

Influence of rice husk ash calcination temperature in determining compressive strength and microstructure of mortar

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Abstract

The construction industry consumes large quantities of cement and is therefore a major source carbon dioxide (CO₂) emissions. As a more sustainable alternative, rice husk ash (RHA) can partially replace cement, taking advantage of its pozzolanic properties. The reactivity of RHA varies depending on the calcination temperature, affecting the strength and microstructure of the mortar (MM). This study evaluates how calcination temperature of RHA affects compressive strength and MM. RHA was heat treated at 600, 650, 700 and 750 °C to analyze the compressive strength of the mortar. Mortar specimens were prepared with RHA replacing cement at 5, 10, 15 and 20% to replace cement and compressive strength tests were performed at 7, 14, 21 and 28 days. In addition, the MM was characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and Thermogravimetric Analysis (TGA) were used to evaluate the MM. The results indicate a maximum gain in compressive strength up to 55.87% when 15% of the cement was replaced with RHA calcined at 700 °C, whose amorphous silica content was 69.40%. Moreover, microstructural analyses evidenced the formation of C-S-H and C-A-S-H gels, which densified the mortar, improved the interfacial zone (ITZ), and reduced overall porosity. It is concluded that the calcination temperature significantly influences the pozzolanic reactivity of RHA, the mechanical strength, and MM; RHA calcined at 650 - 700 °C exhibited the best mechanical performance, attributable to higher amorphous silica content and greater microstructural densification. It is recommended for future works to investigate calcination temperatures above 750°C, analyze the relationship between RHA particle size and its amorphous silica content, and evaluate the mortar's long-term durability. In addition, studies should assess the interaction of RHA with other alternative materials to identify synergistic effects and optimize the RHA dosage.

Keywords: Construction industry, Rice husk ash (RHA), Calcination temperature, Compressive strength, Microstructure

1. Introduction

Despite advances in the use of RHA as an alternative material for concrete production, there is a need to deepen our understanding of how calcination temperature influences the pozzolanic activity of RHA and the mechanical properties of mortar. Although several studies have shown that partial replacement of cement with RHA calcined at specific temperatures can improve the compressive strength of mortar, there is no clear consensus on the optimal calcination temperature range that maximizes pozzolanic activity while maintaining the structural integrity of mortar. Furthermore, the relationship between the particle size of RHA and its amorphous silica content is not well established, which prevents the establishment of precise guidelines for its application in the construction industry. Furthermore, the effect of heat treatment on the microstructural properties of mortar, especially with regard to the formation and distribution of C-S-H and C-(A)-S-H gels in the interfacial transition zone, requires further research. Based on the review, it was found that there are numerous studies on the use of RHA in mortars and concretes, whose properties depend both on the source (chemical composition) and the calcination temperature.

However, most studies have focused exclusively on evaluating the mechanical strength of mortars incorporating RHA, without including complementary analyses such as X-ray diffraction (XRD) and scanning electron microscopy (SEM) of both the RHA itself and the resulting mortars made with it. Therefore, the main objective of this study is to evaluate the influence of RHA calcination temperature on the compressive strength and microstructure of mortar, establishing parameters such as the optimum calcination temperature and optimum RHA content to determine the most suitable combination. In summary, despite advances in the application of RHA as a cement substitute, there are still gaps in knowledge regarding the precise relationship between calcination conditions, the resulting microstructure, and the mechanical properties of the mortar, which are crucial aspects for more efficiently exploiting the

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pozzolanic properties of RHA and ensuring its viability as a sustainable material in construction.

According to Prayuda et al. [1] state that rapid urbanization and industrialization have increased the demand for infrastructure and housing worldwide, in this context, Jeffy et al. [2] highlight that the construction industry is one of the main sources of pollution and global warming, cement use being a significant contributor to this problem. This view is supported by Hamada et al. [3] who point out that environmental degradation continues to increase due to the depletion of raw materials and the growing demand for concrete products. Therefore, it is crucial to emphasize that researchers and scientists are concentrating efforts on developing eco-friendly concretes by using renewable materials [4]. In this sense, the development of sustainable building materials is essential to reduce environmental pollution, during both the production and operational phases of their life cycle. This approach has aroused great interest in the construction industry globally [5]; as a consequence, the industry has driven the advancement of alternative materials that promote economic and sustainable development [6], leading designers and planners to explore innovative options for application in construction [7].

Lu et al. [8] claim that the use of local materials is an effective strategy to address raw material shortages and save energy. However, excessive use of these materials may compromise the strength of the products. To ensure the strength of cement-based materials and maximize the use of local resources, additives such as fly ash (FA) are often incorporated, improving both the mechanical properties and sustainability of concrete, reducing costs, and contributing to efficient waste management [9]. A prominent example is the incorporation of RHA in the production of concrete, which not only contributes to saving natural resources by reducing the amount of cement required [10], but also minimizes or even eliminates the alkali-silica reaction (ASR) between aggregates and cement paste, thanks to the pozzolanic properties of RHA [11], in addition, RHA contains a high concentration of amorphous silica, which ensures that the strength of concrete is not compromised when cement is partially substituted [12]. This substitution also has the additional benefit of reducing CO₂ emissions, favoring a more sustainable construction [13].

Tanash et al., [14] report that exposure to high temperatures significantly affects the mechanical properties of concrete, underscoring the importance of investigating how calcination can optimize the characteristics of RHA. For the RHA exhibit strong pozzolanic characteristics, it is necessary to control the calcination temperature, and perform compressive strength tests to analyze the pozzolanic activity, thereby achieving better mechanical performance when replacing cement with RHA in the concrete production [15]. In this regard, thermal treatment of RHA increases the pozzolanic performance by reducing loss on ignition (LOI), as verified by Rithuparna et al. [16], who reported an optimum combustion temperature of RHA that ranges between 500 and 700 °C, and noted that ash crushing improves the pozzolanic activity index. Consistently, Mostafa et al. [17] showed that the compressive strength of mortar incorporating RHA depends mainly on RHA particle size and the content of amorphous silica content.

Thiedeitz et al. [18] reported that, 25% substitution of cement by RHA calcined at 650 °C, the compressive strength increased by 14.29% relative to the standard mortar, with the RHA exhibiting an amorphous silica content of 94.97%. Similar results were reported by Muñoz Perez et al. [19] substituting 20% the cement with RHA calcined at 700 °C increased the compressive strength by 23.80% relative to the control mortar, with an amorphous silica content of 71.50%. Likewise, Wang et al. [20] reported that with 10% RHA the compressive strength increases 15% with a silica content of 89.16%, and the flowability decreased as the RHA content increased. They also observed three types of hydrates, including C-S-H gels and two forms of (Na, K)-S-H gels. The high specific surface area of RHA particles favors cement hydration and the formation of C-S-H and C-(A)-S-H gels within the paste pores, improving the interfacial transition zone bonding, which led to a reduction of cracks [21, 22] and, consequently, a significant improvement in the mechanical characteristics of the concrete [23].

In this context, the use of RHA has also been investigated by Guo et al. [24], who found that combining it with calcined oyster shell powder (OCSP) resulted in a 20% increase in compressive strength compared with the control mortar, although this benefit decreases when certain incorporation percentages are exceeded. This decrease is due to the limited availability of calcium hydroxide, which is essential for the pozzolanic reaction, and affects the quality of the material when the pozzolanic material content is exceeded [25]. Therefore, it is crucial to optimize the proportions of these materials to avoid adverse effects on the strength and durability of the mortar while maintaining the sustainability of the process [26].

The main chemical composition of RHA is silica, with cristobalite and quartz as the predominant crystalline phases [27]. This finding was confirmed in the study by Muñoz Pérez et al. [19] which reported concentrations of 20.7% cristobalite, 7.7% quartz, and 71.6% amorphous crystalline phase. Likewise, the study by Lima & Chagas [28], the results confirmed the presence of fully amorphous silica in RHA.

Based on the literature reviewed, there are knowledge gaps regarding how the calcination temperature of RHA influences its pozzolanic reactivity and the mechanical properties of mortar. Although temperatures between 600 and 700 °C have been identified as the most beneficial, there is no consensus on the optimal range that maximizes the strength and durability of mortar. Many studies have focused only on mechanical strength, without exploring in detail the microstructure of RHA and mortar using techniques such as DFRX, SEM, EDS, TGA, and FTIR. Studies such as those by Ji et al. [29] use XRF, SEM, and XRD, but omits FTIR and TGA, while Jaya et al. [30] incorporate FTIR and TGA, but not SEM or EDS. For their part, Muthukrishnan et al. [31] explore the use of rice husk biochar combined with RHA to improve mortar durability but do not analyze the impact of calcination temperature on the reactivity of the treated RHA. This lack of studies on the interaction between calcination temperature and biochar creates a knowledge gap that limits the optimization of the mechanical, microstructural, and durability properties of mortar. This highlights the need for further research into these techniques to improve the use of RHA as a sustainable material in construction.

2. Materials and methods

2.1 Materials

2.1.1 Aggregate and water

Their physical properties, including granulometry and density, were evaluated, with results presented in Table 1, to assess suitability for the mix design [32]. For mix preparation and curing of the hardened samples, tap water was used in accordance with ASTM C1602 [33], ensuring proper hydration and optimal strength development.

Table 1 Physical characteristics of aggregates

Aggregate characteristics	Fine aggregate	ASTM Standard
Specific gravity	2.60	ASTM C-128 [34]
Dry loose unit weight Kg/m ³	1,54	ASTM C-29 [35]
Percentage of absorption %	1.58	ASTM C-127 [36]
Moisture content %	2.03	ASTM C-566 [37]
Fineness modulus	3.00	ASTM C-136 [32]

2.1.2 Rice husk ash

RHA is an agricultural by-product derived from the combustion of rice husks, used as fuel. This material contains compounds with pozzolanic and cementitious properties, making it a potential additive to improve concrete strength when mixed with cement and water, owing to its high pozzolanic activity. Its main components include silica and alumina, the proportions of which depends significantly on the calcination temperature. Therefore, it is essential to perform a analysis of the thermal conditions of calcination to maximize the amounts of silica and alumina available, thus optimizing its effectiveness as a supplementary material in cementitious mixtures [38]. In this research, pozzolanic activity was evaluated in accordance with ASTM C618 [39]. The RHA samples were subjected to calcination at temperatures of 600, 650, 700 and 750 °C; they were then ground and sieved through a #325 mesh (45 µm), as illustrated in Figure 1.

**Figure 1** RHA passing #325 mesh at 600, 650, 700 and 750 °C.

To evaluate the characteristics of the obtained ash, an X-ray fluorescence (XRF) analysis was conducted, and the results are presented in Table 2 and Figures 2 to 5. The X-Ray Fluorescence analysis of the calcined RHA at different temperatures (600°C, 650°C, 700°C and 750°C) show that silica is the predominant component, together with other elements such as K, Ca, Fe, and Mg. At 600°C and 650°C, silica remains mostly amorphous, favoring its pozzolanic reactivity. At 700°C, a transition to higher crystallinity begins, and at 750°C the crystalline phase intensifies, reducing its reactivity. This suggests that temperatures between 600°C and 700°C are optimal for obtaining highly reactive ash.

The XRF spectra reveal significant peaks corresponding to elements such as iron (Fe), silicon (Si), and potassium (K), whose concentrations decrease as the calcination temperature increases, according to the data in the table. For example, iron decreases from 9.788% at 600°C to 2.671% at 750°C, and silicon, although decreasing, maintains its predominant presence. Potassium shows a slight decrease from 18.268% at 600°C to 17.658% at 750°C. Elements such as calcium, manganese, and zinc are also identified in the spectra, with their concentrations reflected in the table. In summary, the XRF spectra validate the results in the table, showing the variation in element concentrations as the temperature increases, evidencing chemical processes and volatilization in the material

Table 2 Chemical composition of the RHA expressed in elements.

Component	Unit	LCM	Result			
			600 °C	650 °C	700 °C	750 °C
Silicium, Si	%	0.001	62.992	69.794	69.401	66.714
Potassium, K	%	0.001	18.268	16.688	17.835	17.658
Iron, Fe	%	0.001	9.788	4.919	4.518	2.671
Calcium, Ca	%	0.001	3.742	3.455	3.053	6.441
Chlorine, Cl	%	0.001	2.383	2.377	2.481	1.985
Sulfur, S	%	0.001	0.948	1.005	1.159	1.626
Manganese, Mn	%	0.001	0.724	0.725	0.843	1.183
Phosphorus, P	%	0.001	0.655	0.599	0.334	1.288
Titanium, Ti	%	0.001	0.166	0.113	0.099	0.105
Zinc, Zn	%	0.001	0.114	0.112	0.114	0.161
Chrome, Cr	%	0.001	0.076	0.093	0.017	0.040
Strontium, Sr	%	0.001	0.047	0.037	0.039	0.048
Rubidium, Rb	%	0.001	0.046	0.034		0.035
Bromo, Br	%	0.001	0.028	0.025	0.026	0.018
Copper, Cu	%	0.001	0.023	0.024	0.024	0.027
Zirconium, Zr	%				0.018	

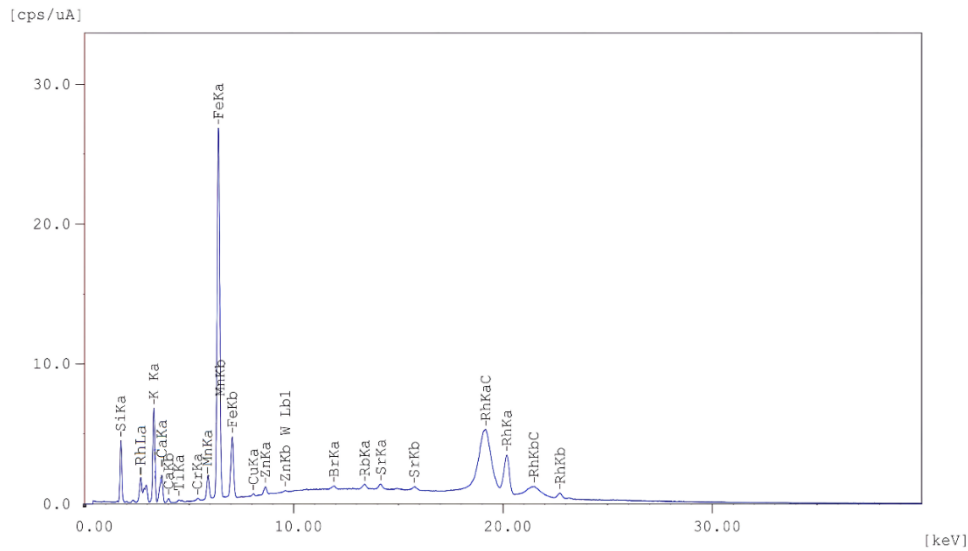


Figure 2 X-ray fluorescence testing on RHA at 600°C

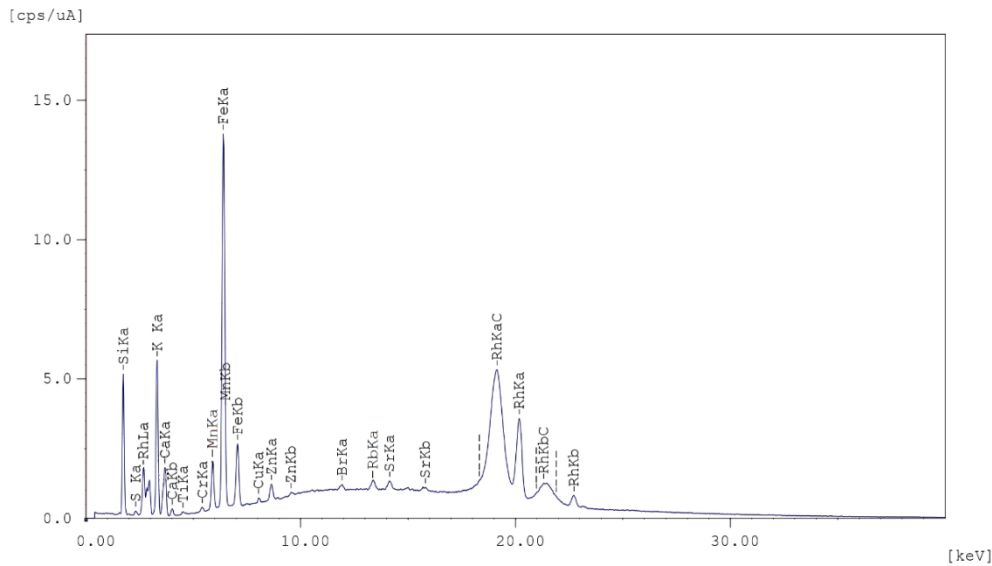


Figure 3 X-ray fluorescence testing on RHA at 650°C

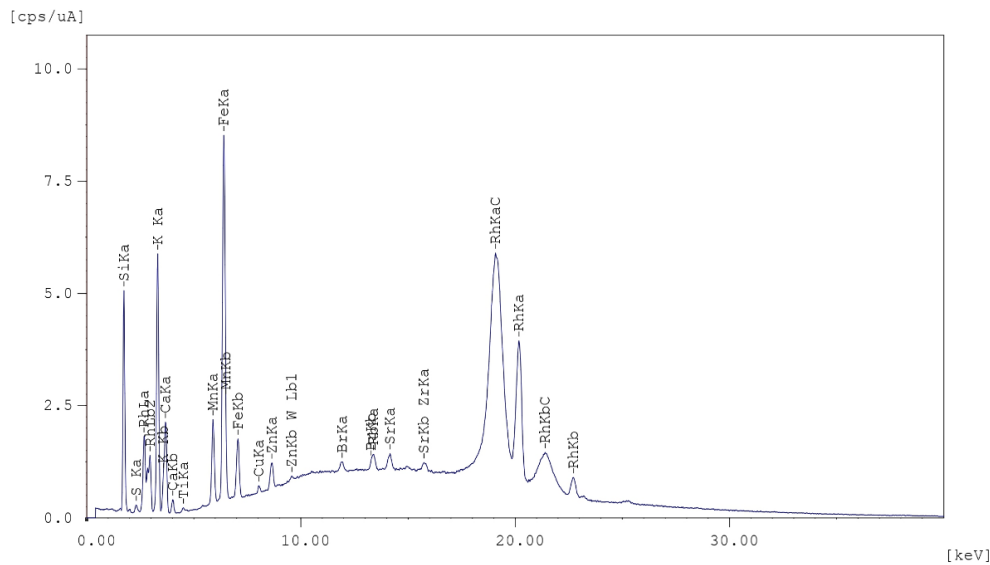


Figure 4 X-ray fluorescence testing on RHA at 700°C

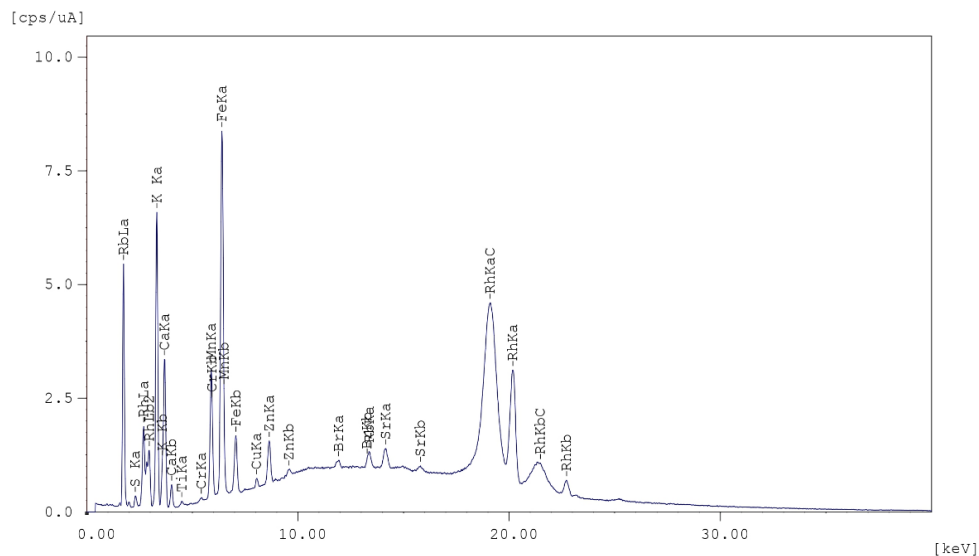


Figure 5 X-ray fluorescence testing on RHA at 750°C

2.2 Methods

The following steps were taken to carry out this research: 1) A complete evaluation of the fine aggregate used in the mortars, passing through mesh No. 04 (4.75 mm), was carried out. This included the determination of its granulometry and physical properties, such as specific gravity, dry loose unit weight, absorption, and fineness modulus. 2) The rice husk was subjected to a calcination at four different temperatures: 600, 650, 700 and 750 °C. The resulting ash obtained after calcination were crushed to homogenize their size and subsequently sieved using a #325 mesh (45 µm) to ensure a uniform distribution of fine particles. A standard mortar composed only of water, fine aggregate and cement was designed, which served as the basis for subsequent comparisons. Following the design of the standard mortar, mixtures were prepared in which 5, 10, 15 and 20% of the cement was replaced by sieved RHA, corresponding to each of the four calcination temperatures (600, 650, 700 and 750 °C). The compressive strength of the mortar samples was evaluated at 7, 14, 21 and 28 days of curing, following the established standards. 3) X-ray diffraction analyses were performed to characterize the crystalline composition of the calcined ash, in order to identify the compounds present and evaluate their pozzolanic activity. Scanning electron microscopy was used to analyze the microstructure of both the standard mortar and the optimum mortars of each RHA temperature at 28 days of curing. This allowed observation of the interactions between the materials and evaluation of the particle distribution in the mixes. This comprehensive approach allowed the determination of the optimum RHA calcination temperature, as well as its impact on the mechanical and microstructural properties of the mortar. The above is detailed in Figure 6.

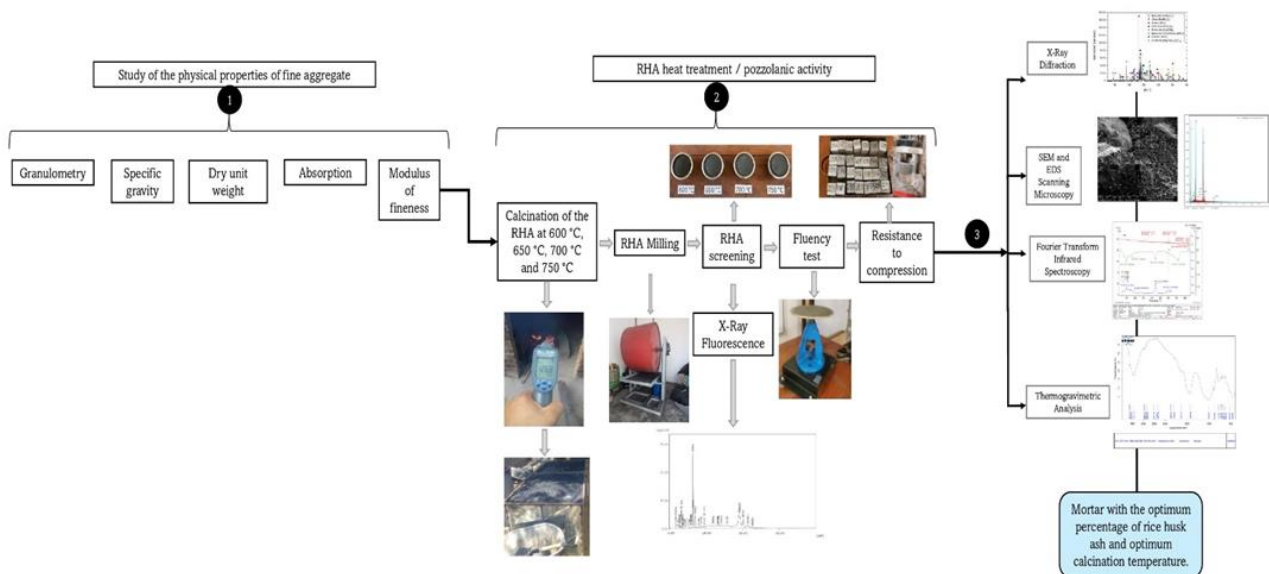


Figure 6 Flowchart of the present investigation

2.2.1 Mix proportions and test methods of mortar

Pozzolanic activity was determined using ASTM C618 [39]. Standard 5 cm × 5 cm × 5 cm mortar cubes were manufactured with a w/c ratio of 0.49. Then, 5, 10, 10, 15 and 20% by weight of cement were replaced with RHA at the four calcination temperatures considered in this study. The specimens were then tested for simple compressive strength under ASTM C39/C39M [40] at a loading

rate of 0.25 ± 0.05 MPa/s, as shown in Figure 7. The mixing ratios of the mortar cubes to determine the pozzolanic activity/calcination temperature are shown in Table 3.

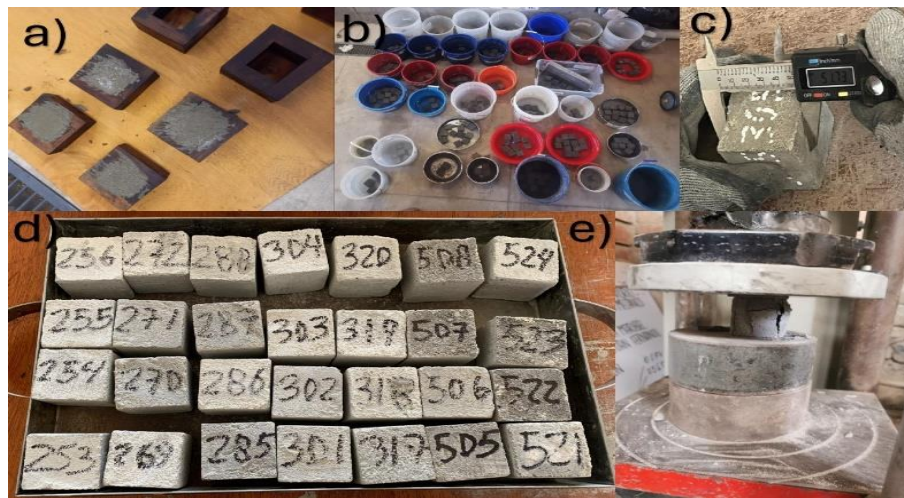


Figure 7 Production and breakage of mortar cubes: a) mortar processing, b) mortar curing, c) mortar cube measurements, d) mortar cube coding, e) mortar cube breakage.

Table 3 Proportion of materials per m^3 for the production of mortars.

Sample	Temperature ($^{\circ}\text{C}$)	Cement (Kg/m^3)	Water (Lt/m^3)	Sand (Kg/m^3)	RHA (Kg/m^3)
M0	600	729.7	357.5	1570.3	
M1		693.2	450	1570.3	36.5
M2		656.7	480	1570.3	73.0
M3		620.4	500	1570.3	109.3
M4		583.7	510	1570.3	146
M5	650	693.2	460	1570.3	36.5
M6		656.7	480	1570.3	73.0
M7		620.4	500	1570.3	109.3
M8		583.7	510	1570.3	146
M9	700	693.2	460	1570.3	36.5
M10		656.7	480	1570.3	73.0
M11		620.4	500	1570.3	109.3
M12		583.7	510	1570.3	146
M13	750	693.2	460	1570.3	36.5
M14		656.7	480	1570.3	73.0
M15		620.4	500	1570.3	109.3
M16		583.7	510	1570.3	146

2.2.2 Microstructural characteristics

2.2.2.1 X-ray diffraction of mortar

From the supplied samples, X-ray diffraction analysis was performed using a Bruker D8 Focus with copper radiation ($\text{CuK}\alpha=0.15418$ nm), 40 mA current and 40 kV accelerating voltage, with a Lynxeye detector with energy selectivity. The analysis was performed over a 2θ from 5° to 60° , with 0.02-degree steps. The time per-step was 1 s.s.

The Reference Intensity Ratio (RIR) method was used to calculate the composition of the crystalline phases and the amorphous part. The minimum concentration for this method is 0.1 wt%

2.2.2.2 SEM and EDS scanning microscopy of mortar

For this test, SEM and EDS were conducted in accordance with ASTM E2809 [41]. The scanning electron microscopy (SEM) analysis was performed using an FEI electron microscopy equipment model Quanta 650 for which an accelerating voltage of 20 kV and a spot size of 3 was applied. The energy dispersive spectroscopy (EDS) was performed using an EDAX detector, mounted on the microscope; for data processing and determination of the chemical composition, the EDAX TEAM software and the eZAF matrix correction algorithm were used.

2.2.2.3 Fourier Transform Infrared Spectroscopy (FTIR) of mortar

Samples were evaluated in accordance with ASTM E1252 [42]. The analysis was carried out using a Bruker Tensor 27 FTIR Infrared Spectrometer, with an uncertainty of ± 0.81 cm^{-1} , considering a coverage factor $K=2$ for a confidence level of 95%. For the

evaluation, a potassium bromide (KBr) pellet was prepared in a 20:1 ratio with the analyzed sample, reaching a total mass of approximately 50 mg

2.2.2.4 Thermogravimetric Analysis (TGA) of mortar

The analysis was performed following ASTM E1131 [43] and ASTM D3418 [44], in a temperature range of 30 to 850°C with a heating rate of 10°C/min. During the process, a purge gas with a flow rate of 50 ml/min was employed, using nitrogen (N₂) up to 600°C and oxygen (O₂) from 600 to 850°C.

2.2.3 Statistical analysis

To evaluate the effects of calcination temperature and RHA replacement percentage on compressive strength, an Analysis of Variance (ANOVA) was performed to determine whether statistically significant differences were present among treatments. A p-value less than 0.05 ($p < 0.05$) indicated significant differences. When ANOVA showed significance, Tukey's Honestly Significant Difference (HSD) post hoc test was applied to identify which specific treatments differed from one another. In Tukey's test, treatments sharing the same letter (e.g., "a") are statistically equivalent, while different letters (e.g., "a", "b") indicate significant differences at $\alpha = 0.05$. The letter "a" represents the group with the highest mean values. Box and whisker plots (boxplots) were used to visualize the distribution of compressive strength data for each treatment. All statistical analyses were performed using RStudio version 4.3.1, with three replicates per treatment combination.

3. Results and discussions

3.1 Pozzolanic activity - compressive strength of mortar

Figure 8 shows the comparison of the treatments evaluated. The ANOVA (Analysis of Variance) test yielded a p-value less than 0.05 ($p < 0.05$) at all curing periods (7, 14, 21, and 28 days), indicating statistically significant differences among treatments. At 28 days of curing, Tukey's multiple comparison test was used to identify which specific treatments differed from one another. In his test, treatments sharing the same letter are statistically equivalent, whereas different letters indicate significant differences. The treatments with 5% RHA replacement at calcination temperatures of 700°C and 750°C both received the letter "a," indicating no significantly differ from each other and achieved the highest compressive strength values among all treatments, with averages of 29.73 MPa and 28.30 MPa, respectively.

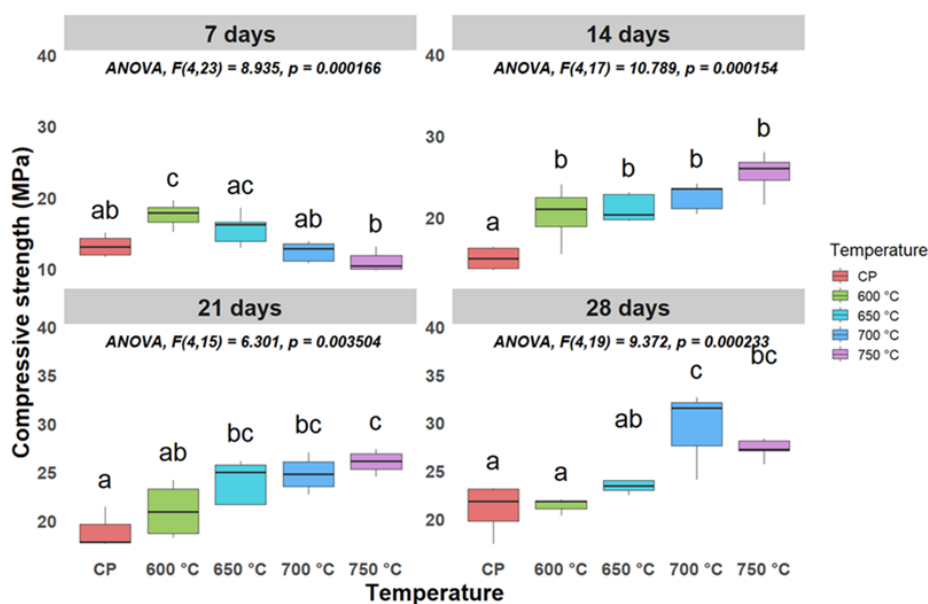


Figure 8 Compressive strength of mortar cubes replacing cement with 5% RHA of RHA calcined at 600, 650, 700, 750 °C, at 7, 14, 21 and 28 days of curing.

In Figure 9, in the comparison of the treatments under study, the ANOVA test presented a p-value of significance lower than 0.05 ($p < 0.05$), both in the evaluation at 7 days of curing ($p = 0.000202 < 0.05$) and 28 days of curing ($p = 0.000342 < 0.05$), on the other hand, a p-value of significance greater than 0.05 ($p > 0.05$), at 14 days of curing ($p = 0.0158 > 0.05$) and 21 days of curing ($p = 0.0775 > 0.05$), it is worth mentioning that at 28 days of curing, the post hoc tukey multiple comparisons post hoc test identified that the treatment of 10% ash percentage at 700 °C calcination temperature of the RHA, presented a significant difference with the other treatments, and it was also the treatment that allowed maximizing the compressive strength, registering an average compressive strength of 32.86 MPa.

In Figure 10, the ANOVA test, to compare the effects of the treatments under study, reached a p-value of significance greater than 0.05 ($p > 0.05$), at 7 days of curing ($p = 0.101 > 0.05$), as well as at 21 days of curing ($p = 0.0709 > 0.05$), while it presented a p-value of significance less than 0.05 ($p < 0.05$), as well as at 14 days of curing ($p = 2.96e-05 < 0.05$), as well as at 28 days of curing ($p = 0.000309 < 0.05$). In addition, the post hoc Tukey test identified at 28 days of curing the treatment of 15% percentage of ash to be replaced and 700°C calcination

temperature of the RHA, as the treatment that allowed maximizing the compressive strength variable and presenting a significant difference with the other treatments evaluated, presenting an average compressive strength of 31.54 MPa.

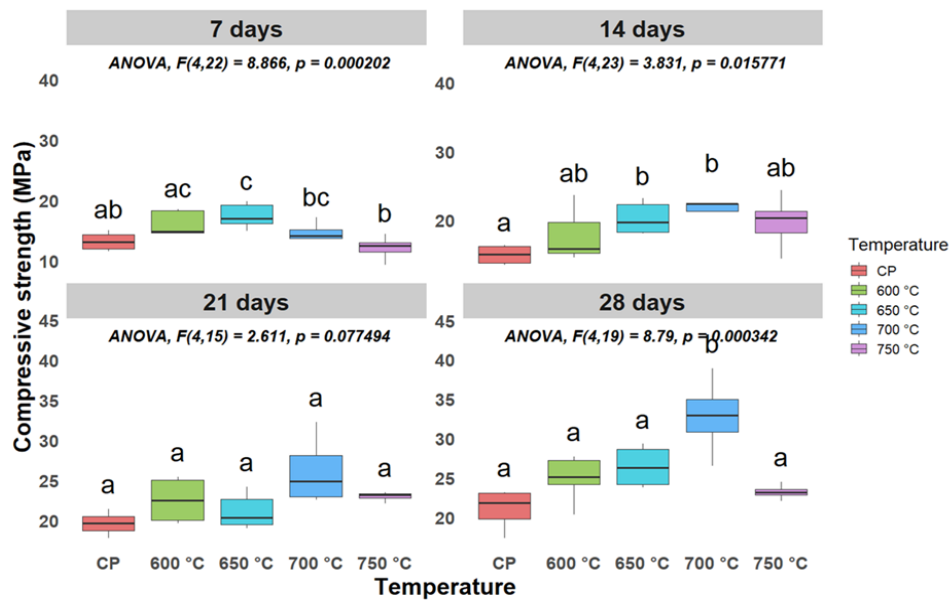


Figure 9 Compressive strength of mortar cubes replacing cement with 10% RHA of RHA calcined at 600, 650, 700, 750 °C, at 7, 14, 21 and 28 days of curing.

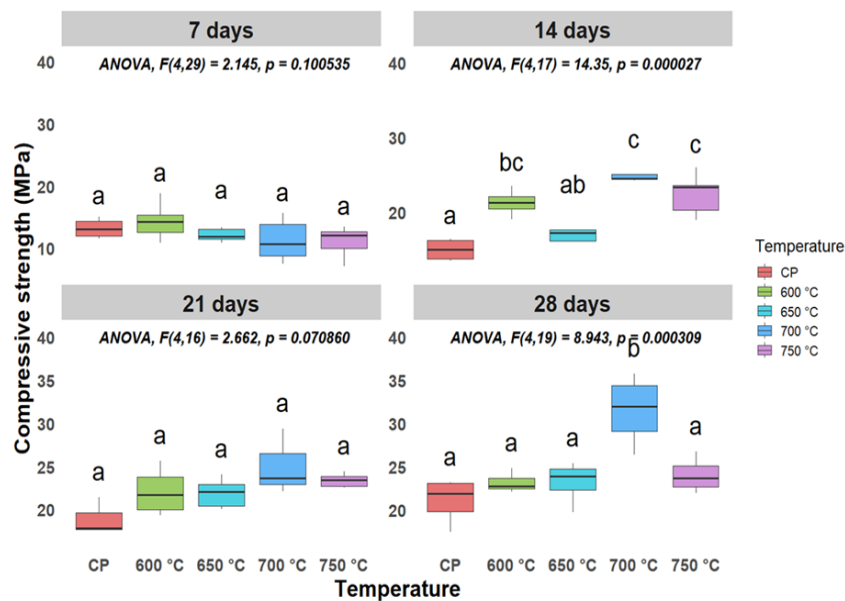


Figure 10 Compressive strength of mortar cubes replacing cement with 15% RHA of RHA calcined at 600, 650, 700, 750 °C, at 7, 14, 21 and 28 days of curing.

In Figure 11, shows that the p-value of significance of the ANOVA test registered a value greater than 0.05 ($p > 0.05$), both in the evaluation of the sympathy of the treatments at 21 days of curing ($p = 0.143 > 0.05$) and at 28 days of curing ($p = 0.563 > 0.05$), while a p-value of significance lower than 0.05, at 7 days of curing ($p = 9.29 \times 10^{-5} < 0.05$), as well as at 14 days of curing ($p = 0.00115 < 0.05$), i.e., where significant differences were found between the treatments. It is worth mentioning that in all the days of curing evaluated, it was the treatment with 20% percentage of ash to be replaced and 750 °C calcination temperature of the RHA, which presented the highest sample average compressive strength with values of 21.75 MPa at 7 days of curing, 22.45 MPa at 14 days of curing, 23.30 MPa at 21 days of curing and 24.41 MPa at 28 days of curing.

The results of Figure 12 show that the p-value of significance of the three-factor ANOVA analysis, associated with the interaction of the three factors under study (days of curing, temperature and percentage of ash to be replaced), presented a value less than 0.05, ($p = 1.10 \times 10^{-5} < 0.05$), i.e., there is a significant effect of the interaction of the three factors on the variable compressive strength, likewise, we can identify that the treatments with the highest sample average compressive strength were: 700 °C temperature with 10% ash percentage to replace 28 days of curing, in addition to 700 °C calcination temperature of RHA with 15% ash percentage to replace 28 days of curing and 700 °C calcination temperature of RHA with 5% ash percentage to replace 28 days of curing, whose average values recorded reached the values of 32.86, 31.54 and 29.73 MPa respectively.

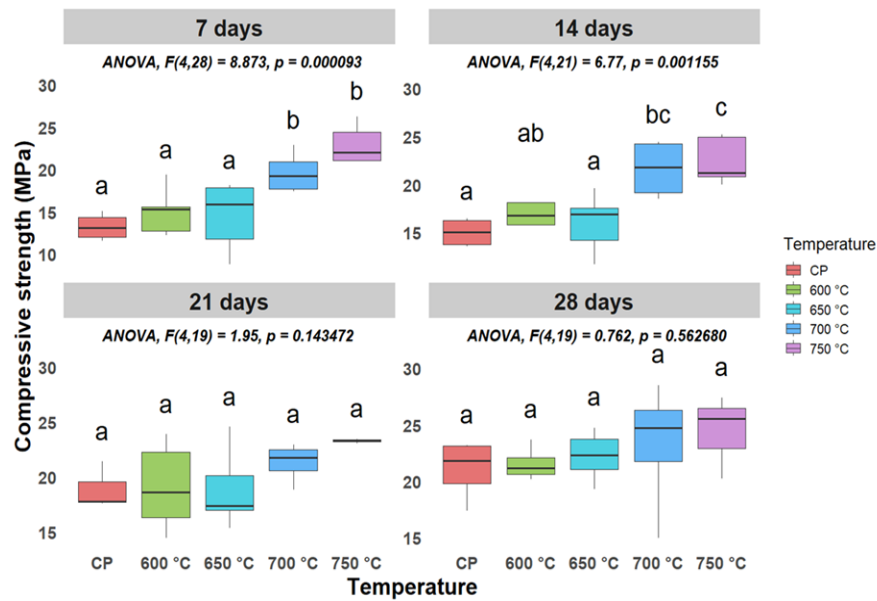


Figure 11 Compressive strength of mortar cubes replacing cement with 20% RHA of RHA calcined at 600, 650, 700, 750 °C, at 7, 14, 21 and 28 days of curing.

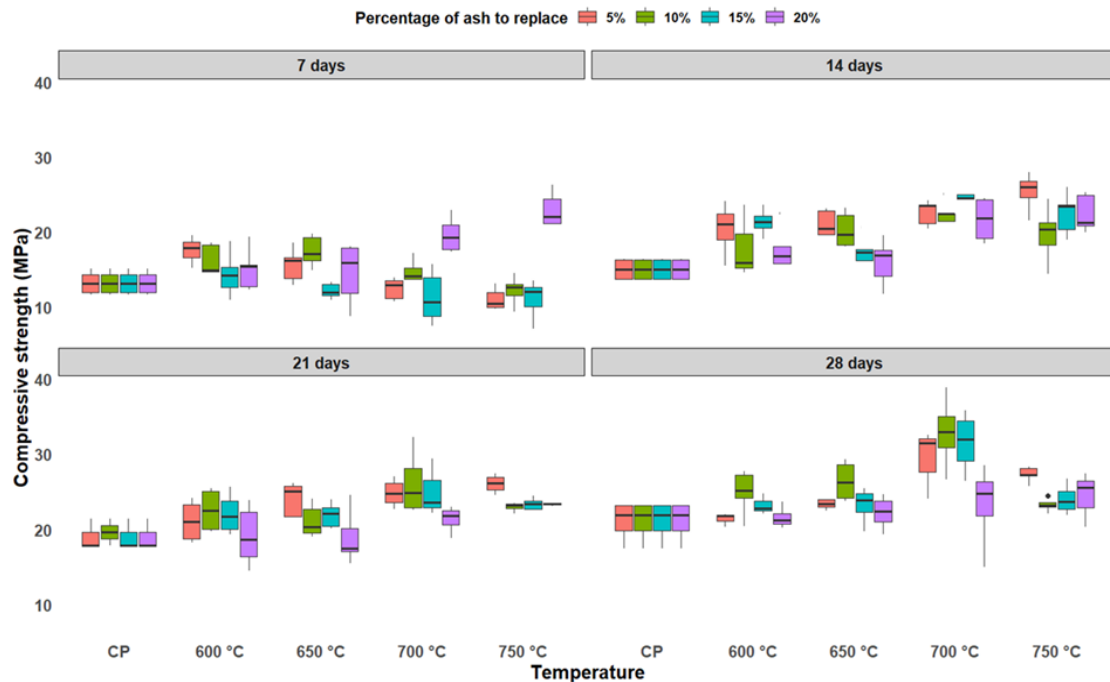


Figure 12 Compressive strength of mortar cubes replacing cement with 5, 10, 15 and 20% RHA calcined at 600, 650, 700, 750 °C, at 7, 14, 21 and 28 days of curing.

Increasing the compressive strength by 55.87% with respect to the standard concrete, coinciding with the studies conducted by Thiedeitz et al. [18], Muñoz et al. [19] and Wang et al. [20], showing that with 25%, 20% and 10% cement substitution by RHA calcined at 650 °C and 700 °C, the compressive strength increased by 14.29%, 23.80% and 15% with respect to the standard concrete, respectively. At lower calcination temperatures (600°C and 650°C), the ashes have a more crystalline structure, which limits their pozzolanic reactivity and generates greater porosity, resulting in lower compressive strength. At higher temperatures (700°C), amorphous silica predominates, favoring the formation of C-S-H gels that densify the structure and improve strength, which is reflected in higher compressive strength at 28 days. At 750°C, the recrystallization of silica and the formation of carbonates decrease reactivity and strength due to greater porosity and less C-S-H gel formation. Thus, the best mechanical performance is obtained with RHA calcined at 700°C, where the greater amount of amorphous silica favors reactivity and improves mortar strength.

3.2 X-Ray diffraction of mortar

Figure 13 represents the X-ray diffractogram of the standard concrete and Figure 14a, 14b, 14c and 14d present the X-ray diffractograms of the standard concrete with RHA at 600, 650, 700 and 750 °C, each diffractogram contains the identified crystalline phases indicated in the respective legends. It is observed that the crystalline phases of quartz, albite, microcline, muscovite, portlandite

and calcite are preserved in both the standard concrete and the RHA-added concrete. At temperatures of 600°C and 650°C, silica remains mostly crystalline, which limits its pozzolanic reactivity. At 700°C, an equilibrium is reached where the decrease in quartz crystallinity suggests the formation of a greater amount of amorphous silica, favoring pozzolanic activity. At 750°C, the recrystallization of silica and the appearance of more stable phases indicate a reduction in pozzolanic reactivity. Tables 4 to 8 summarize the concentrations of crystalline phases calculated using the Reference Intensity Ratio (RIR) method. These results agree with the studies of Muñoz Pérez et al. [19] and Wang et al. [20] who observed that replacing cement with RHA in the standard concrete by 20% and 10%, respectively, at calcination temperatures between 600 °C and 700 °C, resulted in an amorphous silica content of 71.50% and 89.16%, respectively.

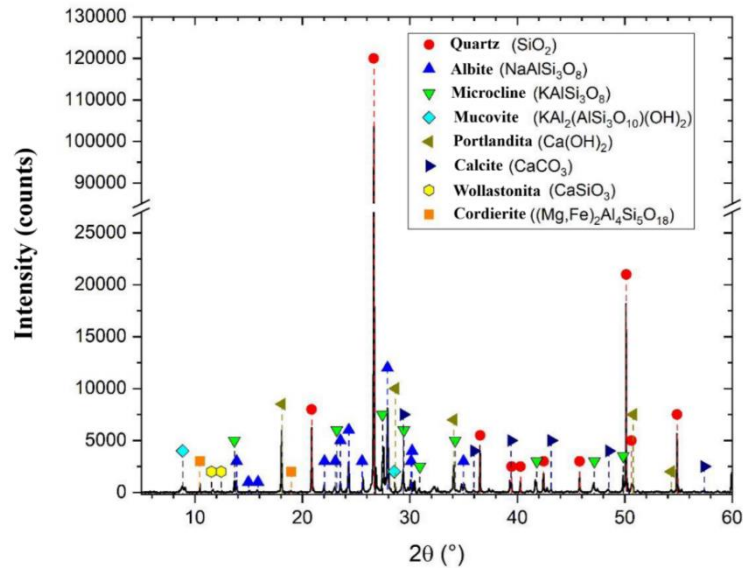


Figure 13 X-ray diffractogram of standard concrete

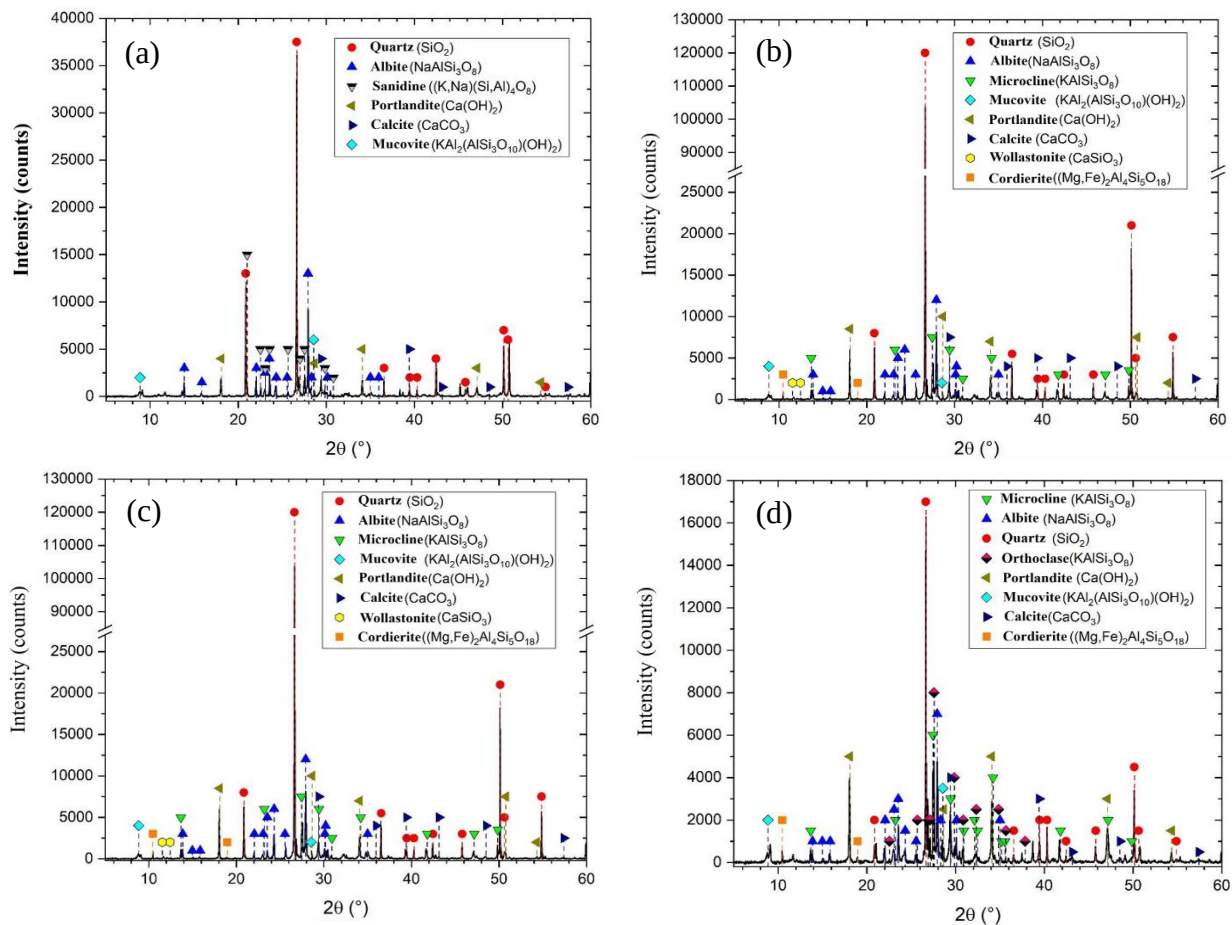


Figure 14 X-ray diffractogram of concrete with RHA additions: a) concrete with RHA addition at 600 °C, b) concrete with RHA addition at 650 °C, c) concrete with RHA addition at 700 °C, d) concrete with RHA addition at 750 °C.

Table 4 Phase concentrations calculated by means of the RIR method of standard concrete

Crystalline phase	Formula	According to database #	Concentration (wt%)
Quartz	SiO ₂	46-1045	52.5
Albita	NaAlSi ₃ O ₈	75-1142	14.2
Microcline	KAlSi ₃ O ₈	84-1455	7.7
Muscovite	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	04-0733	2.6
Portlandite	Ca(OH) ₂	42-0550	2.1
Calcite	CaCO ₃	05-0586	1.6
Wollastonite	CaSiO ₃	70-1869	1.5
Cordierite	(Mg, Fe) ₂ Al ₄ Si ₅ O ₁₈	77-0293	0.5
Amorphous	-		17.2

Table 5 Phase concentrations calculated by the RIR method of the standard concrete sample with ashes at 600 °C.

Crystalline phase	Formula	According to database #	Concentration (wt%)
Quartz	SiO ₂	46-1045	32.2
Albita	NaAlSi ₃ O ₈	75-1142	27.5
Sandina	(K, Na) (Si, Al) ₄ O ₈	87-0684	9.1
Portlandite	Ca(OH) ₂	42-0550	4.3
Calcite	CaCO ₃	05-0586	2.7
Muscovite	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	04-0733	2.5
Amorphous	-		21.7

Table 6 Phase concentrations calculated by the RIR method of the standard concrete sample with ashes at 650 °C.

Crystalline phase	Formula	According to database #	Concentration (wt%)
Microcline	KAlSi ₃ O ₈	84-1455	20.6
Quartz	SiO ₂	46-1045	19.6
Albita	NaAlSi ₃ O ₈	75-1142	17.8
Sandina	(K, Na)(Si, Al) ₄ O ₈	87-0684	12.8
Portlandite	Ca(OH) ₂	42-0550	4.7
Calcite	CaCO ₃	05-0586	3.1
Muscovite	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	04-0733	2.0
Amorphous	-		19.5

Table 7 Phase concentrations calculated by the RIR method of the standard concrete sample with ashes at 700 °C.

Crystalline phase	Formula	According to database #	Concentration (wt%)
Quartz	SiO ₂	46-1045	32.6
Sandina	(K, Na) (Si, Al) ₄ O ₈	87-0684	15.0
Microcline	KAlSi ₃ O ₈	84-1455	10.6
Albita	NaAlSi ₃ O ₈	75-1142	10.5
Muscovite	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	04-0733	3.6
Portlandite	Ca(OH) ₂	42-0550	2.6
Diopside	CaMgSi ₂ O ₆	86-0003	2.2
Calcite	CaCO ₃	05-0586	2.1
Amorphous	-		20.8

Table 8 Phase concentrations calculated by the RIR method of the standard concrete sample with ashes at 750 °C.

Crystalline phase	Formula	According to database #	Concentration (wt%)
Microcline	KAlSi ₃ O ₈	84-1455	17.2
Albita	NaAlSi ₃ O ₈	75-1142	15.2
Quartz	SiO ₂	46-1045	15.0
Orthoclase	KAlSi ₃ O ₈	75-1190	11.1
Portlandite	Ca(OH) ₂	42-0550	6.1
Muscovite	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	04-0733	4.9
Calcite	CaCO ₃	05-0586	3.6
Cordierite	(Mg, Fe) ₂ Al ₄ Si ₅ O ₁₈	77-0293	1.2
Amorphous	-		25.7

3.3 SEM and EDS scanning microscopy of mortar

Figures 15 to 19 present the micrographs of the samples, as well as the result of the chemical analysis of the samples. The elements found by EDS correspond mostly to the crystalline phases identified by XRD.

Figure 15 shows a dense and compact structure with little presence of porosities. no particles of irregular morphology or agglomerations are identified, Si and Ca predominate, which is characteristic of the traditional cementitious matrix, the presence of Al and Fe is detected, elements that may be associated with the phases of hydrated cement, the Si/Ca ratio is lower compared to the samples with ashes, indicating that portlandite is more present and less transformed into secondary cementitious compounds such as C-S-H.

Figure 16 shows the microstructure with irregular particles and dispersed porosities. Agglomerations of particles indicate low reactivity, some zones with unreacted particles, suggesting moderate pozzolanic reactivity, predominantly Si and Ca and contains high presence of Ca indicating coexistence of portlandite and carbonates. Crystalline silica still present. Figure 17 evidences lower porosity with respect to the 10% RHA sample at 600 °C, particles with more homogeneous morphology and better dispersion, presence of fibrous structures associated with C-S-H gel, increase in Si/Ca ratio, suggesting higher interaction with the cementitious matrix, lower free Ca indicates conversion of portlandite into secondary cementitious compounds.

Figure 18 shows a compact and homogeneous structure. Globular formations characteristic of the C-S-H gel, indicating high pozzolanic reactivity. Decrease in porosity improves ash integration, increase in Si and Al, with notable reduction of free Ca. High Si/Ca ratio, favoring conversion of portlandite into cementitious compounds. Possible formation of hydrated aluminosilicates, improving mechanical strength. Figure 19 shows signs of recrystallization, more defined particles, less cohesion with the cementitious matrix, stiffer structures, less C-S-H gel formation, higher porosity indicating less reactivity, decrease of reactive Si, suggesting loss of pozzolanic capacity. Lower Si/Ca ratio, reducing conversion of portlandite into C-S-H gels. Fe and K present, but without significant impact on reactivity.

SEM images and EDS analyses show how the addition of calcified RHA at different temperatures influences the microstructure and strength of concrete. In standard concrete, quartz (SiO_2), albite ($\text{NaAlSi}_3\text{O}_8$), and microcline (KAlSi_3O_8) peaks predominate, along with portlandite ($\text{Ca}(\text{OH})_2$) and calcite (CaCO_3). When RHA is incorporated, especially with 10% ash calcined at 700°C, there is an increase in the presence of amorphous silica and a decrease in quartz crystallinity, which improves pozzolanic reactivity and promotes the formation of C-S-H gels, improving concrete densification. With 20% RHA calcined at 750°C, the orthoclase and carbonate peaks intensify, indicating lower silica reactivity and decreased strength. EDS analyses confirm that samples with a higher percentage of RHA and calcined at 700°C have a higher concentration of silicon (Si) and calcium (Ca), indicating greater C-S-H gel formation and a denser structure. However, at higher temperatures (750°C), the recrystallization of silica reduces its pozzolanic capacity, which decreases the strength of the concrete. The use of RHA calcined between 600°C and 700°C optimizes pozzolanic reactivity and improves concrete strength, while temperatures above 750°C reduce its effectiveness due to silica recrystallization.

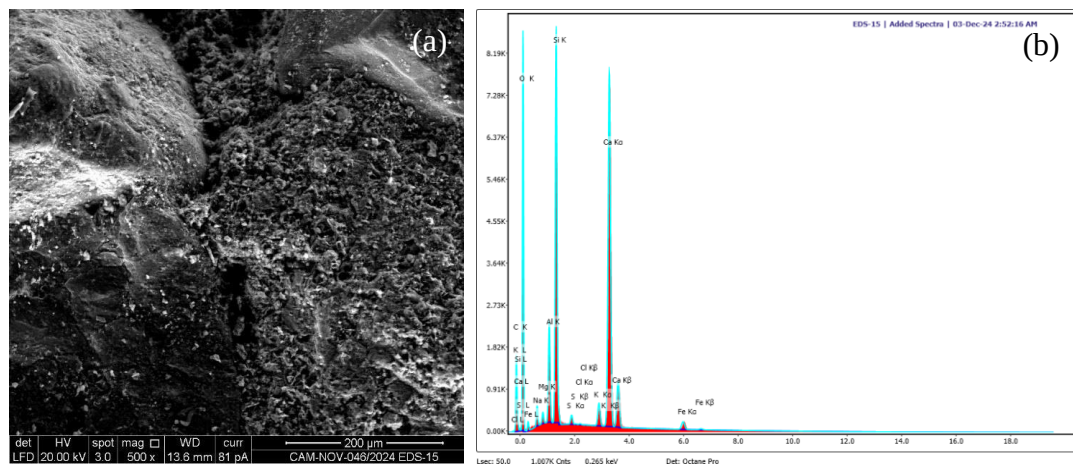


Figure 15 a) Micrograph of the standard sample at 500X magnification, b) EDS spectrum of the chemical composition of the standard sample.

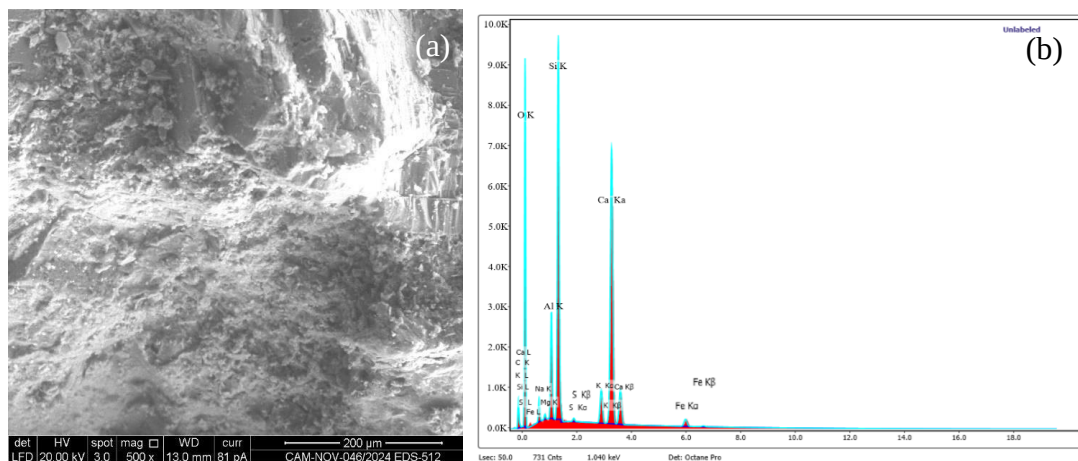


Figure 16 a) Micrograph of the sample with ashes at 600 °C at 500X magnification, b) EDS spectrum of the chemical composition of the sample with ashes at 600 °C.

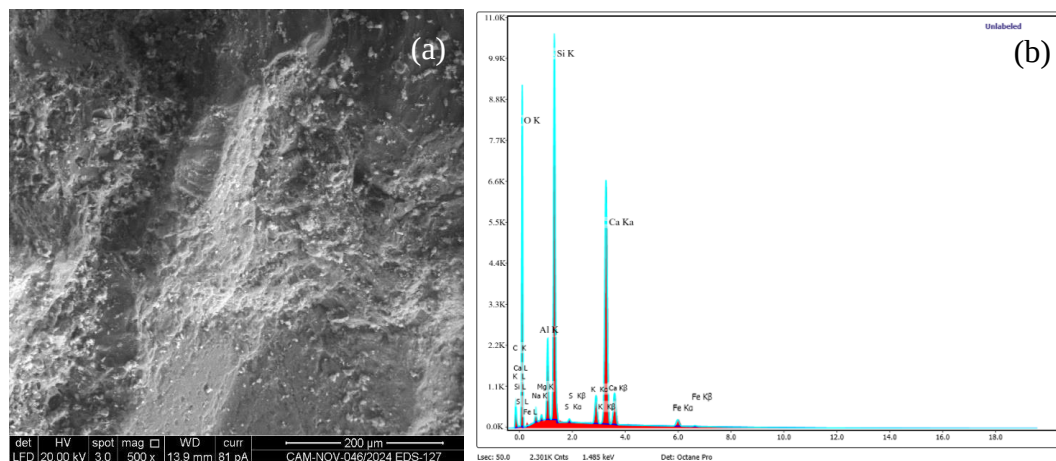


Figure 17 a) Micrograph of the sample with ashes at 650 °C at 500X magnification, b) EDS spectrum of the chemical composition of the sample with ashes at 650 °C

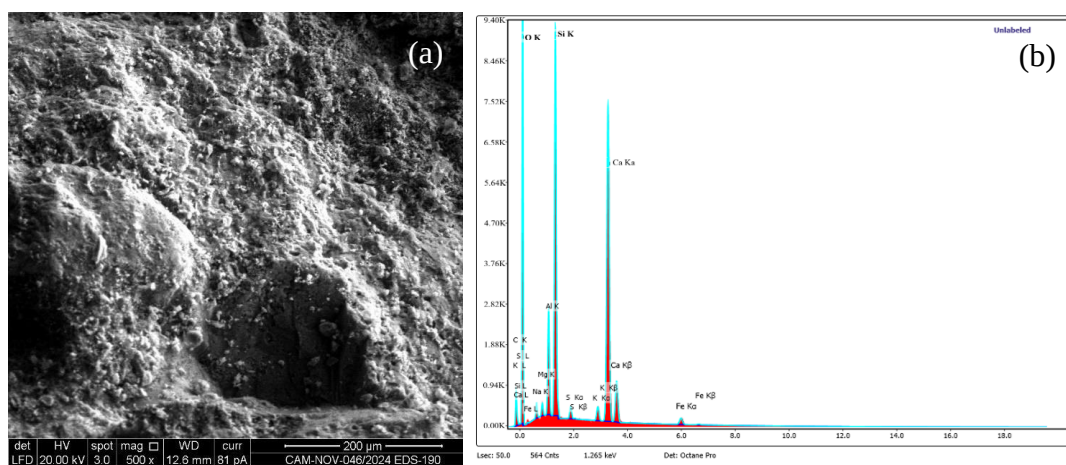


Figure 18 a) Micrograph of the sample with ashes at 700 °C at 500X magnification, b) EDS spectra of the chemical composition of the sample with ashes at 700 °C

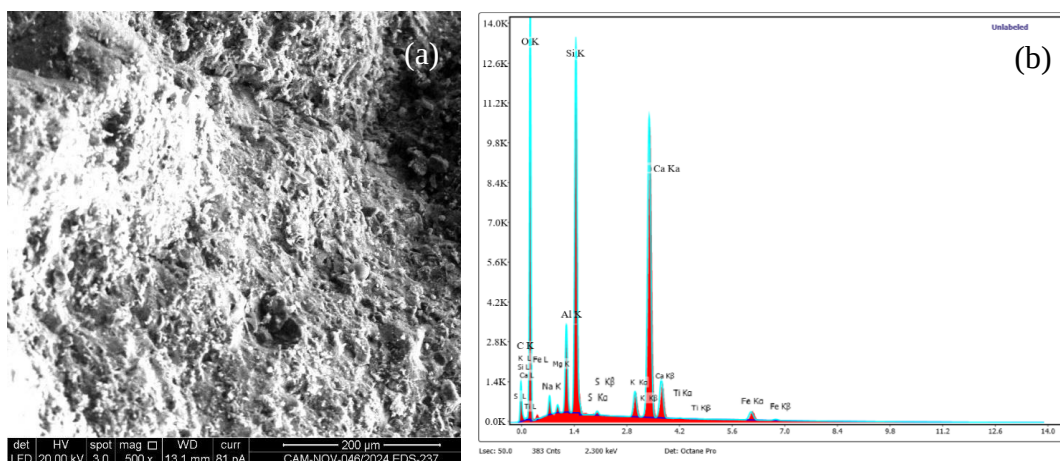


Figure 19 a) Micrograph of the sample with ashes at 750 °C at 500X magnification, b) EDS spectra of the chemical composition of the sample with ashes at 750 °C

3.4 Fourier Transform Infrared Spectroscopy (FTIR) of mortar

Figure 20 shows the presence of compounds containing hydroxyl (-OH), carbonyl (C=O), aliphatic C-H bonds and possibly aromatic structures. This FTIR spectrum confirms the presence of the main products of hydrated concrete such as hydrated calcium silicate (C-S-H), responsible for mechanical strength, calcium hydroxide ($\text{Ca}(\text{OH})_2$), calcium carbonate (CaCO_3) and adsorbed Water. Figure 21 shows the analyzed FTIR spectra corresponding to the concrete samples with different percentages of RHA calcined at specific temperatures. Differences in infrared absorption are observed, reflecting changes in the chemical composition and reactivity

of the materials. The spectrum corresponding to the sample with 10% RHA calcined at 600°C shows a prominent broad band in the $\sim 3400\text{ cm}^{-1}$ region, indicating a higher presence of -OH groups associated with adsorbed water. In addition, the Si-O-Si band at $\sim 1100\text{ cm}^{-1}$ is presented with lower intensity, suggesting a less reactive silica compared to other calcination temperatures.

As the calcination temperature increases to 650°C, a slight increase in the intensity of the Si-O-Si band is observed, indicating a higher amount of amorphous silica and thus a possible increase in pozzolanic activity. The band of the -OH groups is slightly reduced, suggesting a lower structural water retention compared to the sample calcined at 600°C.

When the temperature reaches 700°C, the band corresponding to -OH groups ($\sim 3400\text{ cm}^{-1}$) attenuates considerably, indicating a significant reduction of adsorbed water. In parallel, the Si-O-Si band ($\sim 1100\text{ cm}^{-1}$) intensifies, suggesting a higher development of amorphous silica, favoring its pozzolanic reactivity. This behavior suggests that this temperature is optimal to obtain a highly reactive material.

In contrast, the sample with 5% RHA calcined at 750°C shows an increase in the carbonate band ($\sim 1400\text{ cm}^{-1}$), suggesting an increased formation of CaCO_3 due to the reaction of Ca(OH)_2 with atmospheric CO_2 . In addition, the Si-O-Si band shows a slight reduction in its intensity, which may indicate a partial transition from amorphous silica to a more crystalline phase, thus reducing its pozzolanic capacity.

The FTIR spectra of the standard concrete and mixtures with calcified RHA at different temperatures show how the addition of RHA affects the reactivity and chemical composition of the concrete. The spectrum of the standard concrete shows typical characteristics, such as bands at 3400 cm^{-1} (O-H), 1100 cm^{-1} (Si-O-Si), and 1450 cm^{-1} (C=O), associated with hydrated compounds such as Ca(OH)_2 and C-S-H. In mixtures with 10% RHA, as the calcination temperature increases, an intensification of the Si-O-Si band is observed, indicating a greater presence of amorphous silica that favors pozzolanic reactivity. At 700°C, amorphous silica reaches its maximum, optimizing the reactivity of the material, while at 750°C, the formation of carbonates and the recrystallization of silica reduce pozzolanic effectiveness, decreasing the strength of the concrete. These results confirm that temperatures between 600°C and 700°C favor reactivity and strength, while temperatures above 750°C are less effective.

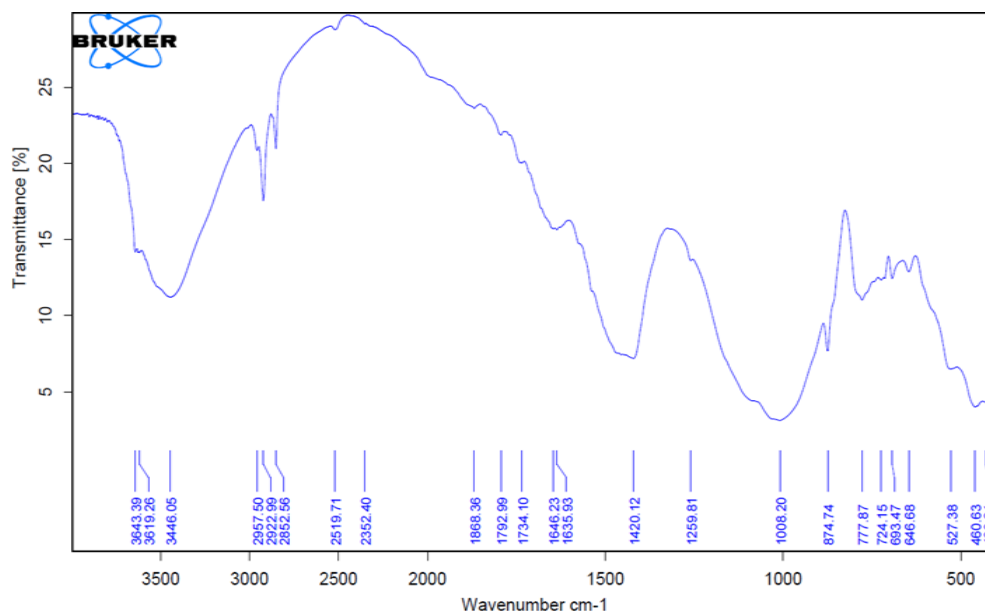


Figure 20 FTIR spectrum of the standard sample.

3.5 Thermogravimetric analysis (TGA) of mortar

Figure 22 shows the TGA of the standard concrete shows three main mass loss stages: (1) dehydration of adsorbed water and portlandite ($\sim 100^\circ\text{C}$ - 200°C , -4.8%), (2) decomposition of the C-S-H gel ($\sim 400^\circ\text{C}$ - 500°C , -2.3%), and (3) decomposition of calcium carbonate ($\sim 600^\circ\text{C}$ - 850°C , -3.5%), leaving a final residual mass of 89.4%.

Thermogravimetric derivative (TGD) analysis reveals peaks at 93.5°C , 141°C , 441°C and 719°C , associated respectively with free water removal, dehydration of secondary cementitious phases, portlandite decomposition and calcium carbonate degradation. On the other hand, differential scanning calorimetric analysis (DSC) identifies endothermic peaks at 97°C , 142°C , 443°C and 574°C , linked to water loss, dehydration of cementitious compounds, portlandite degradation and structural changes in the quartz present in the mixture. The values of the results are presented in Table 9.

Figure 23 evidences the thermogravimetric analysis (TG) shows the evolution of mass loss as a function of temperature, with three main stages: adsorbed water removal ($\sim 100^\circ\text{C}$ - 200°C), C-S-H gel decomposition ($\sim 400^\circ\text{C}$ - 500°C) and calcium carbonate degradation ($\sim 600^\circ\text{C}$ - 850°C), varying the residual mass according to the calcination temperature and the percentage of RHA. The thermogravimetric derivative (TGD) reflects mass loss rates, with key peaks in free water removal ($\sim 90^\circ\text{C}$ - 110°C), secondary phase dehydration ($\sim 140^\circ\text{C}$ - 160°C), portlandite decomposition ($\sim 440^\circ\text{C}$ - 460°C), and calcium carbonate degradation ($\sim 570^\circ\text{C}$ - 730°C). These peaks vary with the calcination temperature of the RHA, indicating differences in its reactivity and effect on cement hydration. Heat flow analysis (DSC) evidences relevant endothermic transitions, such as loss of adsorbed water ($\sim 100^\circ\text{C}$), dehydration of secondary phases ($\sim 140^\circ\text{C}$ - 150°C), decomposition of portlandite ($\sim 450^\circ\text{C}$) and structural change of quartz ($\sim 570^\circ\text{C}$ - 730°C). The intensity and displacement of these peaks reflect the impact of RHA on the thermal stability of the cementitious material.

Also, the maximum mass loss temperatures of water and organic materials 79 , 103 and 454°C (DTG, green curve) and the decomposition of inorganic materials in the presence of oxygen at 713°C of the standard concrete with 10% RHA calcined at 600°C are identified, the values of the results are presented in Table 10. Table 11 shows the maximum mass loss temperatures of water and

organic materials 71, 101 and 446°C (DTG, green curve) and the decomposition of inorganic materials in the presence of oxygen at 695°C of the standard concrete containing 10% RHA calcined at 650°C. Table 12 shows the values of the maximum mass loss temperatures of water and organic materials 71, 104 and 450°C (DTG, green curve) and the decomposition of inorganic materials in the presence of oxygen at 726°C of the standard concrete with 10% RHA calcined at 700°C. Table 13 shows the values of the maximum mass loss temperatures of water and organic materials 78, 105 and 451°C (DTG, green curve) and the decomposition of inorganic materials in the presence of oxygen at 722°C of the standard concrete with 5% RHA calcined at 750 °C.

TGA reveals different stages of mass loss, highlighting the decomposition of adsorbed water, cementitious compounds, and carbonates, depending on the calcination temperature of the RHA. The results indicate that the higher amorphous silica content in RHA calcined at 700 °C favors the formation of C-S-H gels, improving matrix densification and reducing porosity, which increases mortar strength. However, at higher temperatures (750 °C), silica recrystallization and carbonate formation decrease pozzolanic reactivity, reducing the effectiveness of RHA and the strength of concrete.

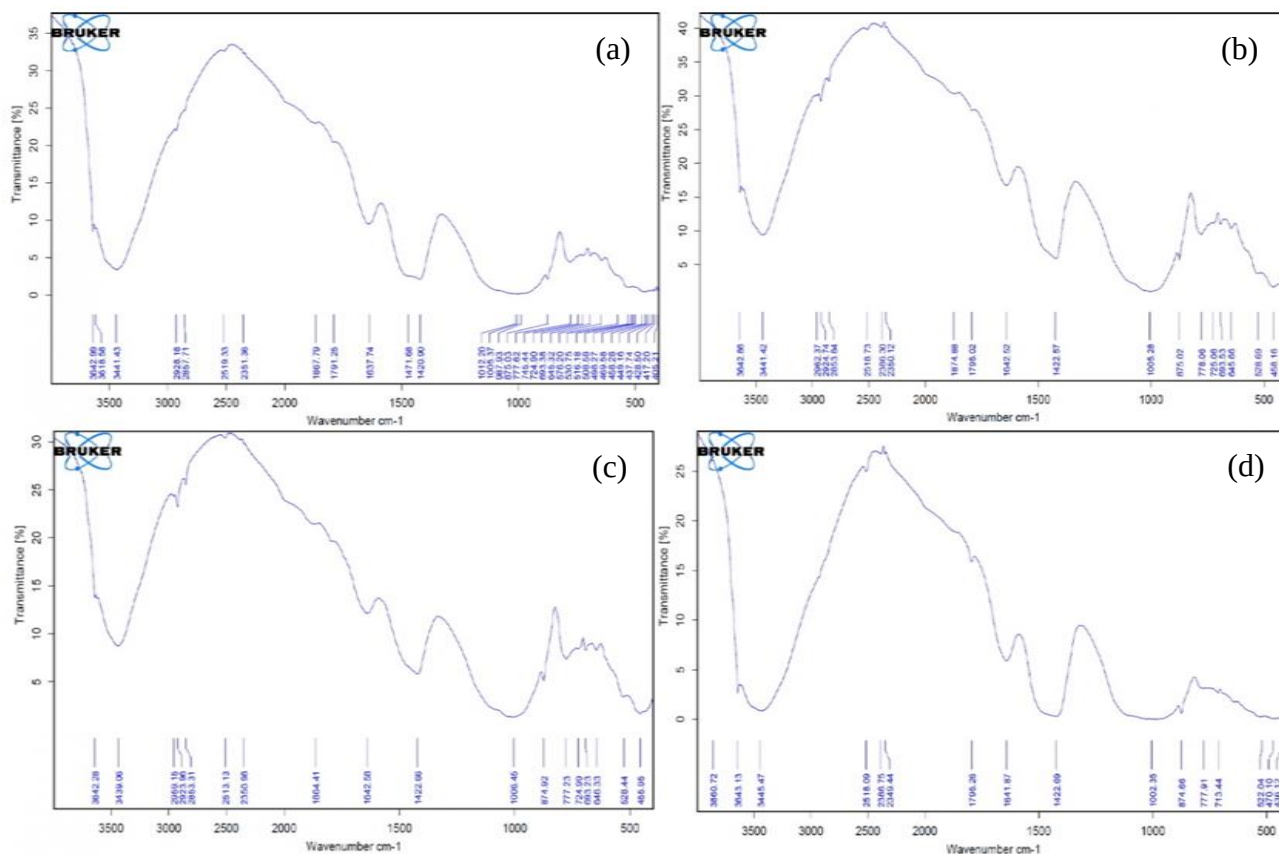


Figure 21 FTIR spectra a) standard concrete with 10% RHA calcined at 600 °C, b) standard concrete with 10% RHA calcined at 650 °C, c) standard concrete with 10% rice husk ash calcined at 700 °C, d) standard concrete with 5% RHA calcined at 750 °C.

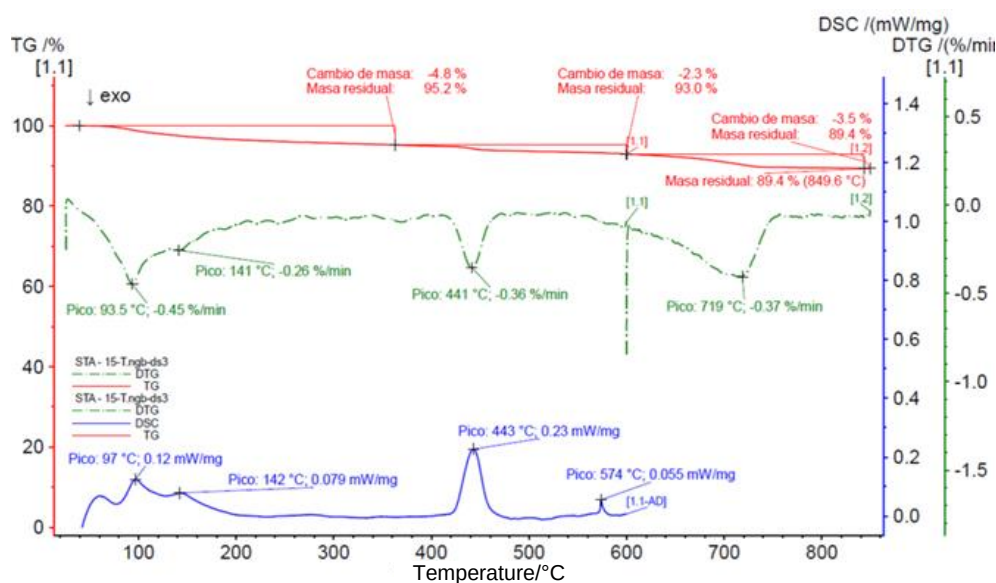
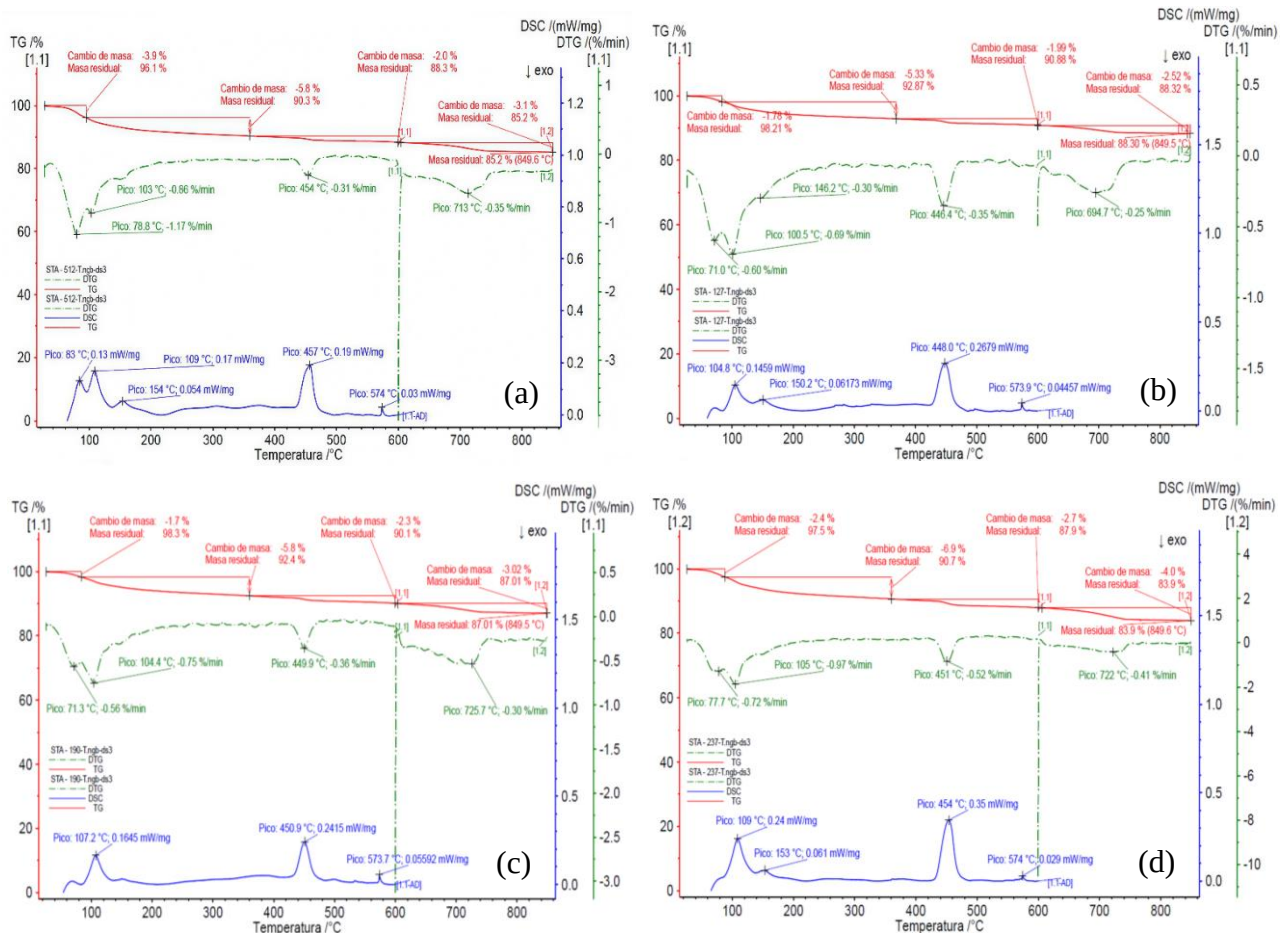


Figure 22 TGA analysis of the standard sample.

Table 9 Results of TGA analysis of the standard sample

Maximum degradation temperatures (*) (°C)	Loss of mass (%)			Residual mass at 850°C (%)
	30 – 360 °C	360 – 600 °C	600 – 850 °C (**)	
79				
103	4.8	2.3	3.5	89.4
454				
713				

(*) From the curve DTG / (**) In oxygen environment.

**Figure 23** TGA analysis a) standard concrete with 10% RHA calcined at 600 °C, b) standard concrete with 10% RHA calcined at 650 °C, c) standard concrete with 10% RHA calcined at 700 °C, d) standard concrete with 5% RHA calcined at 750 °C, e) standard concrete with 10% RHA calcined at 700 °C, f) standard concrete with 10% RHA calcined at 700 °C.**Table 10** Results of TGA analysis of standard concrete with 10% RHA calcined at 600 °C.

Maximum degradation temperatures (*) (°C)	Loss of mass (%)			Residual mass at 850°C (%)
	30 – 360 °C	360 – 600 °C	600 – 850 °C (**)	
79				
103	9.7	2.0	3.1	85.2
454				
713				

(*) From the curve DTG / (**) In oxygen environment.

Table 11 Results of TGA analysis of standard concrete with 10% RHA calcined at 650 °C.

Maximum degradation temperatures (*) (°C)	Loss of mass (%)			Residual mass at 850°C (%)
	30 – 360 °C	360 – 600 °C	600 – 850 °C (**)	
71				
101	7.1	2.0	2.5	88.3
446				
695				

(*) From the curve DTG / (**) In oxygen environment.

Table 12 Results of the TGA analysis of the standard concrete with 5% calcined RHA at 700 °C

Maximum degradation temperatures (*) (°C)	Loss of mass (%)			Residual mass at 850°C (%)
	30 – 360 °C	360 – 600 °C	600 – 850 °C (**)	
71				
104	7.5	2.3	3.0	87.0
450				
726				

(*) From the curve DTG / (**) In oxygen environment.

Table 13 Results of TGA analysis of standard concrete with 5% RHA calcined at 700 °C.

Maximum degradation temperatures (*) (°C)	Loss of mass (%)			Residual mass at 850°C (%)
	30 – 360 °C	360 – 600 °C	600 – 850 °C (**)	
78				
105	9.3	2.7	4.0	83.9
451				
722				

(*) From the curve DTG / (**) In oxygen environment.

4. Limitations

Despite the advances made in this study on the use of rice husk ash (RHA) in mortars, there are important limitations to consider. Variability in the quality of available RHA may influence the results, as pozzolanic behavior depends on the calcination temperature and the material's chemical composition. In addition, the long-term durability of mortar with RHA, especially under actual exposure conditions, was not evaluated; therefore, further studies addressing resistance to freeze-thaw cycles, sulfate attack, and abrasion are needed. This study was limited to four calcination temperatures (600 °C, 650 °C, 700 °C, and 750 °C), so it is essential to extend the research to a wider range of temperatures, including values above 750 °C, to gain a more complete understanding of the impact of calcination on the reactivity and durability of RHA. Finally, although the results are promising, it is crucial to continue discussing and compare these findings with previous studies to confirm their consistency and advance the development of practical recommendations for the construction industry.

5. Conclusions

This study has demonstrated that the calcination temperature of rice husk ash (RHA) significantly influences the compressive strength and microstructural characteristics of mortars. The best performance was achieved at a calcination temperature of 700°C, where the mortar with 15% RHA showed a 55.87% improvement in compressive strength compared with the standard mortar, with an average value of 31.54 MPa at 28 days of curing. At this temperature, the amorphous silica content reached 69.40%, which favored pozzolanic reactivity and enhanced the formation of C-S-H and C-A-S-H gels, resulting in a denser matrix with lower porosity. These results indicate that the use of RHA calcined at 700°C could be a viable alternative to improve mortar strength, contributing to sustainability in construction by reducing cement use and CO₂ emissions.

The practical implications of these findings are clear: the use of RHA calcined at temperatures between 600°C and 700°C can be implemented in the construction industry as a partial substitute for cement without compromising mortar strength, especially in applications that do not require extremely high strength. This approach has the potential not only to reduce costs but also to promote more sustainable practices by reusing agricultural by-products and lowering the carbon footprint of construction. However, further studies are recommended to evaluate the long-term durability of mortars with RHA and to explore calcination temperatures above 750°C to gain a better understanding of their impact on material strength and durability.

6. Recommendations

It is recommended to conduct further research on the impact of calcination temperatures above 750°C on the reactivity and durability of RHA, and to explore the relationship between RHA particle size and amorphous silica content to optimize its properties in mortars. Additionally, it is essential to evaluate the long-term durability of mortars with RHA, considering their behavior under freeze-thaw cycles, sulfate attack, and abrasion. It is also suggested to investigate the interaction of RHA with other alternative materials, such as fly ash or oyster shell powder, to enhance both the strength and durability of the mortar. Lastly, further studies on the microstructure of mortars with RHA at different curing ages are recommended to understand how it evolves over time and improve performance in terms of strength and durability.

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