REVISTA CIENTÍFICA - RECIMA21 ISSN 2675-6218

# OPTIMIZING CALCINATION CONDITIONS OF CORN COB ASH FOR PARTIAL REPLACEMENT OF PORTLAND CEMENT IN CONCRETES

# OTIMIZAÇÃO DAS CONDIÇÕES DE CALCINAÇÃO DAS CINZAS DE SABUGO DE MILHO PARA SUBSTITUIÇÃO PARCIAL DO CIMENTO PORTLAND EM CONCRETOS

# OPTIMIZACIÓN DE LAS CONDICIONES DE CALCINACIÓN DE CENIZAS DE MAZORCA DE MAÍZ PARA LA SUSTITUCIÓN PARCIAL DEL CEMENTO PORTLAND EN HORMIGONES

Ayra Mariah Campos[1], Arthur Aviz Palma e Silva[2], Kárita Christina Soares Kanaiama Alves[3], Suylan Matias da Cruz[4], Julio Roberto Uszacki Junior[4]

e717140
https://doi.org/10.47820/recima21.v7i1.7140

PUBLISHED: 01/2026

## ABSTRACT
The use of agro-industrial residues as supplementary cementitious materials contributes to environmental sustainability and resource efficiency in concrete production. This study investigates the pozzolanic potential of corn cob ash (CCA) produced under different thermal conditions. Corn cobs were calcinated at 650°C, 750°C, and 850°C for durations of 2 and 4 hours. The resulting ashes were evaluated through physical characterization (specific gravity, fineness, particle size distribution, and loss on ignition), as well as chemical and mineralogical analyses (XRF and XRD). The ashes calcinated at 750°C for 2 hours exhibited the most favorable properties, including the highest specific gravity (2.87 g/cm³) and an improved particle size distribution. This condition also resulted in a higher amorphous content, identified by broader halos in the XRD patterns, which is a desirable characteristic for pozzolanic reactivity. Conversely, samples calcinated at 850°C showed increased crystallinity and lower fineness, indicating a reduction in pozzolanic potential. XRF analysis revealed a high $SiO_2$ content in all ashes, particularly in those produced at 750°C, confirming compliance with pozzolanic standards ($SiO_2$ + $Al_2O_3$ + $Fe_2O_3$ ≥ 73%). Loss on ignition (LOI) values reached up to 78% at 850°C, reflecting the presence of volatile organic matter, while the lowest LOI values were observed in samples treated at 650°C. Although the ash produced at 750°C for 2 hours did not meet the ideal fineness threshold (<15% retained on the 75 µm sieve), it presented a fineness index close to acceptable limits (19.9%). Overall, the results demonstrate that CCA produced at 750°C for 2 hours provides the best balance of physical and chemical characteristics for use as a pozzolanic material in cement-based composites, whereas calcination at 850°C may impair performance due to increased crystallinity.

**KEYWORDS:** Corn cob ash. Pozzolanic activity. Thermal treatment.

## RESUMO
O uso de resíduos agroindustriais como materiais cimentícios suplementares contribui para a sustentabilidade ambiental e para a eficiência no uso de recursos na produção de concretos. Este estudo investiga o potencial pozolânico da cinza de sabugo de milho (CCA) produzida sob diferentes condições térmicas. Os sabugos de milho foram calcinados a 650°C, 750°C e 850°C por períodos de 2 e 4 horas. As cinzas obtidas foram avaliadas por meio de caracterização física (massa específica, finura, distribuição granulométrica e perda ao fogo), além de análises químicas e mineralógicas (FRX e DRX). As cinzas calcinadas a 750°C por 2 horas apresentaram as

[1] Graduanda em Engenharia Civil, Instituto Federal do Tocantins (IFTO), Gurupi-TO, Brasil.
[2] Professor do curso de Engenharia Civil, Estácio Brasília, Brasília-DF, Brasil.
[3] Professora do curso de Engenharia Civil, Instituto Federal do Tocantins (IFTO), Gurupi-TO, Brasil.
[4] Universidade de Brasília (UnB), Programa de Pós-Graduação em Estruturas e Construção Civil (PECC), Brasília-DF, Brasil.

REVISTA CIENTÍFICA - RECIMA21 ISSN 2675-6218



propriedades mais favoráveis, incluindo a maior massa específica (2,87 g/cm³) e uma distribuição granulométrica mais adequada. Essa condição também resultou em maior teor de fase amorfa, identificado pela presença de halos mais largos nos difratogramas de DRX, característica desejável para a reatividade pozolânica. Em contrapartida, as amostras calcinadas a 850°C apresentaram aumento da cristalinidade e menor finura, indicando redução do potencial pozolânico. A análise por FRX revelou elevado teor de $SiO_2$ em todas as cinzas, especialmente naquelas produzidas a 750°C, confirmando o atendimento aos requisitos para materiais pozolânicos ($SiO_2 + Al_2O_3 + Fe_2O_3 \geq 73\%$). Os valores de perda ao fogo (PAF) atingiram até 78% a 850°C, refletindo a presença de matéria orgânica volátil, enquanto os menores valores de PAF foram observados nas amostras tratadas a 650°C. Embora a cinza produzida a 750°C por 2 horas não tenha atingido o limite ideal de finura (<15% retido na peneira de 75 μm), apresentou um índice de finura próximo aos valores aceitáveis (19,9%). De forma geral, os resultados demonstram que a CCA produzida a 750°C por 2 horas apresenta o melhor equilíbrio entre características físicas e químicas para uso como material pozolânico em compósitos cimentícios, enquanto a calcinação a 850°C pode comprometer o desempenho devido ao aumento da cristalinidade.

**PALAVRAS-CHAVE**: Cinza de sabugo de milho. Atividade pozolânica. Tratamento térmico.

**RESUMEN**
El uso de residuos agroindustriales como materiales cementantes suplementarios contribuye a la sostenibilidad ambiental y a la eficiencia en el uso de recursos en la producción de concreto. Este estudio investiga el potencial puzolánico de la ceniza de mazorca de maíz (CCA) producida bajo diferentes condiciones térmicas. Las mazorcas de maíz fueron calcinadas a 650°C, 750°C y 850°C durante períodos de 2 y 4 horas. Las cenizas obtenidas fueron evaluadas mediante caracterización física (densidad aparente, finura, distribución granulométrica y pérdida por ignición), así como mediante análisis químicos y mineralógicos (XRF y XRD). Las cenizas calcinadas a 750°C durante 2 horas presentaron las propiedades más favorables, incluyendo la mayor densidad aparente (2,87 g/cm³) y una mejor distribución del tamaño de partículas. Esta condición también condujo a un mayor contenido amorfo, identificado por la presencia de halos más anchos en los difractogramas de XRD, una característica deseable para la reactividad puzolánica. En contraste, las muestras calcinadas a 850°C mostraron un aumento de la cristalinidad y una menor finura, lo que sugiere una reducción del potencial puzolánico. El análisis por XRF reveló un alto contenido de $SiO_2$ en todas las cenizas, especialmente en aquellas producidas a 750°C, confirmando el cumplimiento de los requisitos para materiales puzolánicos ($SiO_2 + Al_2O_3 + Fe_2O_3 \geq 73\%$). Los valores de pérdida por ignición (LOI) alcanzaron hasta un 78% a 850°C, lo que refleja la presencia de materia orgánica volátil, mientras que los valores más bajos de LOI se observaron en las muestras tratadas a 650°C. Aunque la ceniza producida a 750°C durante 2 horas no alcanzó el umbral ideal de finura (<15% retenido en el tamiz de 75 μm), presentó un índice de finura cercano a los límites aceptables (19,9%). En conjunto, los resultados demuestran que la CCA producida a 750°C durante 2 horas ofrece el mejor equilibrio entre características físicas y químicas para su uso como material puzolánico en compuestos cementicios, mientras que la calcinación a 850°C puede perjudicar el desempeño debido al aumento de la cristalinidad.

**PALABRAS CLAVE**: Ceniza de mazorca de maíz. Actividad puzolánica. Tratamiento térmico.

## 1. INTRODUCTION

Brazil is one of the world’s largest corn producers, harvesting 55.37 million tons over 13.93 million hectares (CONAB, 2022), which generates significant agro-industrial waste, particularly corn husks and cobs—often discarded despite their high reuse potential. Corn cobs are lignocellulosic materials composed of cellulose, hemicellulose, and lignin, which represent approximately 90% of the carbon biomass in the biosphere, according to Jeffries (1990). Reusing these residues in construction not only supports local economies but also aligns with environmental goals through circular economy strategies (Vaz Junior, 2020). When subjected to combustion, corn cobs yield corn cob ash (CCA), which has been widely studied for its potential as a supplementary cementitious material due to its favorable chemical composition. According to Adesanya and Raheem (2008), the pozzolanic activity of CCA is influenced by its content of $SiO_2$, $Al_2O_3$, and $Fe_2O_3$—oxides that, per ABNT NBR 12653 (2014), must total at least 70–73% for pozzolanic classification. Various studies confirm this potential: Udoeyo and Abubakar (2005) found 79.29% $SiO_2$ and a total oxide content of 80.91%, while Komalpreet Singh (2017) reported totals of 78.51% and 79.10%, respectively. Oluborode and Olofintuyi (2011) observed the highest oxide sum (83.03%) despite lower $SiO_2$ (56.39%), offset by elevated $Al_2O_3$ (17.57%) and $Fe_2O_3$ (9.07%). Similarly, Nnochiri et al. (2018) reported 64.90% $SiO_2$, 10.79% $Al_2O_3$, and 4.75% $Fe_2O_3$ (totaling 80.44%).

## 2. BIBLIOGRAPHY

Studies by Adesanya and Raheem (2008) and Desai (2018) showed totals of 78.30%, and 72.95%, respectively. Although Desai (2018) and Suwanmaneechaot, Nochaiya and Julphunthong (2015) reported values slightly below the 73% threshold (72.95% and 70.87%), they still indicated pozzolanic viability, especially when favorable physical properties are present. Overall, the literature highlights variability in CCA composition and calcination conditions, ranging from 600 °C to uncontrolled open-air burning (see Table 1).

**Table 1.** Reviewed research data for CCA

| Study (Author, Year) | Temperature (°C) | Duration | Observed Effectiveness |
|---|---|---|---|
| **Adesanya & Raheem [12]** | 650 °C (reached gradually in a charcoal-fired furnace) | 8 h | Considered suitable as a pozzolanic material. |
| **Udoeyo & Abubakar [13]** | Open-air burning | — | Showed good pozzolanic activity despite uncontrolled conditions. |
| **Suwanmaneechot et al. [11]** | 600 °C | 4 h | High reactivity. |

The literature consistently highlights the potential of corn cob ash (CCA) as a sustainable supplementary cementitious material, with most studies indicating 600–700 °C as the optimal calcination range to balance the decomposition of organic matter and the preservation of amorphous silica, thus maximizing pozzolanic reactivity (Adesanya; Raheem, 2008; 2009). Alternatively, Udoeyo and Abubakar (2005) demonstrated that higher temperatures and extended durations can also yield reactive ashes, suggesting an inverse relationship between time and temperature, where greater thermal energy may compensate for shorter exposure periods. However, such approaches raise concerns related to energy efficiency and the risk of generating crystalline phases that could impair pozzolanic behavior.

From an environmental standpoint, incorporating CCA into cementitious matrices offers a promising path to reduce the carbon footprint of construction materials. Multiple studies confirm that when properly processed, CCA can meet or exceed the chemical and mechanical benchmarks for pozzolanic materials, including strength activity indices. Yet, despite encouraging laboratory outcomes, further research is needed to assess the scalability of calcination and processing methods, as well as the long-term performance of CCA in diverse concrete formulations. In this context, the present study investigated the influence of different calcination temperatures (650 °C, 750 °C, and 850 °C) and durations (2 and 4 hours) on the physical and chemical properties of corn cob ash to evaluate its viability as a pozzolanic additive in concrete.

## 3. METHODOLOGY

The experimental procedure for producing and characterizing corn cob ash (CCA) involved several sequential steps, as shown in Tab. 2.

**Table 2**. Experimental program steps

| Step | Description |
|---|---|
| 1. Material Collection | Collection of corn cob waste |
| 2. Drying | Oven drying to remove moisture |
| 3. Calcination | 650 °C, 750 °C, 850 °C (2 and 4 hours) |
| 4. Physical Characterization (only for 4h calcination) | Fineness by 75 µm sieve, *Specific gravity, Particle size distribution, Loss on ignition (LOI)* |
| 5. Chemical Characterization | XRD (X-ray Diffraction), |

Corn cob samples were obtained from local producers in Gurupi, Tocantins, and initially stored at approximately 4 °C to control moisture levels. The material was then oven-dried at 105 °C for 24 hours at the Civil Engineering and Building Laboratory of the Federal Institute of Tocantins – Gurupi Campus. Subsequently, the dried cobs were subjected to controlled combustion in a muffle furnace at three temperatures: 650 °C, 750 °C, and 850 °C, each for 2 or 4 hours (Figure 1).

These findings demonstrate that higher calcination temperatures not only improve particle size refinement but also enhance the visual and potentially chemical quality of the ash—important factors for its use as a supplementary cementitious material.

ISSN: 2675-6218 - RECIMA21

**Fig. 1.** Corn cobs inside Muffle furnace. After combustion, the ashes produced were subjected to both physical and chemical characterization in order to evaluate their suitability as a supplementary cementitious material

Fineness was assessed using the 75 µm sieve, in accordance with NBR 11579 (1991). Loss on ignition (LOI), related to the decomposition of organic matter into mineral-rich ash, was determined using a method adapted from NBR NM 18 (2012). Specific gravity was measured via the pycnometer method (Figure 2), using a 10 mL vessel and ethanol as the displacement fluid. Particle size distribution was determined following ABNT NBR 7181 (2018), using sieves ranging from 2.0 mm to 0.075 mm. XRD analysis was conducted on ashes calcined at 650 °C, 750 °C, and 850 °C for 2 and 4 hours to identify crystalline phases and mineralogical composition. A RIGAKU diffractometer with CuKα radiation (λ = 1.54056 Å) operated at 35 kV and 15 mA, with a 2θ range of 2°–60°, a 0.05° step size, and 5°/min scan rate. Phase identification was performed using JADE 3.0 software and the ICDD database.

To estimate the degree of crystallinity of silica ($SiO_2$) in corn cob ash (CCA), an adaptation of the method originally proposed by Segal et al. (1959) was employed. This method, widely used to assess the crystallinity of cellulose via X-ray diffraction (XRD), calculates the crystallinity index based on the ratio between the maximum intensity of a crystalline peak and the minimum intensity in the amorphous region. In this study, the formula was adapted by replacing the characteristic peaks of cellulose with those of crystalline quartz, the predominant mineralogical phase in CCA, as shown in Eq. 1.

$$CrI_{sio2} = \frac{I26.6\circ - Iam}{I26.6\circ} x\ 100 \qquad (1)$$

where $I26.6\circ$ is the intensity of the main quartz peak at approximately 26.6° 2θ—corresponding to the (101) plane. The selection of this peak is supported by crystallographic data from the RRUFF database RRUFF ID: R100134 [23] and is consistent with the structural descriptions of quartz presented by Götze *et al.,* (2021) and Scrivener *et al.,* (2016). This adapted approach enables a semi-quantitative comparison of amorphous content across different calcination conditions without requiring Rietveld refinement or internal standards. and $I$am is the minimum intensity in the amorphous halo region, typically between 18° and 22° 2θ, following the approach described by Madsen *et al.,* (2011). This semi-quantitative adaptation allows for relative comparisons of the crystallinity of $SiO_2$ between samples subjected to different calcination conditions, without requiring full Rietveld refinement or internal standard methods.

## 4. RESULTS

Fineness was assessed according to NBR 11579 (1991), using 50 g samples and a 75 µm sieve. The ash calcinated at 850 °C had the lowest fineness, with only 15.8% passing (42.10 g retained), followed by 750 °C with 19.9% passing (40.05 g), and 650 °C with 20.46% (39.77 g). As per Costa *et al.,* (2011), materials intended for pozzolanic use should have at least 70% passing, i.e., no more than 15 g retained. None of the samples met this criterion, suggesting the need for post-calcination grinding. The poor fineness at 850 °C may be due to particle agglomeration or sintering at higher temperatures.

Loss on ignition (LOI) was determined according to NBR NM 18 (2012), in the absence of a specific standard for CCA. LOI increased with temperature: 65% at 650 °C, 70% at 750 °C, and 78% at 850 °C, reflecting progressive decomposition of organic components such as cellulose, hemicellulose, and lignin. These results align with values reported by Mota (2020) for sugarcane bagasse ash (81–82%), which typically contains more volatiles. The findings confirm that even at 850 °C, thermal treatment effectively removes organic matter from CCA. Specific gravity varied with calcination temperature: the highest value was recorded at 750 °C (2.87 g/cm³), followed by 850 °C (2.68 g/cm³) and 650 °C (2.31 g/cm³). These differences likely reflect temperature-induced changes in mineral structure and porosity. Compared to the 2.19 g/cm³ reported by Fiegenbaum (2020), the values found here underscore the strong influence of thermal processing on ash properties, particularly regarding phase composition and particle compactness.

Particle size distribution was assessed for CCA samples calcinated at 650 °C, 750 °C, and 850 °C for 4 hours using sieves ranging from 2.0 mm to 0.075 mm. The 850 °C sample showed the highest proportion of fine particles, primarily retained on the 0.15 mm and 0.075 mm sieves, indicating greater thermal degradation. The 750 °C sample displayed a more balanced distribution, while the 650 °C ash retained more material in coarser sieves, suggesting less efficient fragmentation. These trends are illustrated in Figure 2.

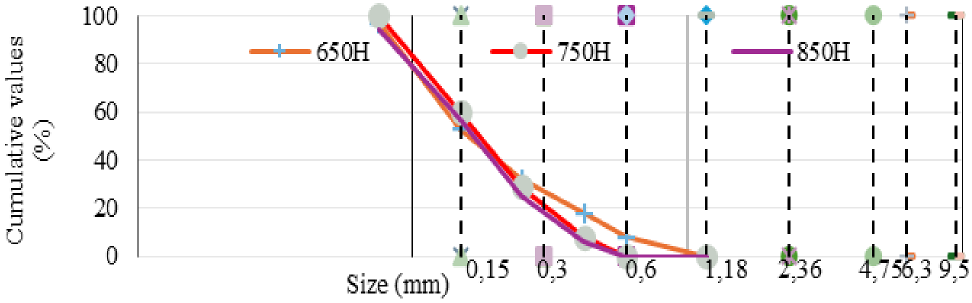


**Fig. 2**. Particle size distribution of CCA

In terms of visual appearance, a clear color gradient was observed with increasing temperature (Fig.3): the 650 °C (a) sample was dark gray, indicative of incomplete combustion and residual carbon; the 750 °C (b) sample presented a medium gray color; and the 850 °C (c) ash was nearly white, suggesting more complete oxidation and higher mineral purity.

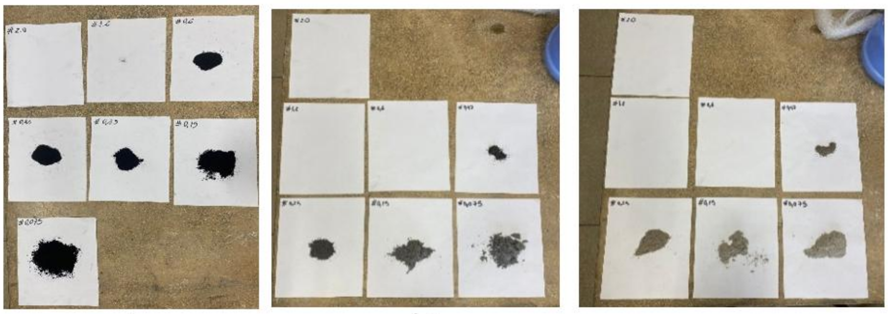


**Fig. 3.** Particle size distribution of calcined corn cob ash samples: (a) Retention of the 650°C sample on each sieve; (b) Retention of the 750°C sample on each sieve; (c) Retention of the 850°C sample on each sieve

These findings demonstrate that higher calcination temperatures not only improve particle size refinement but also enhance the visual and potentially chemical quality of the ash—important factors for its use as a supplementary cementitious material. XRD analysis revealed that quartz ($SiO_2$) was present in all CCA samples. Magnesium silicates, such as orthochrysotile and clinochrysotile, were also detected, appearing only in 750-2H, indicating a possible phase transition induced by specific thermal conditions; such phases can facilitate the formation of C-M-S-H, enhancing microstructural densification and durability. Soluble salts like sylvite (KCl) and halite (NaCl/KCl) were consistently present across samples and, although common in biomass-derived ashes, may induce undesirable effects such as efflorescence, higher ionic conductivity, or alkali–silica reactions.

ISSN: 2675-6218 - RECIMA21

REVISTA CIENTÍFICA - RECIMA21 ISSN 2675-6218



Trace levels of arsenic-bearing phases, including realgar, schneiderhohnite, and rooseveltite (the latter exclusive to 750-2H), were also identified, reinforcing the need for leaching and immobilization assessments to ensure environmental safety. Additionally, the detection of 2-fluoronaphthalene exclusively in the 850-4H sample points to potential recombination of residual organics or incomplete combustion, with associated environmental or health concerns. Overall, calcination at 750 °C for 2 hours yielded the most favourable profile, combining increased amorphous content with the emergence of potentially reactive mineral phases. Summarized results are provided in Table 3.

**Table 3.** Phases found in samples

| Phase / Compound | 650-2H | 650-4H | 750-2H | 750-4H | 850-2H | 850-4H | Notes |
|---|---|---|---|---|---|---|---|
| Quartz ($SiO_2$) | ✓✓ | ✓✓ | ✓ | ✓✓ | ✓✓ | ✓✓ | Strong in most; lower in 750-2H |
| Sylvite (KCl) | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | Stable; detected in all conditions |
| Halite (NaCl/KCl) | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | May require washing before use |
| Orthochrysotile | ✓ | ✓ | – | ✓ | ✓ | ✓ | Mg silicate; stable with higher T and time |
| Clinochrysotile | – | – | ✓ | – | – | – | Found only in 750-2H; suggests structural transition |
| Realgar (AsS) | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | Trace levels; environmental attention recommended |
| Schneiderhohnite | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | Iron-arsenate; consistent in all samples |
| Rooseveltite ($BiAsO_4$) | – | – | ✓ | – | – | – | Detected only at 750-2H |
| Jokokuite ($MnSO_4{\cdot}5H_2O$) | ✓ | – | – | – | – | – | Only in 650-2H; disappears with higher T or time |
| 2-Fluoronaphthalene ($C_{10}H_7F$) | – | – | – | – | – | ✓ | Organic aromatic; forms only at high T and longer exposure (850-4H) |

At 650 °C, the XRD patterns (Figure 4) indicate that the sample calcined for 2 hours shows slightly sharper and more intense crystalline peaks of $SiO_2$, along with a distinguishable amorphous halo. In contrast, the sample burned for 4 hours exhibits lower peak intensity and a broader, more diffuse halo between 15° and 35° 2θ. These changes suggest a progressive reduction in crystallinity and an increase in amorphous content with longer calcination time.

ISSN: 2675-6218 - RECIMA21

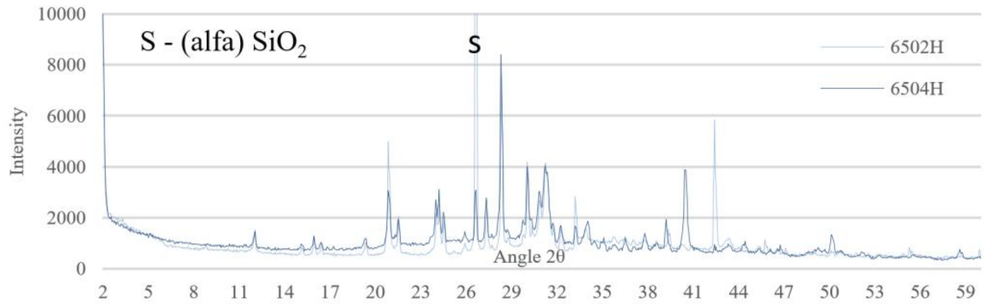


**Fig. 4**. $SiO_2$ peak results for samples calcined at 650°C for 2 and 4 hours

At 750 °C (Figure 5), the sample calcined for 4 hours displayed more intense $SiO_2$ peaks and a clearer crystalline pattern compared to the 2-hour sample, which exhibited weaker diffraction peaks and a more prominent amorphous halo.

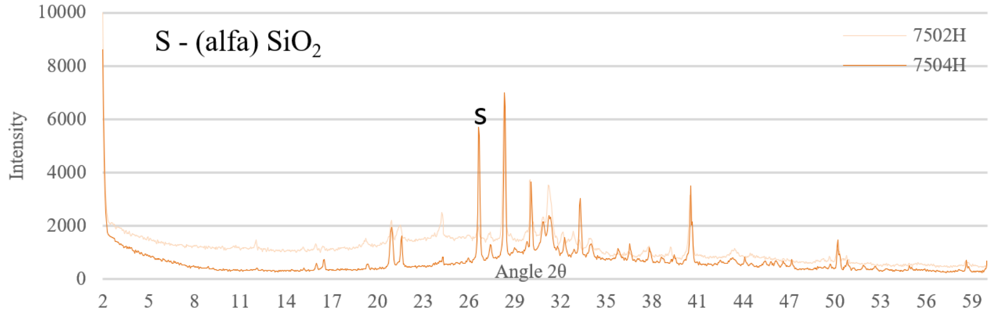


**Fig. 5.** $SiO_2$ peak results for samples calcined at 750°C for 2 and 4 hours.

At 850 °C (Figure 6), the sample calcined for 2 hours exhibited sharper and more intense $SiO_2$ peaks, reflecting a higher degree of crystallinity. The 4-hour sample, on the other hand, displayed lower peak intensity and a broader, elevated amorphous halo between 15° and 35° 2θ. This supports the interpretation of partial crystallinity loss due to prolonged heating, likely caused by sintering or vitrification processes at this temperature. This suggests that prolonged exposure at this intermediate temperature promotes crystallization, as also reported by Adesanya and Raheem [12] and Suwanmaneechot *et al.,* [11].

ISSN: 2675-6218 - RECIMA21

REVISTA CIENTÍFICA - RECIMA21 ISSN 2675-6218



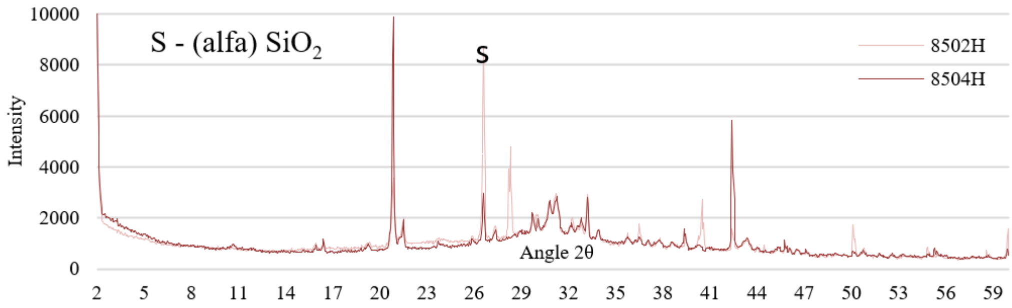


**Fig. 6**. $SiO_2$ peak results for samples calcined at 850°C for 2 and 4 hours

Figures 7 and 8 present a comparative analysis of samples subjected to calcination at 650 °C, 750 °C, and 850 °C for 2 and 4 hours, respectively. In the 2-hour calcination (Figure 7), the sample treated at 750 °C exhibited the most pronounced amorphous halo, especially between 15° and 35° 2θ, indicating the highest degree of structural disorder and, consequently, greater pozzolanic potential. Among the 4-hour calcination conditions (Figure 8), the sample treated at 650 °C exhibited the most favorable structural balance. It showed a broad amorphous halo indicative of pozzolanic potential while retaining sharp and intense quartz peaks, particularly at 26.6° 2θ. Therefore, 650 °C for 4 hours appears to be the most effective condition among the prolonged calcinations, preserving both reactivity and structural clarity. A condensed expression of results is exhibited in Tab 4.

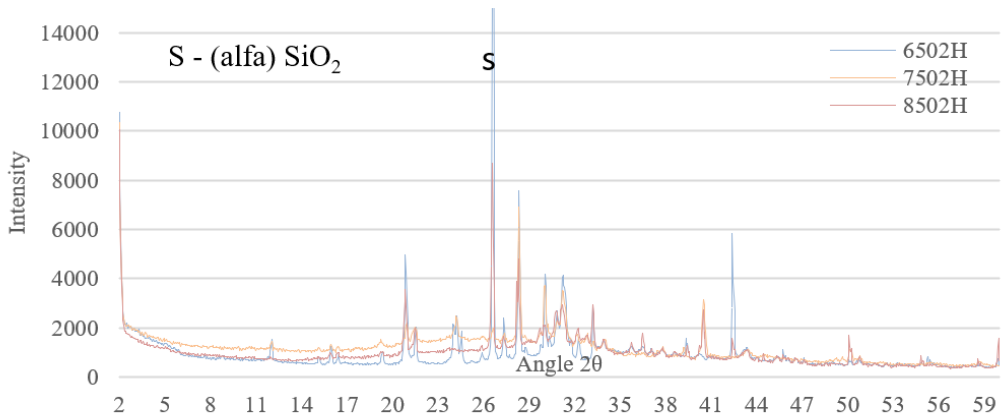


**Fig. 7**. Comparative XRD results for samples calcined at 650°C, 750°C, and 850°C for 2 hours

REVISTA CIENTÍFICA - RECIMA21 ISSN 2675-6218



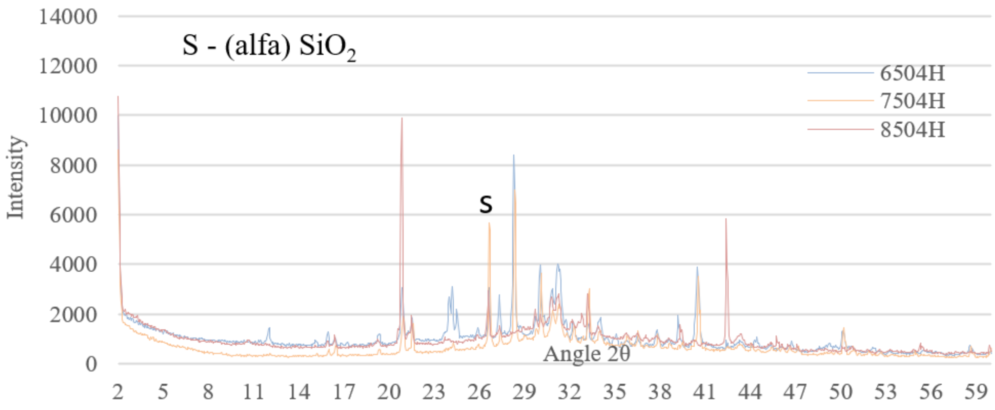


**Fig. 8**. Comparative XRD results for samples calcined at 650°C, 750°C, and 850°C for 4 hours

These results demonstrate that both calcination temperature and duration significantly affect the mineralogical transformation of corn cob ash. Higher temperatures tend to increase crystallinity, while extended durations may lead to structural reorganization, reducing amorphous content and altering phase composition.

The crystallinity index of $SiO_2$ (CrI_$SiO_2$), calculated from the quartz peak (~26.65° 2θ) and the amorphous minimum (18–22° 2θ), revealed clear trends influenced by calcination conditions, as shown in Tab 4. The 750 °C/2h sample showed the lowest crystallinity (45.1%), indicating the highest degree of structural disorder and suggesting greater pozzolanic potential. In contrast, the 650 °C/2h sample exhibited the highest crystallinity (97.5%), reflecting the dominance of well-ordered quartz.

**Tab. 4**. Semi-quantitative crystallinity index (CrI_$SiO_2$) based on XRD analysis, calculated from quartz peak intensity (26.6° 2θ) and the amorphous background region (18–22° 2θ) for each calcined corn cob ash sample

| Sample | $I_{26.6°}$ | $I_{am}$ | $CrI_{SiO2}$ (%) |
|---|---|---|---|
| 650-2H | 20442 | 507 | 97.52 |
| 650-4H | 3083 | 729 | 76.35 |
| 750-2H | 1978 | 1085 | 45.15 |
| 750-4H | 5692 | 316 | 94.45 |
| 850-2H | 8713 | 752 | 91.37 |
| 850-4H | 2944 | 646 | 78.06 |

Extended calcination time generally increased crystallinity, as seen in the 750 °C/4h (94.4%) and 850 °C/2h (91.4%) samples, indicating recrystallization with prolonged exposure. The 850 °C/4h sample showed a slightly lower value (78.1%), suggesting partial structural degradation. At 650 °C, increasing the duration to 4h reduced the CrI to 76.4%, showing mild amorphization without sufficient energy for complete transformation. Overall, calcination at 750 °C for 2 hours provided the most favorable condition for generating amorphous, reactive silica in corn cob ash.

## 5. CONCLUSIONS

Calcination temperature and duration significantly affect the reactivity and structural quality of corn cob ash (CCA) for use as a supplementary cementitious material. Among the tested conditions, 750 °C for 2 hours produced the most reactive ash, with high amorphous content and reduced crystallinity. 650 °C for 4 hours yielded the best structural balance among prolonged treatments, preserving both amorphous and crystalline features. In contrast, 850 °C led to higher crystallinity and coarser particles, reducing its pozzolanic potential. These results highlight 750 °C/2h and 650 °C/4h as the most promising calcination conditions for partial cement replacement.

## 6. ACKNOWLEDGMENTS

The authors acknowledge the financial support of the Coordination for the Improvement of Higher Education Personnel (CAPES), the University of Brasília (UnB), the Brazilian National Council for Scientific and Technological Development (CNPq), and the Research Support Foundation of the Federal District (FAPDF). The authors also gratefully acknowledge the Federal Institute of Tocantins (IFTO) and the Estácio University Center of Brasília.

## REFERENCES

ADESANYA, D. A.; RAHEEM, A. A. Characterization of corn cob ash (CCA) and groundnut shell ash (GSA) as pozzolana. **Journal of Minerals and Materials Characterization and Engineering**, v. 7, n. 2, p. 93–101, 2008. DOI: 10.4236/jmmce.2008.72008.

ADESANYA, D. A.; RAHEEM, A. A. Development of corn cob ash blended cement. **Construction and Building Materials**, v. 23, n. 1, p. 347–352, 2009. DOI: 10.1016/j.conbuildmat.2007.11.013.

ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. **NBR 11579**: Cimento Portland – Determinação da finura por meio da peneira 75 µm. Rio de Janeiro: ABNT, 1991.

ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. **NBR 12653**: Materiais pozolânicos – Requisitos. Rio de Janeiro: ABNT, 2014.

ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. **NBR 7181**: Solo – Análise granulométrica. Rio de Janeiro: ABNT, 2018.

ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. **NBR NM 18**: Análise química – Determinação de perda ao fogo. Rio de Janeiro: ABNT, 2012.

REVISTA CIENTÍFICA - RECIMA21 ISSN 2675-6218



COMPANHIA NACIONAL DE ABASTECIMENTO (CONAB). **Acompanhamento da Safra Brasileira**: Safra 2021/22: décimo primeiro levantamento. Brasília: CONAB, 2022. v. 9.

DESAI, J. Experimental study on corn cob ash powder as partial replacement of cement in concrete. International **Research Journal of Engineering and Technology**, v. 5, n. 6, p. 724–728, 2018.

FIEGENBAUM, F. A. **Cinzas de biomassa como material pozolânico**: avaliação da influência das características químicas, mineralógicas e morfológicas. 2020. Tese (Doutorado) – Universidade Federal do Rio Grande do Sul, Porto Alegre, 2020.

GÖTZE, J.; PAN, Y.; MÜLLER, A. Mineralogy and mineral chemistry of quartz: A review. **Mineralogical Magazine**, v. 85, n. 5, p. 639–664, 2021. DOI: 10.1180/mgm.2021.48.

JEFFRIES, D. F. Pozzolanas – Their properties and use in concrete. **Cement and Concrete Composites**, v. 12, n. 3, p. 165–170, 1990. DOI: 10.1016/0958-9465(90)90007-F.

KOMALPREET SINGH, K.; KUMAR, R.; BANSAL, P. A sustainable environmental study on corn cob ash subjected to elevated temperature. **Current World Environment**, v. 13, n. 1, p. 144–150, 2017.

MADSEN, I. C.; SCARLETT, N. V. Y.; KERN, A. Description and survey of methodologies for the determination of amorphous content via X-ray powder diffraction. **Zeitschrift für Kristallographie – Crystalline Materials**, v. 226, n. 12, p. 944–955, 2011. DOI: 10.1524/zkri.2011.1437.

MOTA, M. C. R. **Avaliação do uso de cinza de sabugo de milho como adição mineral ao cimento Portland**. 2020. Dissertação (Mestrado) – Universidade Federal de Goiás, Goiânia, 2020.

NNOCHIRI, E. S. Effect of corn cob ash on lime stabilized lateritic soil. **Journal of Civil Engineering**, Special Issue, p. 73–85, 2018. DOI: 10.1515/sspjce-2018-0007.

OLUBORODE, O. O.; OLOFINTUYI, O. A. Pozzolanic potentials of corn cob ash. **Journal of Emerging Trends in Engineering and Applied Sciences**, v. 2, n. 3, p. 561–564, 2011.

RRUFF DATABASE. Quartz – $SiO_2$ (ID: R100134). Disponível em: https://rruff.info/quartz/R100134

SCRIVENER, K.; SNELLINGS, R.; LOTHENBACH, B. **A practical guide to microstructural analysis of cementitious materials**. Boca Raton: CRC Press, 2016.

SEGAL, L.; CREELY, J. J.; MARTIN JR., A. E.; CONRAD, C. M. An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer. **Textile Research Journal**, v. 29, n. 10, p. 786–794, 1959. DOI: 10.1177/004051755902901003.

SUWANMANEECHOT, P.; NOCHAIYA, T.; JULPHUNTHONG, P. Improvement, characterization and use of waste corn cob ash in cement-based materials. **IOP Conference Series: Materials Science and Engineering**, v. 103, n. 1, p. 012023, 2015. DOI: 10.1088/1757-899X/103/1/012023.

UDOEYO, F. F.; ABUBAKAR, S. A. Characteristics of maize cob ash blended cement. **Construction and Building Materials**, v. 19, n. 8, p. 570–576, 2005.

**ISSN: 2675-6218 - RECIMA21**

REVISTA CIENTÍFICA - RECIMA21 ISSN 2675-6218



UDOEYO, F. F.; ABUBAKAR, S. A. Maize-cob ash as filler in concrete. **Journal of Materials in Civil Engineering**, v. 15, n. 2, p. 205–208, 2003. DOI: 10.1061/(ASCE)0899-1561(2003)15:2(205).

VAZ JUNIOR, S. **Utilization of agro-industrial residues**: A sustainable approach. Brasília: Embrapa Agroenergy, 2020. (Documents No. 31).