequate disposal of this waste can cause negative impacts on both the environment and the population. Figure 2 shows the in natura rock dust waste from an ornamental rock processing plant.

Figure 2 – Ornamental rock waste from the region of the municipality of Parelhas/RN.
Source: Authors (2025).

For the development of this research, approximately 160 pieces were manufactured, which were subjected to the firing process. After this stage, the samples were glazed. Each glaze formulation was applied to between 38 and 40 pieces, using different proportions of ornamental rock waste (ORW): 0%, 5%, 10%, and 15%. In addition, other raw materials such as clay, albite, and quartz were used, as described in Table 1, where the adopted formulations can be observed.

Consequently, a white-firing clay from the municipality of São Gonçalo do Amarante was used for the production of this work, which was stored in the Materials and Minerals Laboratory (LT2M) at the Federal Institute of Education, Science and Technology of Rio Grande do Norte, Natal Central Campus. Initially, the clay was comminuted in a mortar.

The particle size of the raw materials was determined based on the consideration that this work is dedicated to the structural ceramics sector, in which there is no established standard for the particle size range in the manufacturing process of the products. The characterization of the clay from São Gonçalo do Amarante, in the metropolitan region of Natal, included a sieving process with a #35 mesh sieve. Subsequently, the clay was compacted to produce test specimens using a hydraulic press with a pressure of 21 MPa., com o objetivo de produzir corpos de prova, utilizando uma prensa hidráulica com uma pressão de 21 MPa.

The test specimens, after the sintering process, will be used as supports for the ceramic glazes. In this sense, four ceramic glaze formulations were produced, three of which were composed with ornamental rock waste. Table 1 describes each formulation.

Table 1 – Composition of the produced ceramic glaze formulations.

Glazes/components	Glaze 1 (standard)	Glaze 2	Glaze 3	Glaze 4
ORW	0%	5%	10%	15%
Clay	40%	40%	40%	40%
Albite	40%	35%	30%	25%
Quartz	20%	20%	20%	20%

Source: Authors (2025).

Table 2 presents the formulation scheme and the thermal firing cycle of the ceramic glazes, establishing the relationship between the firing temperatures (1000°C, 1100°C, 1150°C, and 1200°C) and the proportions of Ornamental Rock Waste (ORW) used (0%, 5%, 10%, and 15%). The firing process was conducted at a heating rate of 10°C/min, with a 60-minute dwell time at each peak temperature. For each formulation, a specific sample coding was defined, identified as Glazes (GL), to ensure experimental organization and traceability.

The formulation with 0% ORW (GL1) corresponds to samples 1 to 10, while the 5% (GL2) formulation covers samples 11 to 20. For 10% ORW (GL3), the samples are between 21 and 30, and finally, the formulation with 15% ORW (GL4) corresponds to samples 31 to 40. It is observed that this distribution is maintained in a standardized way for all analyzed firing temperatures.

Table 2 – Formulation and firing scheme.

	Título			
Firing	**0%**	**5%**	**10%**	**15%**
1000°C	1-10 (GL 1)	11-20 (GL 2)	21-30 (GL 3)	31-40 (GL 4)
1100°C	1-10 (GL 1)	11-20 (GL 2)	21-30 (GL 3)	21-30 (GL 4)
1150°C	1-10 (GL 1)	11-20 (GL 2)	21-30 (GL 3)	21-30 (GL 4)
1200°C	1-10 (GL 1)	11-20 (GL 2)	21-30 (GL 3)	21-30 (GL 4)

a) GL - Glaze

Source: Authors (2024).

In the manufacture of the glazes, the particle size of all materials (Clay, Albite, Quartz, and ornamental rock waste - ORW) is in the 200-mesh range. In the glazing scheme, four sinterings (firings) were performed, and the pieces were subjected to technological tests.

The technological tests include the variables used in the mathematical formulas applied to the glazed test specimens.

$$WA\% = \frac{Ww - Dw}{Dw} x\ 100$$ (1) Water Absorption (WA%)

where:

Ww = Wet weight.

Dw = Dry weight.

$$AP\% = \frac{(Wm - Dm)}{Wm - Im} x\ 100$$ (2) Apparent Porosity (AP%)

where:

Dm = Dry mass.

Wm = Wet mass.

Im = Immersed mass.

$$LS\% = \frac{W0 - Wf}{Wf} x\ 100$$ (3) Linear Shrinkage (LS)

where:

W0 = Initial width..

Wf = Final width.

$$LOI\% = \frac{Gw - Dw}{Dw} x\ 100$$ (4) Loss on Ignition (LOI)

where:

Gw = Green weight.

Dw = Dry weight.

$$AD\% = \frac{Dm}{Wm - Im} x\ 100$$ (5) Apparent Density (AD)

where:

Dm = Dry mass.

Wm = Wet mass.

Im = Immersed mass.

3. Results and discussion

The XRD analysis was performed using the X-RAY Diffractometer XDR-7000 through a non-destructive method, reading at an angle of 10° to 80° (2θ). Figure 10 presents the result of the analysis on ornamental rock waste collected at the processing plant in the municipality of Parelhas/RN. This result was first published by Ribeiro et al. (2023) and shared in this work, as the authors are part of the same research and worked with the same waste.

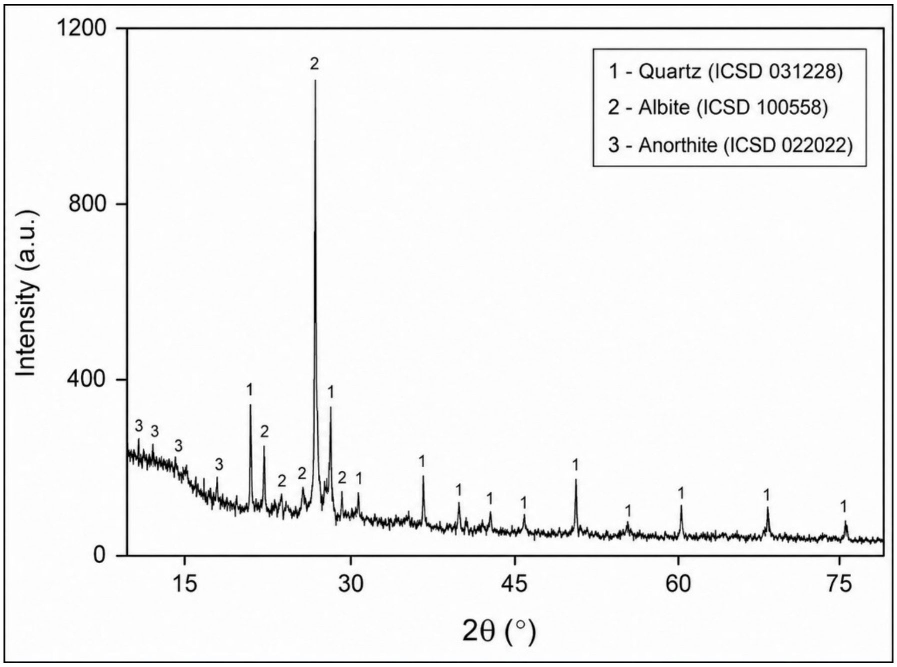

Figure 3 – X-ray Diffraction mineralogical analysis of the rock waste.
Source: Ribeiro Neto et al. (2023). Adapted by the Authors (2023).

From the X-ray diffraction analysis, the predominance of quartz is observed, also highlighting high albite peaks, along with an initial presence of anorthite. This result is important, especially regarding the mineral composition of the waste, which is essential for understanding its properties and potential application in ceramic materials.

X-ray fluorescence was obtained after analysis in a portable XRF. Table 3 presents the results of the analysis performed on the granite ornamental rock waste from the Seridó region.

Table 3 – X-ray Fluorescence analysis of the rock waste.

Óxidos	**SiO2**	**K2O**	**Fe203**	**P2O5**	**Al203**	**CaO**	**TiO2**	**SO3**	**MnO**	**MgO**
%	72,93	8,72	6,16	4,00	3,63	3,19	0,66	0,37	0,32	<LOD

Source: Ribeiro Neto et al. (2023). Adapted by the Authors (2023).

Table 3 shows the percentage composition of several oxides in a sample. SiO_2 is the most abundant oxide, representing almost 73% of the sample's composition. This compound is the main component of silica, a very predominant mineral in the Earth's crust. Following this, K_2O is the oxide with the highest concentration of potassium in the sample, representing almost 9% of the composition. Also present in the sample is Fe_2O_3, with an iron concentration higher than 6%. Fe_2O_3 is an oxide that can impart a red or brown color to minerals and rocks, which may explain the repeated color change in the glazed ceramic pieces.

The following figures present the test specimens at 1000°C, 1100°C, 1150°C, and 1200°C across the different glaze formulations with the solid waste. Figure 7 shows the test specimens sintered at 1000°C. First, the color of the test specimens in their four compositions is observed, where an increase in the amount of ORW leads to a greater darkening of the pieces. The subsequent figures show the other images of the sintered test specimens. It is possible to notice that the test specimens acquire whiteness as the temperature is raised. In Figure 10, showing sintering at 1200°C, the glazed test specimens apparently have a uniform color.

Furthermore, it is observed that there is a color change in the glazed ceramic pieces, making it possible to visualize shades ranging from white and orange, reaching a gray tone. In this sense, at lower temperatures, the bodies take on light and orange tones, while at higher temperatures, they acquire gray tones.

Figure 4 – Sintering of the glazed pieces at 1000°C – (F1).
Legend: a) F1 (1 to 10) - 0% ORW; F1 (11 to 20) - 5% ORW; F1 (21 to 30) - 10% ORW; F1 (31 to 40) - 15% ORW.
Source: Authors (2025).

Figure 5 – Sintering of the glazed pieces at 1100°C – (F2).
Legend: a) F2 (1 to 10) - 0% ORW; F2 (11 to 20) - 5% ORW; F2 (21 to 30) - 10% ORW; F2 (31 to 40) - 15% ORW.
Source: Authors (2025).

Figure 6 – Sintering of the glazed pieces at 1150°C – (F3).
Legend: a) F3 (1 to 10) - 0% ORW; F3 (11 to 20) - 5% ORW; F3 (21 to 30) - 10% ORW; F3 (31 to 40) - 15% ORW.
Source: Authors (2025).

Figure 7 – Sintering of the glazed pieces at 1200°C – (F4).
Legend: a) F4 (1 to 10) - 0% ORW; F4 (11 to 20) - 5% ORW; F4 (21 to 30) - 10% ORW; F4 (31 to 40) - 15% ORW.
Source: Authors (2025).

The white-firing clay in its raw material state exhibits a dark color, indicating the presence of organic matter. When subjected to sintering temperatures, this organic matter is burned off, resulting in greater porosity in the final product (LEGGERINI, 2005). Based on the practice performed, by introducing the glaze with ornamental rock waste onto the test specimens, it is possible to note a greater resistance in the ceramic bodies.

The use of ornamental rock waste in the production of ceramic tiles offers the possibility of increasing hardness and densification. This occurs due to the enrichment of the clay body with quartz present in the waste and the formation of a greater amount of glassy phase during the sintering or firing process of the ceramic. The quartz added to the ceramic body increases the mechanical strength of the final product, making it more durable and resistant to breakage. In addition, the presence of the glassy phase improves properties such as corrosion resistance and impermeability (ALMEIDA, 2020).

The tests performed and the technological analyses encompassed the behavior of the clay together with the ceramic glaze incorporating the ornamental rock waste (ORW). Water absorption (WA%), apparent porosity (AP%), linear shrinkage (LS%), loss on ignition (LOI%), and apparent density (AD, g/cm³) tests were conducted on all test specimens.

Figure 8 shows the result of the apparent density - AD test. Initially, it is noted that the apparent density increases proportionally with the elevation of the sintering temperature of the test specimens, indicating that the materials become denser and less porous as the temperature increases.

The graph also shows how temperature affects the apparent density of the materials. It is observed that the variation in apparent density is greater at higher temperatures, especially between 1150°C and 1200°C, where there is a more pronounced change in values. This suggests that temperature has a more significant effect on the density and porosity of the materials when they are more heated.

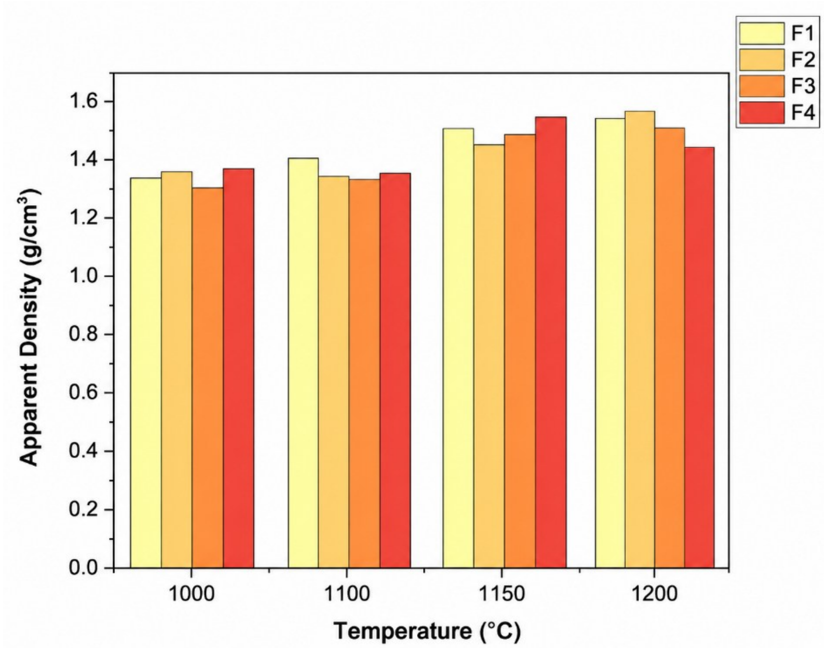

Figure 8 – Result of the Apparent Density – AD technological test.
Legend: a) F1 0% ORW b) F2 5% ORW c) F3 10% ORW d) F4 15% ORW.
Source: Authors (2025).

Figure 9 presents the result of the loss on ignition (LOI%) test. The test was carried out with the test specimens before and after glazing. Formulation F1, which contains glaze with 0% ORW, mostly showed the lowest loss on ignition (LOI%). The only exception where F1 was surpassed is at the temperature of 1000°C, where formulation F4 lost less mass during the sintering process. In general, analyzing the data, it is observed that the formulation with 10% ORW lost more mass during the sintering process. It is also noted that test specimens glazed with a larger amount of ORW waste have a greater loss on ignition. For example, formulation F2, with 5% ORW in the glaze, loses proportionally more mass than F1 and less than the other formulations.

Mass loss during the sintering process can occur due to a greater microstructural organization of the particles in the glassy phase formation stage, for example. Because the test specimens are being coated with glazes that have solid waste in their composition, this material will influence the chemical composition of the glazes.

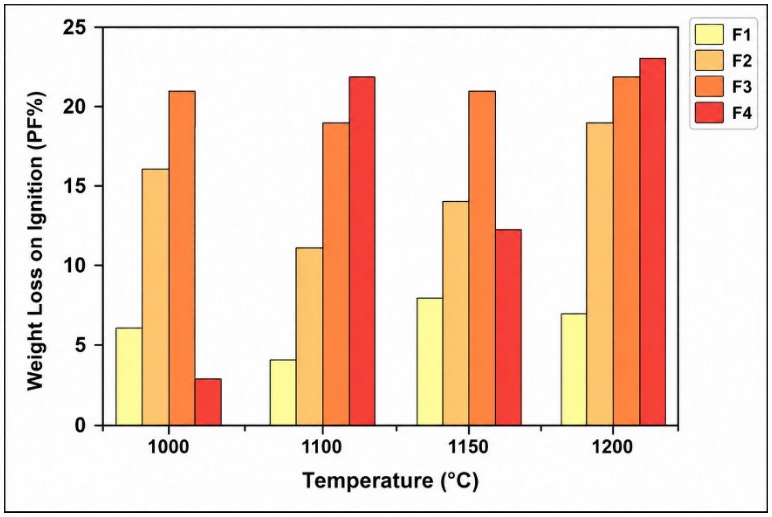

Figure 9 – Result of the Loss on Ignition – LOI% technological test on glazing.
Legend: a) F1 0% ORW b) F2 5% ORW c) F3 10% ORW d) F4 15% ORW.
Source: Authors (2025).

Figure 10 shows the result of the apparent porosity technological test. By analyzing the graph, it can be seen that F1, with 0% ORW, is the most porous formulation, presenting the highest percentage across temperatures.

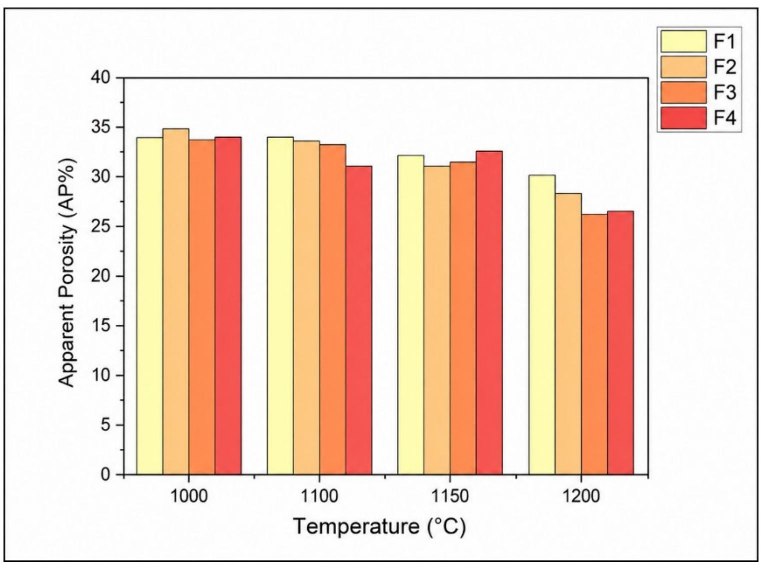

Figure 10 – Result of the Apparent Porosity – AP (%) technological test.
Legend: a) F1 0% ORW b) F2 5% ORW c) F3 10% ORW d) F4 15% ORW.
Source: Authors (2025).

F4, on the other hand, with 15% ORW, is the formulation with the lowest porosity. The samples followed the established pattern, where increasing the sintering temperature results in lower apparent porosity, given that there is a decrease in pores due to the formation of glassy phases.

Figure 11 presents the result of the linear shrinkage test of the test specimens. When analyzing the data from the technological test, it is observed that most samples did not shrink; on the contrary, there was linear expansion. Therefore, the presented result evidences the occurrence of an unusual phenomenon, pyroexpansion.

Zandonadi (1995) explains this phenomenon as follows:

> [...] The pyroexpansion phenomenon can be explained by the decomposition into new mineralogical forms of some minerals as they undergo an increase in temperature while simultaneously reacting with fluxing materials, forming liquid silicates or glasses. Still according to the author, the intense formation of glass blocks the pores before the release of all gases occurs, which can cause the expansion of the body, characterizing what is called pyroexpansion or thermal swelling.

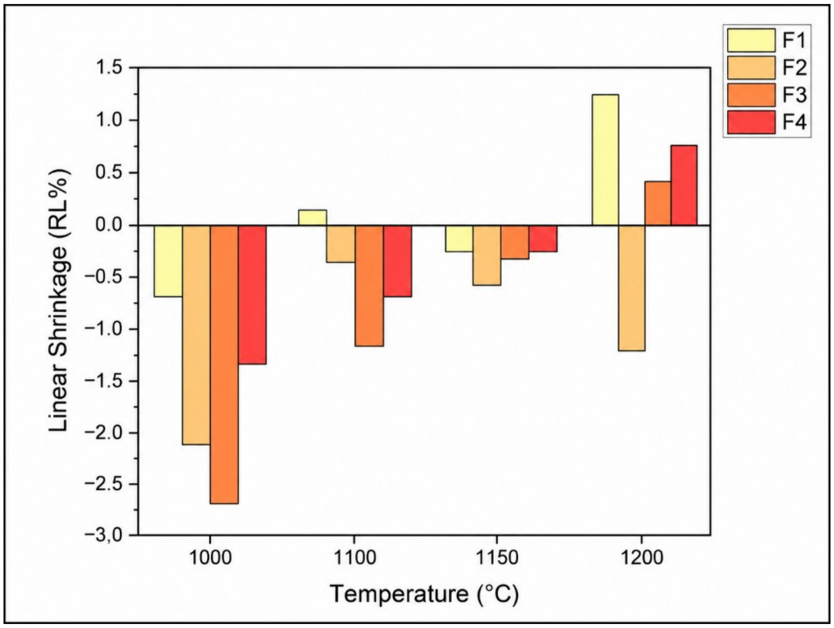

Figure 11 – Linear Shrinkage – LS (%) technological test.
Legend: a) F1 0% ORW b) F2 5% ORW c) F3 10% ORW d) F4 15% ORW.
Source: Authors (2025).

Figure 12 shows the result of the water absorption (WA%) test of the sintered test specimens. The water absorption and apparent porosity tests are generally proportional in ceramic materials. This fact is verified in this work. The data presented also show that the test specimens decrease the WA% index as the sintering temperature increases. This is primarily the result of the formation of glassy phases at higher temperatures and the reorganization of particles.

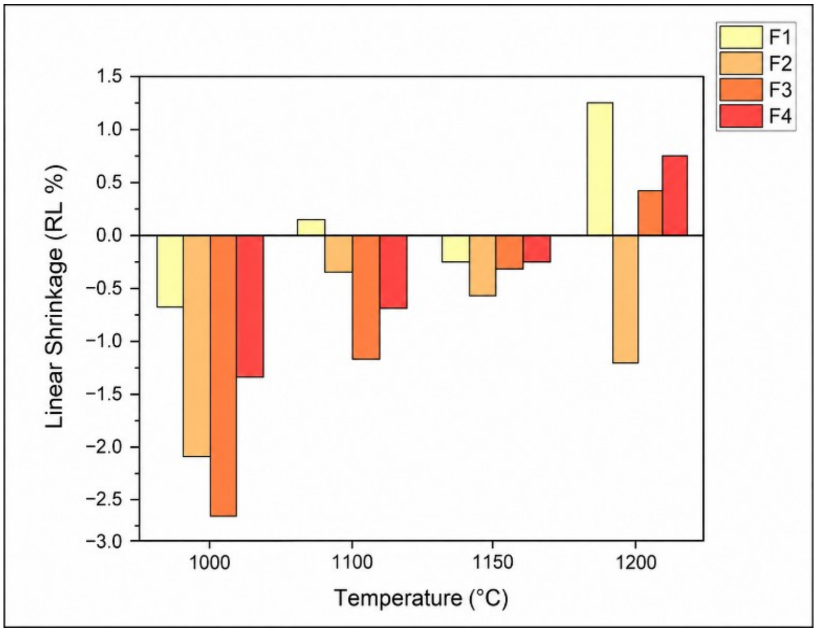

Figure 12 – Linear Shrinkage – LS (%) technological test.
Legend: a) F1 0% ORW b) F2 5% ORW c) F3 10% ORW d) F4 15% ORW.
Source: Authors (2025).

The samples with 0% ORW waste, F1, absorb more water at all temperatures, indicating they are the most porous and permeable. In contrast, F3 and F4 are the formulations that present the smallest variation in water absorption as a function of temperature, remaining between 10% and 15% at all temperatures. However, it is important to highlight that at 1200°C, all four formulations present lower values compared to the other temperatures.

4. Final considerations

The studies conducted aim to evaluate the potential of ornamental rock waste (ORW) in the formulation of ceramic glazes and its impact on product properties. The results indicate that the ORW, composed mainly of quartz and albite, demonstrates vitrifying properties, providing benefits such as good sintering and adherence to the glazes. The presence of the waste in the ceramic pieces resulted in greater mechanical strength, lower water absorption, and reduced porosity compared to unglazed pieces.

The technological tests provided a comprehensive understanding of the behavior of the ceramic materials under different conditions, highlighting the significant influence of temperature and composition on the final properties. The pyroexpansion observed during linear shrinkage and the decrease in water absorption with increasing temperature are crucial aspects for optimizing processes and formulations. It is emphasized that, as analyzed, the glazes produced with ORW, if used industrially, would not be recommended for low-temperature glazes or for the manufacture of porcelain tiles. At most, they could be used for porous ceramic wall tiles, with water absorption exceeding 10%.

The variation in color of the ceramic pieces glazed with ORW at different temperatures and waste concentrations highlights the versatility of this material in diversification, offering shades of white, orange, and gray. We conclude that ORW is a promising material for the production of ceramic glazes and can significantly contribute to the valorization of solid mining waste and the expansion of ceramic products.

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