Original Article

Characterization of Brazilian biochar from rice husk for application in cementitious pastes

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Abstract

Biochar is a product derived from biomass pyrolysis and used as a soil conditioner. Researches have evaluated its applicability in civil construction as supplementary cementitious materials. However, the properties of biochar are linked to the properties of its raw material and the processing method, highlighting the necessity of other characterization studies of this material before its application. This study aims to carry out a characterization of the Brazilian biochar, with a view to its application on cementitious pastes. The characterization techniques applied were: granulometric determination by laser diffraction, X-ray fluorescence spectrometry, X-ray diffraction, thermogravimetric analysis and Chapelle method. The morphological structure of the material was evaluated by scanning electron microscopy. The results showed that the use of biochar as a pozzolanic material, i.e, mineral addition is dependent on the physical-chemical properties obtained by the pyrolysis conditions applied, even when amorphous silica is presented.

Keywords: biochar; supplementary cementitious materials; rice husk; pozzolanic material; mineral addition.

1 Introduction

The rice processing generates several residues, the main ones being bran, broken grains (grit), and husk, which corresponds to 20% of the total grain [1,2]. Disposal of rice husks occurs in two ways: in landfills for decomposition or subjected to uncontrolled burning of diverse industrial processes. Both options generate environmental impacts. The first for releasing a high volume of methane, due to the decomposition of the bark being very slow, combined with the fact that it is easily transported by air and the second is by the emission of greenhouse gases [3]. Rice husk has a substantial calorific value and volatile content, which facilitates its use as fuel, thus characterizing it as agricultural biomass [4]. According to the National Electric Energy Agency [5], biomass is understood to be any organic matter that can be transformed into mechanical, thermal, or electrical energy. It can be of forest, agricultural, or waste origin (whether urban or industrial).

Due to the uncontrolled burning process, rice husk ash tends to have low amorphous silica content or even unburned husk particles. However, alternatively, the valorization of rice husks can be done through their conversion into biochar: a bioproduct formed from the pyrolysis process of biomass [6]. Pyrolysis is the process of chemical decomposition of organic materials at high temperatures, in the absence or low concentration of oxygen [7-10]. Appropriate ovens and/or reactors are used in this process, consisting of closed and controlled systems that allow the capture and use of carbonized by-products, involving the minimal release of CO_2 and other gases into the atmosphere [11]. Biomass compounds decompose when subjected to pyrolysis and, due to the absence of oxygen, carbon is retained in their physical structure. Thus, biochar is characterized as a solid material of low density, highly heterogeneous, and basically composed of stable carbonaceous residues and unreacted organic solids [7,10]. Depending on the raw material, biochar can be chemically inert or pozzolanic, having the ability to chemically react with $Ca(OH)_2$ in the presence of water, producing C-S-H gel [12].

The composition of the rice husk consists of a complex mixture, with a high organic content (from 75% to 90%) combined with inorganic components, varying according to factors such as the type of rice, the region of production, harvest, climate, and soil. In theory, it has an ash content of approximately 11%, which are composed of 80% to 90% of SiO_2, in addition to K_2O, P_2O_5, CaO and, in smaller amounts, Fe, Mg and Na [8,13,14]. Thus, biochar derived from the burning of rice husks generally contains amorphous silica theoretically capable of pozzolanic activity.

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Thus, in terms of applicability, biochar from rice husk has a very diversified use, with a large part of the literature focused on the agricultural sector, where biochar is used for soil correction, fertilizer, and carbon sequestration [7,11,15]. However, its applicability is also studied in the construction industry, especially as a partial substitute for cement in the composition of cement matrices [16,17].

Biochar is a carbon-negative material, which has the ability to carbon capture in a stable form, which is adsorbed in its pores due to its affinity for nonpolar compounds and its high surface area [18,19]. A ton of biochar can uptake 2 to 2.6 tons of CO_2 [20]. Cement-based composites can also capture CO_2 through carbonation reactions. Carbon sequestration in cement-based matrices occurs, from CO_2 diffusion into the pore solution, reacting with alkaline compounds converting to calcium carbonate [21,22], according to the reactions shown in the Equations 1-4:

$$Ca\left(OH\right)_2 + CO_2 \rightarrow CaCO_3 + H_2O \tag{1}$$

$$C_3S + \left(3-x\right)CO_2 + yH_2O \rightarrow CxSHy + \left(3-x\right)CaCO_3 \tag{2}$$

$$C_2S + \left(2-x\right)CO_2 + yH_2O \rightarrow CxSHy + \left(2-x\right)CaCO_3 \tag{3}$$

$$C-S-H + 2CO_2 \rightarrow SiO_2 + 2CaCO_3 + H_2O \tag{4}$$

However, the efficiency of carbon sequestration in cement-based materials can be improved, by testing other materials on their composition. Thus, there is another alternative, incorporating biochar as a CO_2 capture and storage technology (CCS) [21] in cement-based materials, enhancing the properties and also turning the material net zero carbon footprint [22-24].

According to [25], there is a beneficial effect associated with the use of biochar in cementitious matrices and its potential for use in CCS: the reduction of cement content, which reduces the environmental impacts arising from the cement industry; the commercial application of biowaste that mitigates problems such as disposal in landfills and open-air burning processes; all this combined with the reduction of the carbon footprint linked to the ability of biochar to capture greenhouse gases [2,10,17,20]. Furthermore, studies indicate that the use of biochar as a substitute for cement or as an additive can induce the micro filler effect by promoting the refinement of the pore structure, accelerating cement hydration by increasing the initial strength of the mixture, reducing permeability, as well as improving thermal properties [15,22,24-27].

The physicochemical properties of biochar, such as carbon content, surface area, degree of pozzolanic activity, size, and distribution of pores are closely linked to the nature of the biomass and the parameters used in its production process [2,28]. Furthermore, the fixation of CO_2 in the biochar structure takes place by chemical adsorption, but, mainly, by physical adsorption. The factors that affect the CO_2 adsorption performance in biochar can be divided into physical properties (surface area, volume, and pore size), chemical properties (surface functional groups, basicity, hydrophobicity, polarity, and aromaticity) and external conditions (pyrolysis temperature, adsorption temperature, and pressure) [21]. So, it is of great importance to understand the functional groups presented as well as the determination of the specific surface area because they provide active sites for the adsorption of CO_2. Also, according to some authors [24], the mechanical strength of cementitious mortars can be affected by the ash content of biochar; a higher ash content presented by biochar from date palm leaves (BioCl) gave rise to a higher compressive strength than biochar from date palm seeds (BioCs), even though both showed higher resistances than the reference mortar. However, according to other authors [28], better mechanical performance is usually accompanied by reduced workability, with the beneficial effects strongly dependent on the microstructure and chemical composition of the biochar particles. In this sense, fine particles of biochar act as a micro aggregate, filling the voids and densifying the matrix, while promoting additional hydration, especially in severe curing conditions, since biochar is capable of absorbing water from mixing and releasing it at later ages, making the internal structure denser and increasing its resistance [28]. Thus, it is assumed that the internal healing capacity of biochar is derived from its porous nature. Scanning electron microscopy (SEM) images indicate that after pyrolysis, several types of pores are formed in biochar, which provides space not only for storing water, but for hydration products deposition and, consequently, more reagents for the carbonation reactions with CO_2 [29].

Regarding morphology, as observed by [24], the shape of biochar particles affects the mechanical strength of concrete. Corroborating what was presented by [30], the authors observed that the rounded particles could densify the mortar leading to a greater load capacity. However, sharp particles do not favor packing, resulting in high crack propagation and, consequently, lower load capacity. On the other hand, the addition of biochar with coarser particles (200 – 300 μm) reduces strength and impairs rheology, leading to a loss of workability. Thus, the composition with finer particles (50 – 80 μm), improves the packing density and favors the rheological properties of biochar [29].

Regarding external conditions, the carbonization temperature is one of the most influential factors in the properties of biochar. With the loss of volatile compounds and formation of compounds with high carbon content, there is the formation of macro, meso, and micropores, which contribute to increasing the specific surface area of biochar. However, when this process occurs at temperatures above 1000 °C, there is the degradation of aromatic compounds and consequent loss of carbon and desirable physical attributes, such as porosity [11]. Furthermore, with regard to the pyrolysis process, prolonged pyrolysis of rice husks for a period longer than 30 minutes resulted in high ash content and formation of a superficial layer due to potassium oxidation, which can reduce the pozzolanic activity of the material [6].

However, other authors [31], showed that the biochar yield and the volatile contents gradually decreased as the pyrolysis temperature increased. According to the authors, biochar derived from rice (whether from the husk or the straw) has a high ash content in all temperature ranges.

So, Concerning the understanding of the behavior of the material and developing its future applicability, it is important to study the physical and chemical properties of the biochar, which will contribute to understanding the relationships between microstructure and the resultant properties expected [32]. Thus, considering the use of biochar in cement-based matrices, characterization studies are necessary. Therefore, this work aims to carry out the physical-chemical, mineralogical, and morphological characterization of biochar from the pyrolysis of rice husks, obtained under specific conditions, in order to predict its possible application in cementitious matrices.

1.1 Materials and experimental program

For the development of the study, biochar was obtained through pyrolysis of rice husks, a residue provided by an industry active in the South and Southeast regions of Brazil, by the company SP Pesquisa e Tecnologia, under a burning temperature of 400 °C for 15 minutes.

Chemical analysis of biochar was performed using X-ray fluorescence spectrometry (FRX). For this purpose, the sample was melted using 0.8g of the material. The equipment used was the model ARX Performer'X 4200 W spectrometer and the software for data analysis was Thermo Scientific OXSA.

The granulometric determination was carried out through the laser diffraction granulometry test, with dispersion in water. A Cilas model 940L laser granulometer was used, equipped with a laser emitter and an ultrasound device. X-ray diffraction (XRD) was also performed using a Bruker diffractometer, model D8 Advance, under a voltage of 40 kV and amperage of 40 mA, in theta/theta system with 0.02 theta/s. The energy source was a tungsten filament (cathode), and the X-ray tube is copper (anode). Data acquisitions were performed on a computer, coupled to the diffractometer, using DiffracPlus software. For data interpretation, Diffrac.EVA software, version 2018, was used.

Fineness was determined by BET specific surface area using nitrogen gas adsorption. For that, a Quantachrome brand analyzer, model autosorb iQ-MP/XR, was used. Degassing took place under a vacuum of 3.75x10-10 mmHg, at 110 °C for one hour; after heat treatment, the samples were cooled to room temperature and weighed. Finally, they were transferred again to the sample holder and submerged in liquid nitrogen, at a temperature of -296 °C, to capture the experimental points. Data collection took place using the ASiQwin software.

The specific mass was determined through the test described by the NBR 16605 standard [33], using the Le Chatelier volumetric flask; the determination of loss on fire was determined according to NBR NM 18 test [34]; and the amount of unburned organic matter was obtained according to the test for determination of insoluble residue, recommended by NBR NM 15 [35]. Pozzolanic activity was verified with lime at seven days of age, according to the test recommended by NBR 5751 [36], pozzolanic activity with Portland cement at 28 days, according to NBR 5752 [37] and through of the direct method, called modified Chapelle, according to NBR 15895 [38], which corresponds to the calcium hydroxide content fixed by the addition.

Furthermore, thermogravimetric analysis (TG/DTG) was carried out using Netzsch STA 449 F3 Nevio equipment. The initial mass of the sample was 21 mg, placed in an open 85 μL alumina crucible. The measurements were carried out in the temperature range of 30 °C to 1100 °C, with a heating rate of 10 °C/min, in an atmosphere of nitrogen gas under a flow rate of 100 mL/min. Finally, the TG/DTG results were analyzed using Originlab to obtain the characteristic curves. Finally, to understand the morphological characteristics of biochar, its particles were characterized by scanning electron microscopy (SEM) using a JEOL microscope, model JSM-6610, equipped with EDS, Thermo scientific NSS Spectral Imaging.

2 Results

Figure 1 shows the granulometric curve obtained. It is particle diameter size (mm) versus accumulated percentage - Cum (%) and percentage of particles in each size range - Diff (%).

It is observed that the granulometry of the material varies from 0.5 μm to 110 μm, presenting an average particle size of d50=26.09 μm and d90=75.62 μm. In this sense, the material has a fineness compatible with that of Portland cement, since it has less than 12% of residue in the #200 sieve (75 μm) as established by Brazilian standard NBR 16697 [39]. However, as shown in Table 1, it can be seen that the biochar did not meet the requirement of the standard, since it presents around 27% of material retained in the #325 sieve (45 μm), while the limit established in the standard is less than 20%.

The effect of biochar particle size on hydration kinetics can be evaluated by investigating whether fine

Table 1. Chemical and physical characterization of biochar

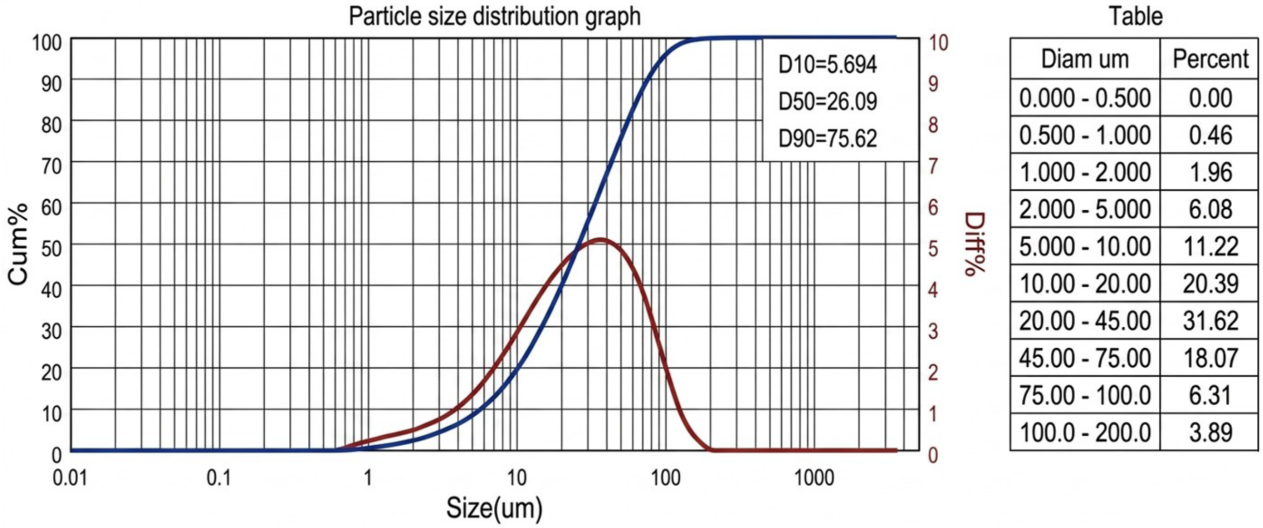
Properties		Result	Requirement according to NBR 12653 (ABNT, 2014) [40]
Density (g/cm^3)		1.63	N.S.[a]
Fineness	Residue in the sieve 325 (%)	27	< 20%
	BET specific surface area ($\text{m}^2\text{/g}$)	7.61	N.S.

(a) N.S. = Not specified. (b) Limit established by Raverdy et al. [41]. *Performed in accordance with Brazilian standard ABNT NBR 15895 [38] in powdered pozzolans.

Table 1. Continued...

Properties		Result	Requirement according to NBR 12653 (ABNT, 2014) [40]
Chemical composition (%)	Loss on ignition (%)	60.37	≤ 6.00
	Insoluble residue	54.04	N.S.
	(SiO_2)	36.71	N.S.
	(Fe_2O_3)	0.18	N.S.
	(MgO)	0.23	N.S.
	(SO_3)	0.00	≤ 5.00
	(CaO)	0.41	N.S.
	(Na_2O)	0.44	N.S.
	(K_2O)	0.88	N.S.
	Alkaline equivalent	0.97	≤ 1.50
	(P_2O_5)	0.3	N.S.
	(MoO_3)	0.13	N.S.
	(MnO)	0.11	N.S.
$SiO_2 + Al_2O_3 + Fe_2O_3$ (%)		36.89	≥ 50.00
Index of pozzolanic activity with lime at the age of 7 days (MPa)		3.2	≥ 6.00
Pozzolanic activity index of with cement at the age of 28 days		90	$\geq 90\%$
Modified Chapelle Test*		477.1	$\geq 436^{(b)}$

(a) N.S. = Not specified. (b) Limit established by Raverdy et al. [41]. *Performed in accordance with Brazilian standard ABNT NBR 15895 [38] in powdered pozzolans.



Diam um	Percent
0.000 - 0.500	0.00
0.500 - 1.000	0.46
1.000 - 2.000	1.96
2.000 - 5.000	6.08
5.000 - 10.00	11.22
10.00 - 20.00	20.39
20.00 - 45.00	31.62
45.00 - 75.00	18.07
75.00 - 100.0	6.31
100.0 - 200.0	3.89

Figure 1. Biochar granulometric curve.

biochar particles significantly improve mortar properties compared to relatively small grains, to determine the need for additional grinding [42]. However, as the authors point out, the milling process is energy intensive, which may lead to a non-compensation of the beneficial effect of reducing the carbon footprint due to biochar application. In addition, although it provides an increase in the specific surface area, grinding will reduce the macroporosity of the biochar, reducing its effectiveness in the internal healing of its particles [43].

In view of the origin of rice husk biochar, it is accepted to classify the material as class E, which consists of pozzolans not included in classes N and C, according to the physicochemical requirements established by NBR 12653 [40] for determination of pozzolanic materials. Thus, the main physicochemical properties of biochar are summarized in Table 1.

With regard to the composition of biochar, there is a predominance of SiO_2, justified by the nature of its biomass, in addition to a high loss on ignition, which can be explained to the organic origin of the material, which has a high content of incorporated carbon, as well as the presence of volatiles not eliminated during pyrolysis at 400 °C. Thus, there is an indicator of lower reactivity of the material. Furthermore, it is observed that the chemical composition of biochar presents SiO_2, Al_2O_3 and Fe_2O_3 contents lower than recommended by the norm. The low silica content and the absence of aluminates indicate that the material does not have the potential to react and consume calcium hydroxide, therefore not forming compounds such as hydrated calcium silicate, hydrated calcium silicoaluminate or hydrated calcium aluminate. However, the pozzolanic potential of a

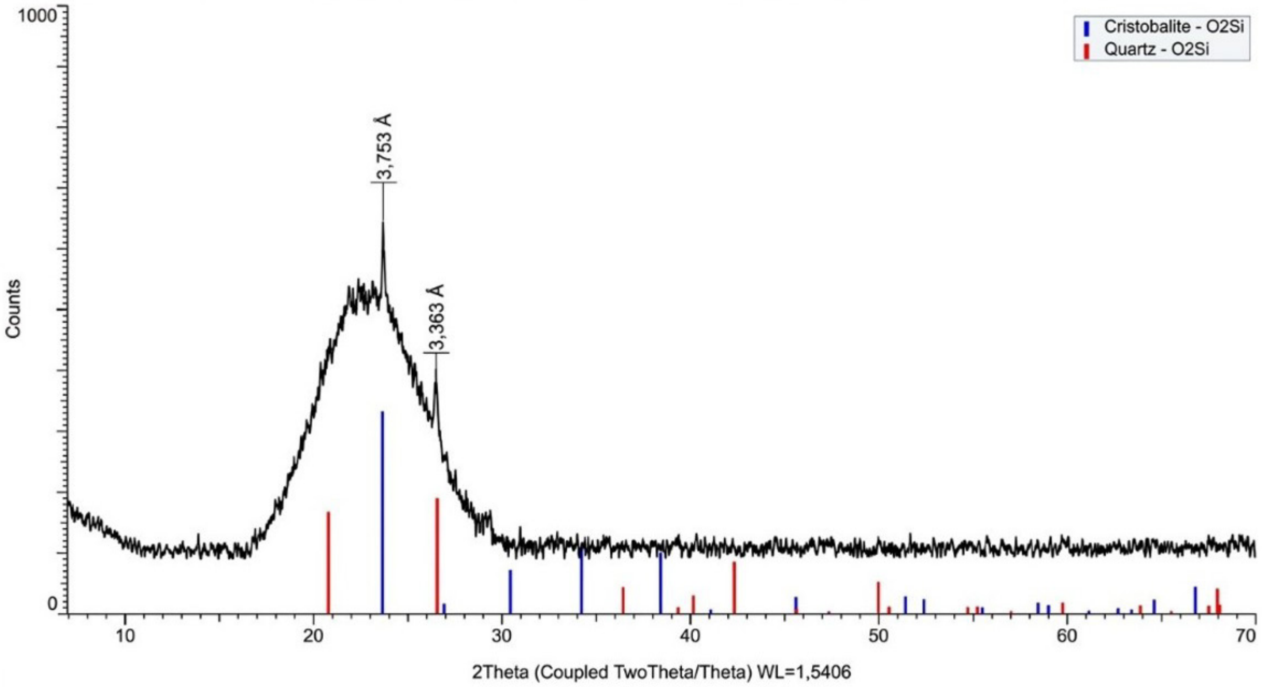


Figure 2. X-ray diffractogram of biochar

material is linked not only to the silica content but mainly to the degree of amorphism of its structure. Thus, Figure 2 shows the X-ray diffraction spectrum of biochar, in which it is possible to assess the crystallinity level of the material. The X-ray diffractogram indicates the amorphism of the biochar structure and confirms that it is a mostly siliceous material.

Thus, the diffractogram obtained shows crystalline peaks related to cristobalite and quartz, polymorphic forms of silicon dioxide (SiO_2), in addition to a diffuse band from the amorphous fraction of the material. This configuration is narrated in the literature. For comparison purposes, the XRD of rice husk ash, which has 89.89% amorphous silica, shows crystalline silica peaks around 2θ=22 [17,44], in turn, already reported in their study that silica crystallinity can be qualitatively evaluated by comparing the peaks around 2θ = 20º - 30º. Thus, the broader rice husk ash peak around 2θ = 22º indicates the presence of amorphous silica. Likewise, peaks at 2θ = 26.26º over the amorphous background confirm the presence of quartz.

Therefore, it can be assumed that rice husk biochar is susceptible to pozzolanic action. For this purpose, the pozzolanicity can be predicted through mechanical tests with lime mortars at 7 days and Portland cement at 28 days. In this sense, the results of compressive strength at 7 days were reported as an average of three samples, none of which presented individual resistance that met the recommendations of the standard. The average resistance, of 3.1 MPa, is below the minimum required by the Brazilian standard, namely, 6 MPa. However, in a complementary way, from the pozzolanic activity test with Portland cement at 28 days, a performance index equal to 90% was obtained, equivalent to the required limit. With regard to the modified Chapelle method, a pozzolanic activity index equal to 477 mg of $Ca(OH)_2$/g of the sample was obtained. [41] establishes the minimum limit of 436 mg of $Ca(OH)_2$/g to classify an addition as a pozzolanic material, allowing us to state, therefore, that biochar presents, according to these two last methods, relative pozzolanic performance.

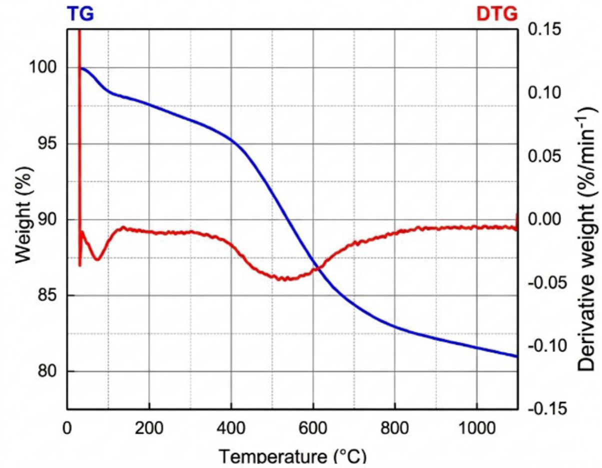


Figure 3. Biochar TG/DTG curves

Figure 3 shows the TG/DTG curves obtained in the thermogravimetric analysis of biochar.

It is possible to observe that the biochar sample loses 19.02% of mass after heating up to 1100 °C. The DTG curve shows two distinct regions, between 0 and 150 °C and between 400 ºC and 700 °C, where the sample loses mass. Initially, according to [45], biochar loses adsorbed water,

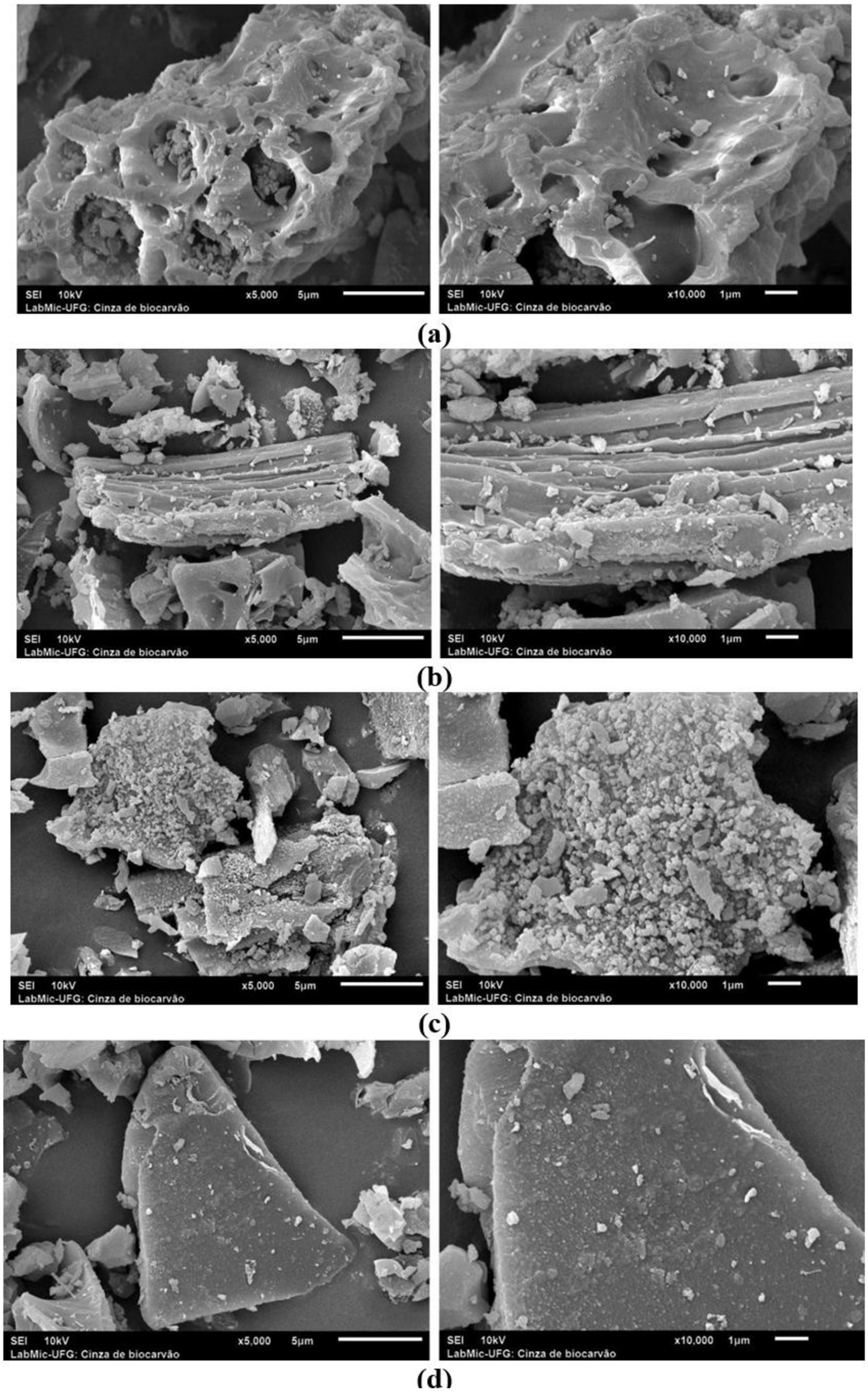


Figure 4. Biochar SEM.

which is released between 35 °C and 200 °C. Between 400 °C and 950 °C, the decomposition of organic matter has been predicted, such as hemicellulose, which decomposes between 35 °C and 350 °C, cellulose between 370 °C and 430 °C, lignin and volatile compounds between 600 °C and 950 °C.

The structure and morphology of the biochar is shown in Figure 4.

It can be observed in the micrographs, morphological variations in the sample, according to parts (a), (b), (c) and (d) of the figure. In 4(a) micropores formed on the surface of the particles during pyrolysis are observed, with diameters around 1-4 µm, approximately. In 4 (b) there is also an irregular surface, however, with fibrous morphology and length around 5 µm, showing granular formation on the surface. In 4(c) irregular particles in the form of granules are observed, in this case not presenting micropores on the surface. Finally, in 4(d) we have a particle with an angular, flattened structure and a smooth surface.

Pores in biochar are created due to the release of volatiles and organic matter during pyrolysis. In this sense, the presence of angular particles portrays the occurrence of incomplete ignition of the material, which compromises the performance as a pozzolanic addition [46]. Finally, uneven surfaces increase roughness, which can improve the transition zone between the biochar and the cementitious matrix [47]

3 Global discussion

From the chemical point of view, the composition of the rice-husk biochar characterized in this study ($SiO_2 \approx$ 36.71%, $Fe_2O_3 \approx$ 0.18%, MgO ≈ 0.23%, SO_3 = 0%, CaO ≈ 0.41%) differs markedly from those of well-established pozzolanic additions such as silica fume, metakaolin and fly ash. For comparison, silica fume typically presents SiO_2 contents in the range of 85–98%, with only minor amounts of Al_2O_3, Fe_2O_3, CaO and MgO [48]. Metakaolin exhibits roughly 50–55% SiO_2 and 40–45% Al_2O_3, with Fe_2O_3, CaO, MgO and other oxides present only in small quantities. Typical Class-F fly ash is richer in silica and alumina, with SiO_2 often between 50–65% and Al_2O_3 in the order of ~20–30%, along with appreciable Fe_2O_3 (e.g. 5–10%) and variable CaO and MgO depending on coal source [49].

Thus, the rice-husk biochar should be considered a mostly siliceous material but with a significantly lower silica content than silica fume or metakaolin, and with a minimal presence of alumina, iron or calcium phases. Moreover, the high observed loss on ignition and presence of insoluble residues indicate that the biochar still contains non-crystalline carbonaceous and mineral remnants, which further distinguishes it from the conventional pozzolanic materials.

From a physical point of view, it presents granulometry compatible with that of cement, but with intermediate fineness among other pozzolanic mineral additions. Namely, it is a finer material than fly ash and less fine than silica fume and metakaolin. Furthermore, it is a light material, with a specific mass lower than all the aforementioned pozzolanic admixtures.

Concerning the reactivity of the biochar, the XRD indicated amorphism susceptible to pozzolanic activity, which was evident from the modified Chapelle test and pozzolanic activity with cement, which presented satisfactory results, indicating some chemical potential of pozzolanic mineral addition.

The calcination process influences the morphology of the material, and this becomes evident from the SEM, when identifying a variable structure, with porous regions mixed with regions of regular surfaces. This undesired configuration can be reversed through an adequate pyrolysis process, promoting the obtainment of a porous biochar with a larger surface area.

In practice, these results indicate that the rice husk biochar studied in this research has potential for application in cementitious pastes (matrices) as a pozzolanic addition. Even though it showed low performance, justified by a possible inadequate processing, better results can be obtained through a suitable processing, especially with regard to the pyrolysis of rice husk.

4 Conclusions

According to the study, it is possible to conclude:

The rice husk biochar characterized in this study has pozzolanic potential for application in cementitious pastes (or matrices) mainly due to the amorphism of its structure and some physical characteristics of the material, which can be improved from a processing directed to that purpose.

The structure of biochar is predominantly amorphous, with some crystallinity. Thus, even though it has a low alumina and iron content in its composition, the presence of amorphous silica available for the occurrence of pozzolanic reactions is observed, which is evident in view of the determinations of the pozzolanic activity index (PAI).

Although the early-age PAI (with lime) at seven days indicated a low reactivity in young pastes — the 28-day PAI (with cement), combined with the results of the Modified Chapelle test, reveals meaningful pozzolanic activity of the rice-husk biochar when incorporated in cementitious pastes. This demonstrates that the biochar is capable of consuming the portlandite of the hydration of Portland cement, thereby producing additional binding phases such as calcium-silicate hydrates (C–S–H). Such behavior is a indicator of its suitability as a supplementary cementitious material (SCM).

In practical terms, this means that partial replacement of cement by rice-husk biochar could contribute to a more sustainable construction system by reducing clinker usage, lowering CO_2 emissions, and valorizing an agricultural waste.

For practical production of rice-husk biochar destined to serve as a supplementary cementitious material, it is crucial to adopt a controlled pyrolysis protocol. Based on literature data [50], an optimal pyrolysis window around 500–550 °C with a residence time of approximately 20–30 minutes and a moderate heating rate of 50 °C/min often yields a biochar with favorable carbon stability, adequate surface area and low residual volatiles — characteristics that enhance pozzolanic reactivity. Under such conditions, the resulting biochar will minimize unburned organic matter, by reducing loss on ignition and insoluble residues and maximize the availability of reactive silica or siliceous ash.

Therefore, a programmed pyrolysis by specifying heating rate, target temperature and residence time, rather than an uncontrolled heat treatment, tend to optimize the biochar's potential as a pozzolanic mineral addition.

Thus, it is expected that from a planned and controlled pyrolytic process, biochar will be obtained with a continuous morphology, characterized by the formation of micropores, with greater specific surface area, lower content of organic matter and, consequently, with properties that enhance its use as a pozzolanic addition in cementitious pastes or matrices.

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