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CHARACTERIZATION OF CASSAVA STARCH BAGASSE ASH FOR USE IN CEMENTITIOUS MATRICES

CARACTERIZAÇÃO DA CINZA DO BAGAÇO DE FÉCULA DE MANDIOCA PARA USO EM MATRIZES CIMENTÍCIAS

CARACTERIZACIÓN DE LA CENIZA DEL BAGAZO DE FÉCULA DE YUCA PARA USO EN MATRICES CEMENTICIAS

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ABSTRACT

Cassava starch bagasse ash (CSBA) is an agro-industrial byproduct abundantly generated in Brazil, yet its applications in civil construction remain underexplored. This study evaluates the potential of CSBA as a supplementary cementitious material through a detailed chemical, physical, and thermal characterization. The experimental methodology included drying and milling the bagasse, followed by thermal treatments at 650°C, 900°C, and 1000°C to produce ashes with varying properties. Analytical techniques such as Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), and differential scanning calorimetry (DSC) were employed to determine its composition and thermal behavior. Microscopic analysis and specific gravity tests assessed physical properties. Results highlighted that combustion at 650°C produced the highest amorphous silica content, which enhances pozzolanic activity. Despite its low silica concentration compared to Portland cement, CSBA demonstrated favorable characteristics for partial replacement in cementitious matrices. This study contributes to sustainable construction by promoting the circular use of agro-industrial residues and reducing environmental impacts. Furthermore, it provides a pathway for incorporating CSBA into eco-friendly concrete and mortar formulations, addressing the need for innovative and sustainable materials in civil engineering.

KEYWORDS: *Agro-waste. Agro-cement. Cassava bagasse.*

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RESUMO

A cinza do bagaço de fécula de mandioca (CSBA) é um subproduto agroindustrial abundantemente gerado no Brasil, porém suas aplicações na construção civil ainda são pouco exploradas. Este estudo avalia o potencial da CSBA como material cimentício suplementar por meio de uma caracterização química, física e térmica detalhada. A metodologia experimental incluiu a secagem e moagem do bagaço, seguidas de tratamentos térmicos a 650°C, 900°C e 1000°C para a produção de cinzas com propriedades distintas. Técnicas analíticas como espectroscopia no infravermelho por transformada de Fourier (FTIR), difração de raios X (DRX), espectroscopia de energia dispersiva de raios X (EDX) e calorimetria exploratória diferencial (DSC) foram empregadas para determinar sua composição e comportamento térmico. Análises microscópicas e ensaios de massa específica avaliaram as propriedades físicas. Os resultados destacaram que a combustão a 650°C produziu o maior teor de sílica amorfa, o que favorece a atividade pozolânica. Apesar da baixa concentração de sílica em comparação ao cimento Portland, a CSBA apresentou características favoráveis para substituição parcial em matrizes cimentícias. Este estudo contribui para a construção sustentável ao promover o uso circular de resíduos agroindustriais e reduzir impactos ambientais. Além disso, fornece um caminho para a incorporação da CSBA em formulações de concretos e argamassas ecológicos, atendendo à necessidade de materiais inovadores e sustentáveis na engenharia civil.

PALAVRAS-CHAVE: Resíduo agroindustrial. Agrocimento. Bagaço de mandioca.

RESUMEN

La ceniza del bagazo de fécula de yuca (CSBA) es un subproducto agroindustrial abundantemente generado en Brasil, sin embargo, sus aplicaciones en la construcción civil aún están poco exploradas. Este estudio evalúa el potencial de la CSBA como material cementante suplementario mediante una caracterización química, física y térmica detallada. La metodología experimental incluyó el secado y la molienda del bagazo, seguidos de tratamientos térmicos a 650°C, 900°C y 1000°C para producir cenizas con diferentes propiedades. Técnicas analíticas como la espectroscopía infrarroja por transformada de Fourier (FTIR), la difracción de rayos X (DRX), la espectroscopía de energía dispersiva de rayos X (EDX) y la calorimetría diferencial de barrido (DSC) fueron empleadas para determinar su composición y comportamiento térmico. El análisis microscópico y los ensayos de masa específica evaluaron las propiedades físicas. Los resultados destacaron que la combustión a 650°C produjo el mayor contenido de sílice amorfa, lo que favorece la actividad puzolánica. A pesar de su baja concentración de sílice en comparación con el cemento Portland, la CSBA mostró características favorables para su sustitución parcial en matrices cementantes. Este estudio contribuye a la construcción sostenible al promover el uso circular de residuos agroindustriales y reducir los impactos ambientales. Además, proporciona una vía para la incorporación de la CSBA en formulaciones de hormigones y morteros ecológicos, respondiendo a la necesidad de materiales innovadores y sostenibles en la ingeniería civil.

PALABRAS CLAVE*: Residuo agroindustrial. Agrocemento. Masa de yuca.*

INTRODUCTION

The challenges associated with solid waste management remain persistent in society, requiring not only solutions for proper disposal but also strategies to systematically reintegrate these materials into production cycles. One concept that supports the reuse of discarded resources is the circular economy, which offers a promising alternative for managing solid waste through the identification of potential applications and the validation of environmental and economic efficiency (Cosenza, Andrade e Assunção, 2020; Foster, Roberto e Igari, 2016; Silva *et al.*, 2021). Thus, the use of renewable resources, the reduction of non-renewable resource consumption, and the promotion of reuse and recycling contribute to minimizing environmental impacts and supporting sustainable development. In this context, Brazil's Law 12.305/2010 (Brasil, 2010) establishes guidelines for integrated solid waste management and encourages the development of management systems aimed at improving production processes and waste utilization.

In this context, the construction industry has demonstrated the capacity to incorporate waste and byproducts into various applications, including the substitution of natural aggregates with recycled materials, the use of alternative cementitious materials, and the incorporation of industrial byproducts into construction systems (Carvalho *et al.*, 2018; Figueiredo *et al.*, 2022; Figueiredo, Reis e Maia, 2022; Friol Guedes de Paiva *et al.*, 2021; Garcia Lodeiro *et al.*, 2020; Pinaffi *et al.*, 2013). The use of industrial byproducts as supplementary cementitious materials is of particular interest because it can reduce energy consumption, lower CO_2 emissions associated with cement production, and decrease the exploitation of natural resources (Bellmann e Stark, 2009; Benittez, 2020; Gupta, Siddique e Belarbi, 2021; Keles, 2011; Santhosh, Subhani e Bahurudeen, 2021).

The potential application of agro-industrial ashes in cementitious systems is strongly related to their chemical, mineralogical, and thermal characteristics, especially the presence of reactive silica and the formation of amorphous phases after calcination. In this sense, calcination temperature plays a fundamental role in the physicochemical transformations of these materials, directly influencing crystallinity and potential pozzolanic behavior. Therefore, the characterization of agricultural residues under different thermal treatment conditions is essential for evaluating their compatibility with cementitious matrices.

Among agro-industrial residues, cassava starch bagasse, a byproduct of cassava starch production, is currently generated in large quantities in Brazil. According to the Food and Agriculture Organization (FAO), global cassava production in 2018 reached 277.8 million tons. Brazil ranks among the world's largest cassava producers, with production of approximately 47 million tons, second only to Nigeria, followed by Thailand and Indonesia (Albuquerque, 2022). However, cassava processing and the residues generated during the production stages represent potential sources of environmental contamination and are considered critical waste points within the production process (Barros, Santos e Batistote, 2019; Oliveira, 2013).

Cassava starch bagasse consists mainly of fibrous material with high moisture content and high perishability, which limits its large-scale reuse and commercial application. Currently, only a small portion of this residue is used as supplementary animal feed due to difficulties associated with drying, transportation, and storage (Peixoto e Resch, 2018; Woiciechowski *et al.*, 2013). In addition, according to Coelho *et al.* (2020), the chemical composition and potential applications of cassava starch bagasse remain insufficiently explored, limiting the understanding of its possible use in engineering materials.

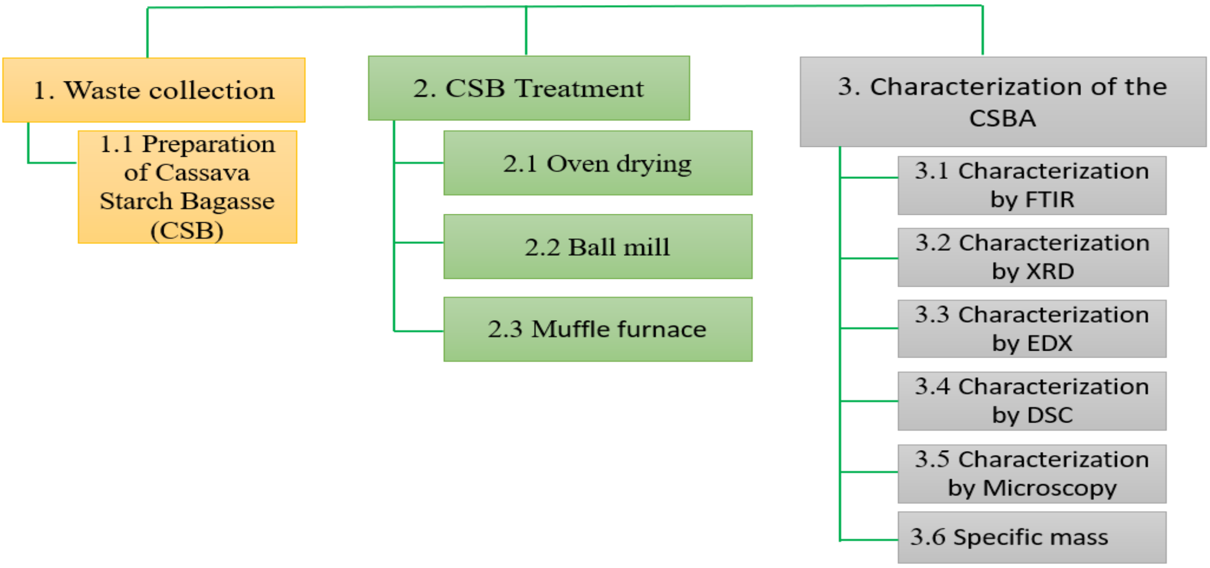
To address environmental concerns and promote sustainable alternatives, several studies have investigated the use of agricultural residues such as sugarcane bagasse ash, rice husk ash, sugarcane straw ash, and palm oil residues in concrete and mortar production (Barroso, 2011; Ganesan, Rajagopal e Thangavel, 2007; He, Kawasaki e Achal, 2020). However, despite the growing interest in agro-industrial ashes as supplementary cementitious materials, limited information is available regarding the influence of calcination temperature on the thermal, mineralogical, and chemical transformations of cassava starch bagasse ash (CSBA), particularly concerning the formation of amorphous silica and its potential compatibility with cementitious matrices.

Therefore, this study hypothesizes that controlled thermal treatment of cassava starch bagasse can promote mineralogical transformations capable of generating a material with characteristics compatible with supplementary cementitious applications. Thus, the present study aims to evaluate the feasibility of thermally treated cassava starch bagasse ash as a supplementary cementitious material by characterizing the chemical, mineralogical, thermal, and physical properties of the material under different burning conditions, as well as investigating its potential application in civil construction.

2. METHODOLOGY

This research can be classified as an experimental, laboratory-based, and exploratory study with a quantitative approach, since it involved controlled thermal treatments and physicochemical characterization tests to evaluate the potential application of cassava starch bagasse ash (CSBA) as a supplementary cementitious material. To achieve the objectives established in this study, the experimental program was divided into three stages, as indicated in Figure 1, and detailed in the following sections.

Figure 1. Methodological Process Scheme



2.1. Characterization of the raw residue

The residue used in this study was supplied by a cassava processing company (COPASUL), a starch factory located in the municipality of Naviraí, in the state of Mato Grosso do Sul, Brazil. The material is a byproduct of the industrialization process of cassava roots for starch production. The cassava starch bagasse was placed on trays for drying in ovens at 160°C for 8 hours. After drying, the material was removed, weighed, and sent to a ball mill, where it was ground for 30 minutes to achieve a particle size similar to cement, resulting in a fine powder that passed through a 75 μm sieve. Following this stage, the powder was subjected to characterization tests, including loss on ignition, moisture content, and mass loss in the oven.

2.2. Production and characterization of ash

Subsequently, the cassava starch bagasse powder (CSRP) was subjected to thermal treatment in muffle furnaces at different temperatures: 650 °C, 900 °C, and 1000 °C. The heating rate adopted for all thermal treatments was 100 °C h^{-1} until the target temperature was reached. The samples were placed in crucibles, each containing 70 g of material, and maintained at the target temperature for 2 h. The total thermal treatment durations varied according to the temperature level due to the heating ramp required to reach each target temperature at a constant heating rate of 100 °C h^{-1}, resulting in total durations of 3 h and 4 h for the 900 °C and 1000 °C treatments, respectively.

The selected thermal treatment temperatures were defined based on previous studies involving agro-industrial ashes and supplementary cementitious materials, aiming to evaluate the influence of calcination temperature on the formation of amorphous phases, reduction of organic matter, and modification of the physicochemical properties of the ash. The temperature of 650 °C was selected due to its potential to preserve amorphous silica phases, while the higher temperatures (900 °C and 1000 °C) were adopted to investigate possible mineralogical transformations and changes in crystallinity caused by more severe thermal conditions.

All experimental procedures and quantitative analyses were performed at least in triplicate to ensure repeatability and experimental reliability. The samples described were labeled as shown in Table 1.

Table 1. Terminology, temperature, and exposure time of the samples

Samples		
Nomenclature	**Temperature**	**Exposure time to maximum temperature**
CSBA1	650ºC	2 hours
CSBA2	900ºC	2 hours
CSBA3	900ºC	3 hours
CSBA4	1000ºC	2 hours
CSBA5	1000ºC	4 hours

2.2.1. Characterization by FTIR (Fourier Transform Infrared Spectroscopy)

FTIR characterization was performed, and the resulting data were presented in two types of graphs: absorbance versus wavenumber and transmittance versus wavenumber. The analysis of the peaks identified in the spectra, along with the comparison of these peaks with existing literature, enables the identification of the chemical compounds in the material, such as SiO_2 and SiOH.

The analysis was conducted using pellets produced by a manual press, which consisted of a mixture of the addition/aggregating agent with pure KBr in a 1:200 ratio.

2.2.2 Characterization by XRD (X-Ray Diffraction)

The samples were exposed to X-rays using the Shimadzu Lab X XRD 6000 equipment, which features a Cu X-ray tube with a maximum output of 2 kW and a vertical goniometer with a scanning radius of 185 mm, located at the Department of Physics at the Federal University of São Carlos (UFSCar). The measurement was conducted between 10° and 60°, with an angular step of 2° per minute.

2.2.3 Characterization by EDX (Energy Dispersive X-Ray Spectroscopy)

For this technique, the sample was exposed to X-rays using the Shimadzu EDX-720 equipment, capable of both qualitative and quantitative analysis of elements with atomic weights ranging from Na to Uranium, at the Ferrous Materials Group (GMF) of UFSCar.

2.2.4 Characterization by DSC (Differential Scanning Calorimetry)

The technique was performed to evaluate the behavior of the cassava starch bagasse powder and the CSBA. The equipment used was the Q-20 Ta Instruments Differential Scanning Calorimeter (DSC), which allowed for precise determination of enthalpy variations of the materials as a function of temperature and time. The analysis was carried out between temperatures of 0 °C and 600 °C (maximum temperature available for research with the equipment), using predetermined heating rates of 5 °C min^{-1}.

2.2.5 Characterization by Optical Microscopy

This technique was essential to determine the average particle size of the CSBA, identify the presence or absence of agglomerates, and assess whether the sample exhibits a magnitude similar to that of cement.

2.2.6 Specific Gravity

The specific gravity test was conducted for the following materials: lime, cement, and CSBA. For this, a Le Chatelier volumetric flask, made of borosilicate glass with a capacity of 250 cm^3 and precision readings of 0.05 cm^3, was used. Kerosene was employed as the fluid to determine the specific gravity of cement, CSBA, and calcium hydroxide (analytical grade).

The flask with kerosene was submerged in a vertical position for 30 minutes in natural water at ambient temperature to ensure thermal equilibrium of the liquids. After this time, the material mass, for which the specific gravity was to be determined, was added to the flask. The mass was sufficient to displace the liquid to a volume between 18 cm^3 and 24 cm^3. The flask was then sealed and gently rotated in a circular motion while tilted.

The specific gravity was calculated using Equation 1 (Brazilian Association of Technical Standards (ABNT), 2000):

$$\boldsymbol{\rho = \frac{m}{V}} \qquad \text{(Eq. 1)}$$

Where:

ρ, is the specific gravity of the tested material (g/cm^3);

m, is the mass of the tested material (g);

V, is the volume displaced by the mass of the tested material (cm^3).

Quantitative results are presented as mean values obtained from at least three independent measurements. When applicable, standard deviation values were calculated to evaluate experimental variability and repeatability.

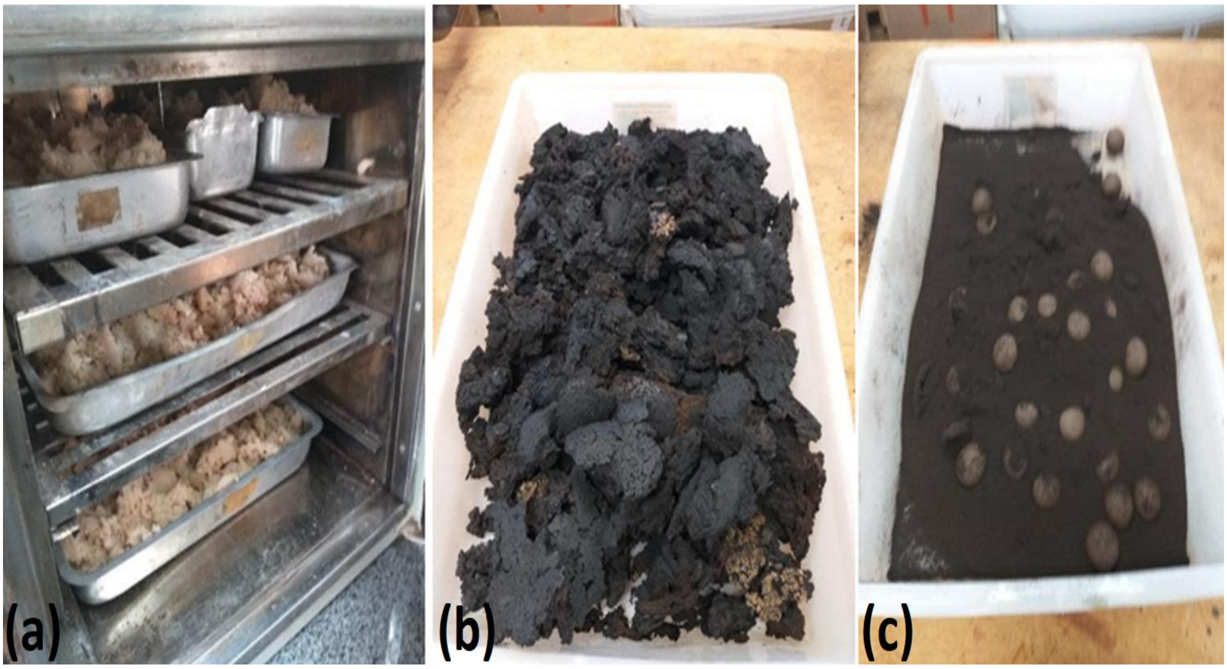
3. RESULTS AND DISCUSSION

The results of the characterization of the raw residue and the characterization of CSBA1 are presented below:

3.1. Characterization of the Raw Residue

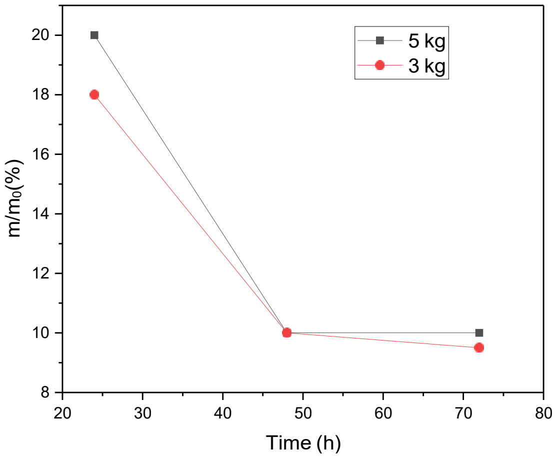
The raw cassava starch bagasse residue has a beige color, with a texture similar to cake batter, being moldable, moist, and odorous. Figure 2a illustrates the wet byproduct, Figure 2b shows the dried residue, and Figure 2c represents the material after ball milling for 30 minutes.

Figure 2. Stages of the Residue during the Preparation Process



The result of the loss on ignition test for the wet sample was analyzed by assessing the mass loss of the wet material when dried in an oven at 105°C for 24, 48, and 72 hours, using two different quantities (3 kg and 5 kg) placed in the same container. After the first 24 hours, the material exhibited a mass loss of over 80% ± 1.2% after drying. However, the material still retained a highly fibrous appearance and was difficult to fragment, which did not favor powder formation. Considering that the water content in the residue is approximately 85%, as indicated by (Fiorda, Soares e Júnior, 2013), stabilization of the mass loss was only observed after 48 hours, with a mass loss of around 90%, as shown in Figure 3.

Figure 3. Results of Mass Variation after Loss on Ignition Test



3.2. Production and characterization of ash

To ensure that the drying process was sufficiently effective and that the resulting material, prior to milling, was homogeneous and dry, the drying methodology for the SBP was optimized by reducing the time and increasing the temperature, performing an 8-hour drying process at 160°C.

After drying, the material was milled in a ball mill for 30 minutes. Only then was the bagasse burned in a muffle furnace at temperatures of 650°C, 900°C, and 1000°C for two hours, with heating ramps of 100°C per hour, as shown in Table 1.

The visual appearance of the cassava starch bagasse ash exhibited two characteristic colors: one white and one black, as observed in Figure 4, corresponding to the combustion of the cassava starch bagasse powder at 650°C for two hours.

Figure 4. Visual appearance of the starch residue after muffle furnace burning

3.2.1 Characterization by FTIR

FTIR spectroscopy was used to evaluate the structural and chemical changes in cassava starch bagasse powder (CSRP) before and after thermal treatment. Figures 5 and 6 present the FTIR spectra of the raw material and the ashes produced at different calcination temperatures, with peak identification based on data reported in the literature.

In the spectrum of the cassava starch bagasse powder (Figure 5), the bands observed near 1052 cm⁻¹ and 1101 cm⁻¹ are associated with Si–O–Si and O–Si–O vibrations, indicating the presence of silicate structures in the material (Chandrasekhar *et al.*, 2002; Real, 2018). The peak identified at 1384 cm⁻¹ is related to C–N stretching vibrations associated with benzamide compounds, while the band observed at 1618.95 cm⁻¹ corresponds to primary amide groups. These bands are characteristic of organic constituents naturally present in cassava starch bagasse and indicate the presence of organic matter and lignocellulosic compounds in the untreated material.

After thermal treatment (Figure 6), significant changes in the FTIR spectra were observed, especially the reduction or disappearance of bands associated with organic functional groups. This behavior suggests the decomposition of organic constituents during calcination, which is consistent with the mass loss observed in the thermal analysis. The reduction of these bands indicates increased thermal stabilization of the material after burning.

The broad band observed in the region between approximately 3000 and 3600 cm⁻¹ is associated with hydroxyl groups (OH), which may be related to adsorbed moisture, and hydroxylated compounds present in the ash. The band identified at 1432 cm⁻¹ is attributed to carbonate vibrations associated with $CaCO_3$ formation (Čiuladienė *et al.*, 2018), suggesting the presence of carbonate phases after calcination. Furthermore, the peak observed at 2359 cm⁻¹ is associated with carbonate-related compounds and vibrations linked to residual organic structures, such as –OH, $–CH_2$, $–CH_3$, N–H, and C–N groups (Haq *et al.*, 2019).

The band identified near 1117 cm⁻¹ may be associated with sulfate compounds, particularly potassium sulfate, as described by Verma e Deb (2007). The persistence of bands associated with silicate structures after calcination suggests the maintenance of mineral phases potentially relevant for supplementary cementitious applications. In addition, differences in band intensity between the ashes produced at 650 °C and 900 °C indicate that calcination temperature directly influences the physicochemical transformations of the material, particularly regarding the decomposition of organic compounds and the stabilization of inorganic phases.

Figure 5. FTIR spectrum curve of cassava starch bagasse powder

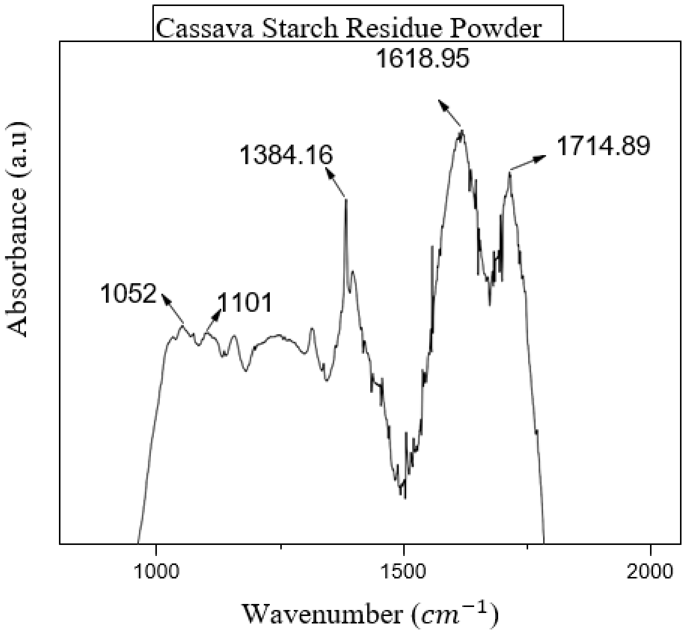
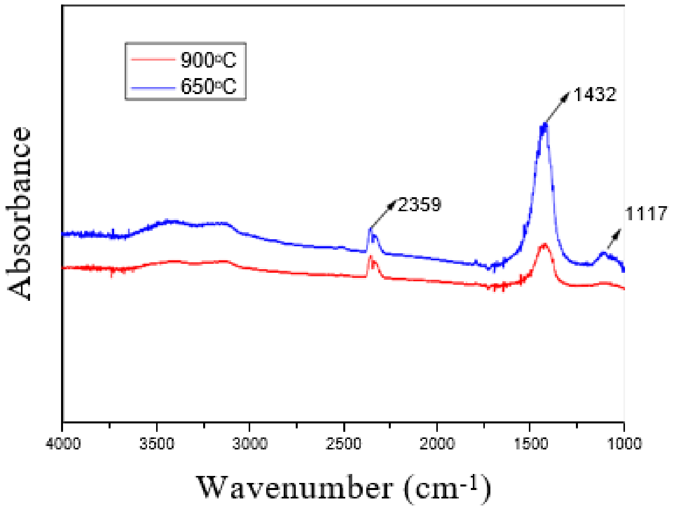


Figure 6. FTIR Spectrum Curve of CSBA after Burning at 650°C and 900°C

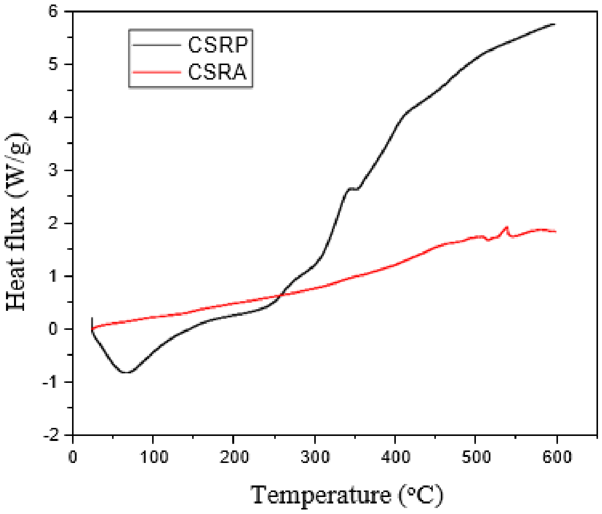


3.2.2. Characterization by DSC

In the analysis of heat gain and/or loss of the bagasse powder (BF), as observed in the graph in Fig. 7, prior to 100°C, the detected heat is below the baseline (negative values), indicating that the process released heat (exothermic direction of the axis). On the other hand, above 100°C, the thermal events occur above the baseline, indicating that the process absorbed heat (endothermic direction of the axis). Between 0°C and 100°C, water loss from the sample occurs, as also observed by (Nascimento *et al.*, 2015) in a DSC test conducted on a rice husk ash (RHA) sample.

It is also possible to observe heat loss transformations and fluctuations up to 350°C, possibly due to the burning of organic materials and/or the chemical transformation of some components of the sample. According to (Ernesto, 2009), within the temperature range of 150°C to 320°C, sucrose decomposition and partial decomposition of cellulose, hemicellulose, and lignin occur, leaving behind a black residue as a result of bagasse carbonization. At temperatures around 510°C and 540°C, the CSBA experiences heat loss, likely due to the combustion of residual starch, as shown in Figure 7, which is present in the sample at this temperature.

Figure 7. Heat Flow vs. Temperature curve obtained by differential scanning calorimetry (DSC) of cassava starch bagasse powder (CSRP) and CSBA after 650°C

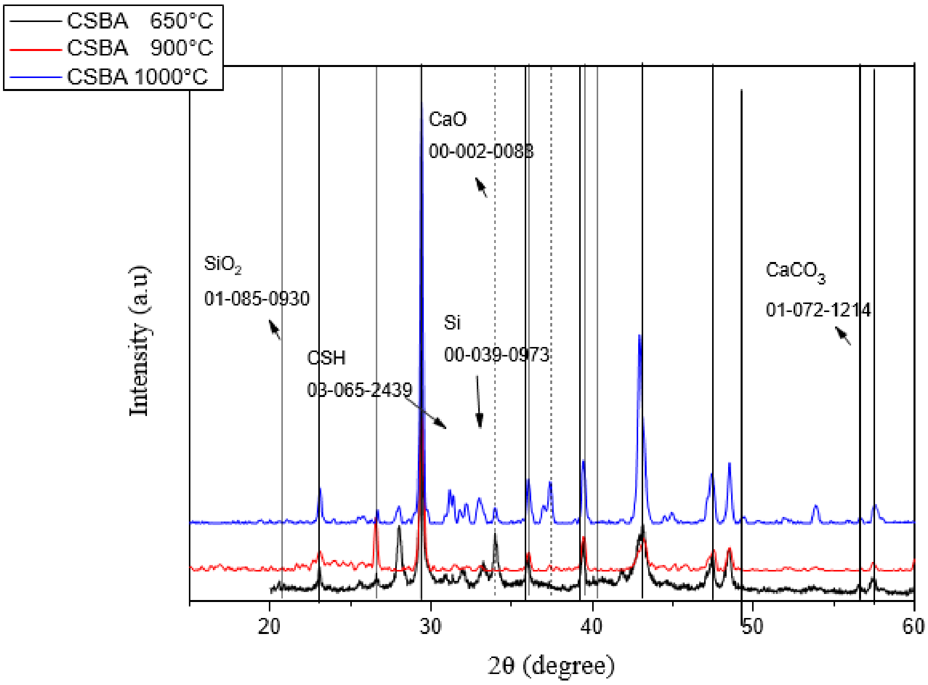


In the case of its application in cementitious composites, the thermometric behavior of cassava starch bagasse powder reinforces the need for thermal treatment to produce CSBA, since the raw material does not exhibit the same thermal stability observed in the ash. The presence of thermal events associated with the decomposition of organic compounds may interfere with the stability of cementitious systems. In contrast, CSBA exhibited a more stable DSC profile, suggesting greater thermal stability and the absence of significant thermal anomalies that could negatively affect the curing process of cementitious composites.

3.2.3. Characterization by XRD

The X-ray diffraction profiles of the CSBA samples are presented in Figure 8.

Figure 8. X-Ray diffraction profiles of CSBA after burning at 650°C, 900°C, and 1000°C



The X-ray diffraction peaks of the CSBA samples were indexed according to the International Centre of Diffraction Data (ICDD) files. Characteristic peaks of calcite (〖CaCO〗 _3) quartz 〖(SiO〗 _2),and calcium oxide (CaO) were identified.

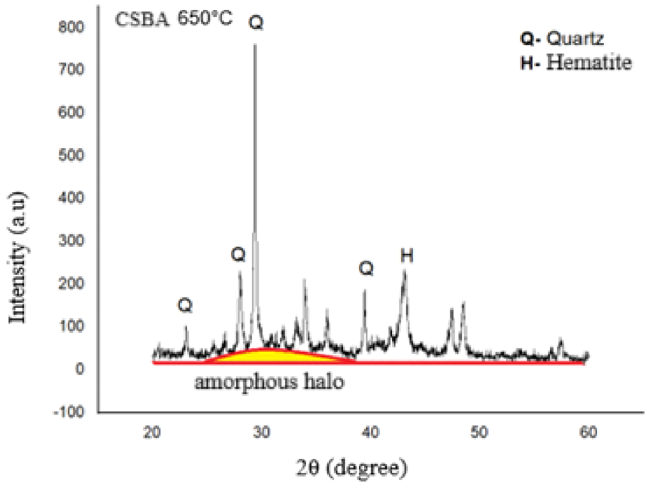
The peak located near 31° (2θ) may result from an overlap of peaks associated with calcium silicate hydrate (CSH) (Filho *et al.*, 2017; Menezes *et al.*, 2020; Romano *et al.*, 2018) and tricalcium and dicalcium silicates (Ghoddousi e Adelzade Saadabadi, 2017; Menezes *et al.*, 2020). CSH, the primary phase resulting from the hydration of Portland cement, has a significant influence on most physical and mechanical properties of cementitious materials (Pelisser, Gleize e Mikowski, 2009). Tricalcium silicate, or Alite (3CaO.SiO2), exhibits properties such as rapid hardening, high heat of hydration, and high early strength. Conversely, dicalcium silicate, or Belite (2CaO.SiO2), is characterized by slow hardening, low heat of hydration, low early strength, but high final strength (Ribeiro, 2002). Both are also constituents of Portland cement.

According to (Menezes *et al.*, 2020) and (Serpell e Lopez, 2015), additional peaks of dicalcium and tricalcium silicates can still be observed between 29° and 35° (2θ). Similar XRD results were obtained by (Batool, Masood e Ali, 2020; Jha, Sachan e Singh, 2021; Schettino e Holanda, 2015). The maximum silica peak in the form of quartz is observed at approximately 29.3° (2θ). The common diffraction angles (2θ) found in similar materials (agro-industrial residues), such as sugarcane bagasse ash (SCBA) and RHA, were observed at peaks in the range of 26.64°, 27.5°, 28.6°, and 30.86°, respectively, as reported by (Alavéz-Ramírez *et al.*, 2012; Castaldelli *et al.*, 2013; Jha, Sachan e Singh, 2021; Khan *et al.*, 2012).

It is known that the reactivity of pozzolans is influenced by the amount of amorphous Si present, which can be identified by X-ray diffraction through a characteristic halo (Hoppe Filho, 2008). There was no significant evidence of this halo formation around 29° (2θ) for the CSBA 900°C and 1000°C samples. However, a narrowing of the peak with increasing burning temperature was observed, characteristic of the crystalline formation of SiO2. The increase in the full width at half maximum reflects the material's crystallinity: the smaller the width, the more crystalline the element appears (Miranda, 2016).

However, when analyzing the diffractogram of the CSBA 650°C sample (Figure 9), a subtle but evident formation of this amorphous halo is observed, suggesting the presence of amorphous silica in this region. This indicates that 650°C would be the ideal burning temperature for the material when aiming to obtain amorphous Si.

Figure 9. XRD Profile of CSBA at 650°C Highlighting the Amorphous Halo



In the diffraction profile of the CSBA burned at 650°C, peaks corresponding to cristobalite, one of the polymorphic forms of silicon dioxide (SiO2), can also be identified at approximately 23° (2θ), as observed by (Hoppe Filho *et al.*, 2017) in rice husk ash. Additionally, the formation of a second amorphous halo related to the hematite peak can be noted, likely associated with the presence of Fe or alginate, as recorded by (Candido, 2019), in the amorphous halo near 40° (2θ).

3.2.4. Characterization by EDX

The compositional analysis result by EDX of the CSBA sample burned at 650°C revealed the presence of three main elements: Calcium (Ca), Potassium (K), and Silicon (Si), in addition to the presence of Phosphorus (P), Iron (Fe), and Sulfur (S) in smaller proportions, as shown in Table 2.

Table 2. Results of the compositional analysis via EDX of the CSBA burned at 650°C

Element	Concentration (%)
CaO	60.50%
K_2O	25.90%
SiO_2	4.20%
P_2O_5	3.23%
Fe_2O_3	3.06%
SO_3	1.14%
TiO_2	0.59%
MnO	0.57%
ZnO	0.39%
Sr	0.34%
Cu	0.08%

It was found that calcium (Ca) exhibited the highest concentration, followed by potassium (K) and silicon (Si). These results indicate a high concentration of alkali elements, particularly potassium K (alkali metal); Group 2, Ca (alkaline earth metal); and Group 14, Si (non-metal). Although these elements do not have a direct similarity to each other, each plays an important role in different chemical contexts and can form compounds with other elements to create materials for various applications.

Calcium and silicon are important constituents in cementitious systems due to their participation in hydration reactions and the formation of calcium silicate hydrate (C-S-H), which contributes to the mechanical performance of Portland cement-based materials (NEVILLE, 2016). However, the isolated presence of calcium does not necessarily indicate cementitious behavior, since the performance of supplementary materials depends on the balance between chemical composition, mineralogical phases, and reactivity.

The presence of reactive silica in aggregates, combined with high alkali contents (sodium and potassium) in cement, can lead to alkali-silica reaction (ASR). This reaction can cause expansion and cracking in the concrete due to the formation of expansive gel, impairing its durability. To mitigate ASR, it is important to control the alkali content in the cement and use appropriate aggregates (Hewlett e Liska, 2019; Neville, 2010).

Table 3. Results of the compositional analysis via EDX for CP-V cement (HSR)

Element %/ Author	CaO	SiO_2	Al_2O_3	MgO	Fe_2O_3	SO_3	K_2O

(Alexandre e Luz, 2020)	54.75	22.38	6.45	4.66	3.41	2.68	-
(Borges, Melo Neto e Mendonça, 2021)	64.60	17.00	6.90	0.90	2.70	3.40	1.30
(Behera, Noman e Petrů, 2020)	63.50	19.10	4.44	2.32	2.68	2.63	1.10

The Ca content observed in the CSBA was comparable to that identified in CP-V (HSR) Portland cement; however, this similarity alone does not indicate equivalent cementitious behavior. Other elements are also present in the composition of both materials, such as Si, Fe, K, and S. The main compositional differences were observed in the SiO_2 and K_2O contents found in both materials: while CP-V (HSR) Portland cement contains an average of 19.50% Si, the CSBA contains only 4.20%, and while the cement contains potassium in small amounts, the CSBA contains 25.90% potassium.

Although potassium may influence hydration kinetics under controlled conditions, excessive alkali contents can negatively affect the durability and dimensional stability of cementitious materials and increase the risk of alkali–silica reaction (Hewlett e Liska, 2019; Kosmatka, Kerkhoff e Panarese, 2002).

The elevated K_2O content identified in the CSBA deserves particular attention because high alkali concentrations in cementitious systems may increase the susceptibility to alkali–silica reaction (ASR) in the presence of reactive aggregates. This reaction may result in expansion, cracking, and durability-related problems in cement-based materials (Hewlett e Liska, 2019; Neville, 2010). Although potassium may influence hydration kinetics under controlled conditions, excessive alkali contents are generally considered a limiting factor for the application of supplementary cementitious materials. Therefore, despite the potential applicability of CSBA in cementitious matrices, the high potassium concentration indicates the need for additional studies involving alkali reactivity, durability, and long-term performance before practical application.

It is important to note that the presence of potassium in concrete is not necessarily harmful, as long as it is within appropriate limits. Proper control of additive dosage, material selection, and correct component proportions are crucial to prevent problems caused by excess potassium in concrete.

3.2.5. Characterization by Microscopy

For this characterization of CSBA, an optical microscopy analysis was performed to investigate the particle morphology of the ash.

In Figures 10 and 11, it is possible to observe the CSBA at two magnifications, where it can be noted that there is no formation of large agglomerates, with a good dispersion of particles with estimated average sizes ranging from 0.02 to 0.05 mm (20 to 50 µm), as the CSBA passed through a 45 µm sieve for the analysis.

Figure 10. Optical microscopy image of CSBA after burning at 650°C with 5x magnification

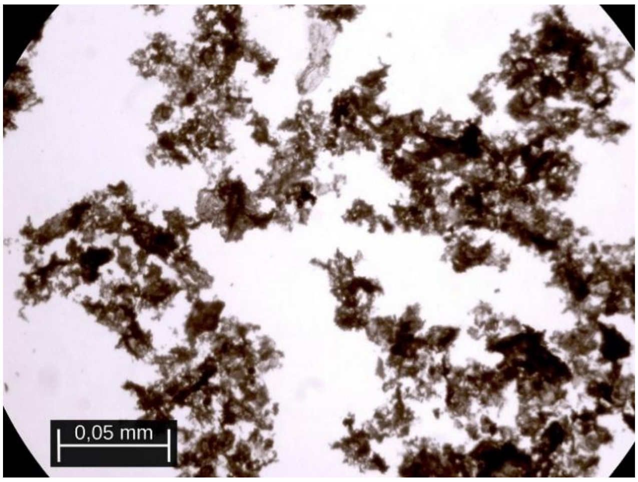
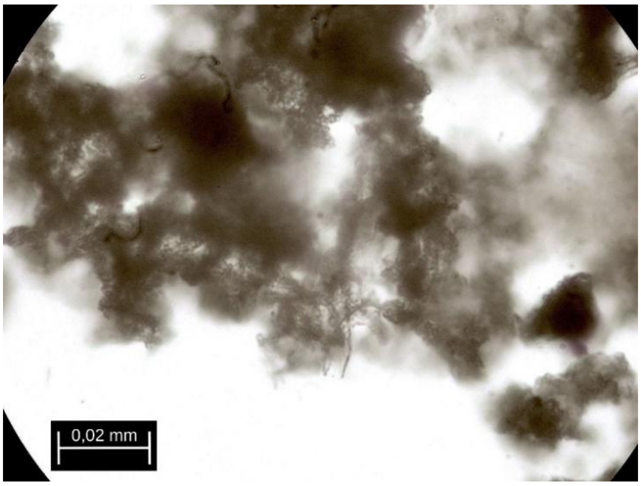


Figure 11. Optical microscopy image of CSBA after burning at 650°C with 50x magnification



The results are in accordance with the expected characteristics for the cement. The particle size distribution of Portland cement in Brazil, governed by the standard (Brazilian Association of Technical Standards (ABNT), 2012), establishes that normal Portland cement should primarily have a maximum particle size of 75 μm. Therefore, the CSBA has a particle size range similar to that of cement, favoring the possibility of substitution and complying with the established standards.

3.2.6. Specific gravity

Based on the standard (Brazilian Association of Technical Standards (ABNT), 2000), it was possible to determine the specific gravity of the cement, calcium hydroxide, and the CSBA burned at

650°C for two hours. Table 4 presents the amount of mass tested and the specific gravity result obtained using the technique employed.

Table 4. Results of the specific gravity test

Material	Tested mass (g)	Displaced volume (cm³)	Specific gravity (g/cm³)
Cement	60.00	20.00	3.00 ± 0,01
Calcium hydroxide	50.00	20.80	2.40 ± 0,05
CSBA1	37.00	20.60	1.80 ± 0,05

The specific gravity of cement is an essential characteristic for producing high-quality concrete and mortar. An adequate density can contribute to achieving concrete with the desired mechanical strengths and mortar with good workability, as well as enabling more accurate calculations for the material proportions in the mix.

The results found for the specific gravity of cement and calcium hydroxide align with the findings in the literature (Hoppe Filho *et al.*, 2017; Medeiros *et al.*, 2015; Pádua, 2012; Paula *et al.*, 2009), which highlights the reliability and reproducibility of the tests performed.

Based on the results, CSBA presented a specific gravity approximately 40% lower than that of Portland cement. This characteristic may influence the density of cementitious composites containing the ash, depending on the replacement level and mixture proportions adopted. However, since no concrete or mortar mixtures were produced in the present study, and no mechanical or density evaluations of cementitious composites were performed, further investigations are necessary to assess the actual effects of CSBA incorporation on the physical and mechanical behavior of cement-based materials.

4. CONCLUSIONS

After drying the bagasse in an oven, the material lost approximately 90% of its mass and presented characteristics visually similar to natural charcoal, indicating potential for use as supplemental fuel in starch drying boilers, partially replacing the firewood currently used.

The drying and burning processes significantly reduced the residue volume, which may contribute to minimizing disposal and storage demands in starch factories, while also enabling energy recovery from the residue.

The combustion product, cassava starch bagasse ash (CSBA), was investigated regarding its physicochemical characteristics and potential applicability in cementitious systems, considering similar approaches reported in the literature for agro-industrial residues.

The FTIR analysis identified characteristic bands mainly associated with Si and Ca, which was consistent with the results obtained by XRD and EDX analyses. In addition to Ca and Si, the EDX results revealed a significant K content in the chemical composition of the ash. The sample burned at 650 °C presented the most pronounced amorphous halo in the XRD analysis, indicating greater amorphous phase formation under this calcination condition.

Although the CSBA presented some chemical similarities to CP-V (HSR) Portland cement, particularly regarding CaO content, the relatively low SiO_2 concentration (4.20%) and the elevated K_2O content suggest limitations that require further investigation before practical application in cementitious composites.

The DSC analysis indicated greater thermal stability of the calcined material compared to the raw residue, while optical microscopy revealed non-agglomerated particles with morphology visually comparable to cement particles.

The results obtained in this study demonstrate the potential of CSBA as an agro-industrial residue of interest for further investigation in cementitious materials. However, additional studies involving pozzolanic activity index, mechanical performance, durability, alkali reactivity, and the incorporation of CSBA in cementitious composites are necessary to evaluate its effective applicability as a supplementary cementitious material.

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