

Research Article

Sugarcane Bagasse Ash Micronized Using Air Jet Mills for Green Pozzolan in Brazil

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This study provided a basis for new possibilities concerning the use of the sugarcane bagasse ash as a green pozzolanic addition to the Portland cement composite. To that effect, a simple micronization method using air jet milling without any other additional thermal procedure was used to control the characteristics of ash particles. This procedure not only maintains the required characteristics of the residues but can also improve some of them. Sugarcane bagasse ash is a residue produced on large scale in Brazil by ethanol and sugar plants as a result of the burning of sugarcane bagasse in energy cogeneration. The residue used in this study was initially characterized by scanning electron microscopy, granulometric and specific mass analyses, N₂ adsorption measurements, X-ray diffraction, X-ray fluorescence spectroscopy, and thermogravimetric analysis with differential thermal analysis. Pozzolan ash activity was evaluated according to the axial compressive strength at 28 days and the modified Chapelle methods. The results showed that the milling fly sugarcane bagasse ash samples presented satisfactory pozzolanic activity.

1. Introduction

The prospect of an increase in Portland cement consumption indicates that by 2025 the release of CO₂ from cement production processes will be as much as 3.5 billion tons per year worldwide. Shi et al. [1] argue that this projection equates to all CO₂ production from industrial activities in Europe at the beginning of the current decade. According to Mehta and Monteiro [2], the annual generation of at least 1.5 billion tons of CO₂ resulting from Portland cement manufacture accounts for approximately 7% of global emissions. Furthermore, the cement industry is the second largest consumer of industrial energy, accounting for 7% of the consumption [3].

On the other hand, a joint report issued by the International Energy Agency (IEA) and the Cement

Sustainability Initiative (CSI) states that the implementation of changes, along with the development of processes and the support of public policies, will allow for a 24% reduction in the levels of CO₂ generated as a result of cement production by 2050 [3]. According to Mehta [4], one of the main contributions to scale down energy consumption and the greenhouse effect is the reduction of cement production processes from raw clinker lumps.

The development of green pozzolanic binders to partially replace Portland cement is a valuable socioenvironmental option. As reported by Yu et al. [5], 80% of fly ashes were successfully used in Portland cement production. In this study, the authors emphasized the valuable reduction in cost, energy consumption, and CO₂ emissions when compared to conventional concretes. Loh et al. [6] emphasize the versatile use of sugarcane bagasse and its by-products as a green raw

material in several composites. According to them, the low pollutant index is one of the highlights of the use of green materials to develop new solutions. Among the new options of green pozzolanic addition under development, the milling fly sugarcane bagasse ash (MFSCBA) stands out, as reported by Soares et al. [7], Cordeiro et al. [8], Delalibera et al. [9], and Bahurudeen and Santhanam [10].

The sugarcane bagasse ash (SCBA) is a residue resulting from the burning of sugarcane bagasse in boilers in power cogeneration processes in sugar plants. The term SCBA represents all ashes generated, that is, the fly sugarcane bagasse ash (FSCBA) from gas washing and the bottom sugarcane bagasse ash (BSCBA) from the burning of the boiler's bottom. FSCBA milling results in MFSCBA. The current processing technology allows all the energy required to operate plant processes (steam, mechanical, and electrical) to be supplied by this cogeneration system, which may lead to energy self-sufficiency in plants [11, 12]. Furthermore, in some cases, plants partner up with power distribution companies to even provide electric power to consumer units connected to the power grid. All these factors put together stress the valuable contribution of the reuse of by-products resulting from the burning of bagasse in electric power production.

In addition, Fairbairn et al. [13] showed that the use of MFSCBA as a pozzolanic material would reduce CO₂ emissions along the cement production chain, enabling the issue of the certified emission reduction (CER) credits. In the referred study, the authors highlighted the possibility of reducing 519.3 thousand tons of CO₂ yearly, given the potential of SCBA generation in the region studied, which is responsible for 60% of the sugarcane production in Brazil.

Based on the data provided by Hojo [14], it is possible to estimate that 11% of the ashes generated in boilers are FSCBA-type resulting from gas and suspended particulate matter washing, whereas 89% of the ashes are BSCBA-type resulting from removing particulates from the bottom of boilers.

Brazil is considered to be the largest sugarcane producer in the world and the main exporter of its derivatives, namely, ethanol and sugar. According to the data from Fiesp/Ciesp [15], in general, 0.260 tons of sugarcane bagasse is generated for each ton of sugarcane processed, producing 6.2 kg of ashes. According to Conab [16], the Brazilian sugarcane production for the 2018/2019 harvest is estimated to be around 615 million tons. These results show that Brazil can potentially produce up to 3.813 million tons of SCBA (FSCBA + BSCBA) in the referred harvest. SCBA is currently mainly disposed of directly in farmlands, with no effective value being added to the soil and pushing up logistics costs which make sugar plant outputs more expensive.

For FSCBA to be ready to be used as pozzolana, a number of prerequisites provided by Brazilian standard NBR 12653 [17] and international standard ASTM C618-17a [18] must be met, as well as prerequisites related to the amorphous structural organization [7, 8, 10, 19, 20]. Ash milling, bagasse burning control, or even ash burning control can be mentioned as the most commonly used preparation methods. In this situation, it is known that

particle size and shape have a significant effect on the viability of Portland cement-based materials [21].

In this sense, FSCBA micronization by air jet mills is a relevant technique that allows microstructural alterations. This application has been used efficiently in other industrial segments, such as mineral milling, pharmaceuticals, and the food industry [22]. Micronization parameterization performed in the laboratory has a simplified set-up, allowing for large-scale production in industrial air jet mills.

The ash was prepared without using any additional burning processes, with thermal control in the laboratory. This study assessed the efficacy of the innovative micronization process of fly sugarcane bagasse ash using air jet mills to obtain and prepare pozzolanicity prerequisites. The performance of pozzolanic materials of milling fly sugarcane bagasse ash was also analyzed using the compressive strength method as well as the chemical reactivity analysis method.

2. Materials and Methods

2.1. Materials. The characterized materials later used in the development of the pozzolanic analyses were as follows:

- (1) Cement CP II F 32 as per Brazilian standards NBR 7215 [23] and NBR 11578 [24]. Cement chemical composition was as follows: 66.1% CaO; 15.5% SiO₂; 4.94% MgO; 4.1% SO₃; 3.72% Al₂O₃; 3.13% Fe₂O₃; 1.1% K₂O; 0.369% Na₂O; 0.272% TiO₂; 0.226% SrO; 0.131% P₂O₅; 0.0802% MnO; 0.0058% ZnO; and 0.3151% of other compounds. The content of carbonate materials was between 6% and 10%. Insoluble residues represented less than 2.5%. The initial setting time was 1 h. The specific area was larger than 260 m²/kg.
- (2) Brazilian standard sand was provided by the Technological Research Institute (IPT-SP), according to the specifications provided by Brazilian standard NBR 7214 [25]. Four standard fractions (retained on the sieve) were used: coarse (1.2 mm to 2.4 mm), medium coarse (0.6 mm to 1.2 mm), medium fine (0.3 mm to 0.6 mm), and fine (0.15 mm to 0.3 mm).
- (3) Glenium 51 was obtained from the manufacturer BASF. It is classified as a third-generation polycarboxylic ether-based chemical additive with total water solubility. The additive is white and cloudy in appearance and was used in its liquid state, with a solid content of 28.5% to 31.5%, pH between 5 and 7, viscosity of <150 (cps), and density of 1.067 g/cm³ to 1.107 g/cm³.
- (4) The water used was in compliance with Brazilian standard NBR 15900-1 [26].
- (5) The fly ashes were collected in a plant located in the city of Paranacity, Brazil (latitude 22°55'48" S/longitude 52°09'04" W). The type of sugarcane used was RB867515, and sandy soil (Caiuá sandstone) is predominant in the land used to cultivate this plant in the region where the sugar plant operates (30 km

radius). Mechanized harvesting accounts for over 95% of all the sugarcane harvested. Similarly to Hojo's study [14], fly ashes represent 11% of the ashes generated in the sugar plant.

2.2. FSCBA Preparation and Characterization Methods. The fly sugarcane bagasse ash generated from the burning of sugarcane bagasse in the boiler, at temperatures between 500°C and 800°C, only underwent sieving separation, chamber drying, and air jet milling. It is important to stress that the FSCBA was collected separately in dedicated intermediate tanks at the exit of the gas and particulate washing process, that is, prior to being mixed to the BSCBA in the settling tanks. Altogether, four 5 kg fractions of wet material (approximately 10% water content) were collected in four consecutive days. The fractions were manually mixed to make up the sample to be analyzed.

FSCBA raw-state samples were chamber-dried at $100 \pm 5^\circ\text{C}$ for 24 hours. Subsequently, the samples were manually sieved (2 mm-mesh) to remove larger impurities, such as the remaining organic straws resulting from the incomplete burning. This initial procedure generated the primary type of FSCBA.

After sieving, the ash was milled and homogenized by air jet milling to generate the MFSCBA samples. Particle micronization consisted in particle collisions that took place inside a steel-resistant chamber, which occurred after compressed air was applied at up to 500 m/s for 10 min. The air jet milling process was performed in laboratory-scale equipment, with an air pressure injection of 0.8 MPa, output particle size of 1 to 45 μm , and production capacity of 500 g to 5000 g of material milled per hour. The first stage in the air jet milling process created suction to aspirate light materials within the chamber which removed lighter organic matter exposed on the upper part of the sample. With the process used, ash conversion efficiency was 95%. Figure 1 shows a scheme of the micronization equipment operation. The second stage of the procedure was intended to achieve the necessary size reduction to enable pozzolanic investigations.

The methods used in FSCBA and MFSCBA characterizations were as follows:

- (1) Scanning electron microscopy (SEM): generation of visual aspects and textural indication. The samples were fixed on to the surface of a double-faced carbon adhesive tape and coated with a thin gold layer. The analyses were performed in a FEI microscope, Quanta 250 model.
- (2) Granulometric analysis: evaluation of the granulometric classes by sieving with sedimentation analysis as per NM 248 [27].
- (3) Granulometric analysis with laser refraction analyzer.
- (4) Specific mass: evaluated as per the NM 52 [28].
- (5) X-ray diffraction (XRD): structural characterization of the samples and identification of the presence or absence of secondary phases. The equipment used was a Shimadzu, XRD 6000 model with a Cuka

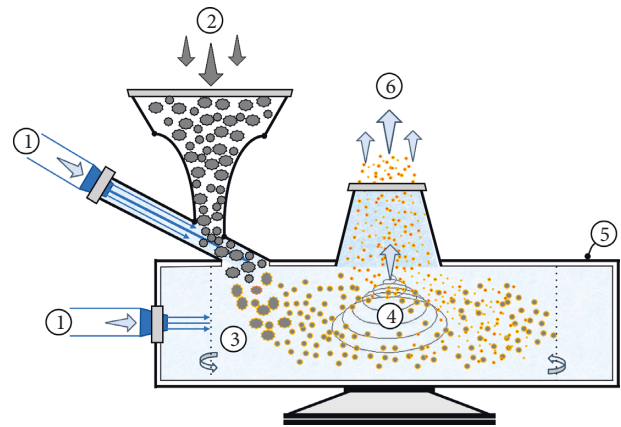


FIGURE 1: Schematic illustration of the air jet milling process. 1, compressed air (feeding); 2, feed funnel; 3, compressed air (milling); 4, milling chamber; 5, steel structure of air jet mills; 6, micronized product (outlet).

source with 40 kV and 30 mA, scan speed of 1°min^{-1} , and range of $4^\circ \leq 2\theta \leq 90^\circ$.

- (6) X-ray fluorescence spectroscopy (XRF): chemical compounds quantification. The equipment used was a Rigaku, ZSX mini II model.
- (7) Thermogravimetric analysis with differential thermal analysis (TGA/DTA): analysis of the mass variation along the temperature exposure and phase transition behavior. The equipment used was a Netzsch, STA 409 PG/4/G Luxx model.

2.3. Pozzolanic Investigation Methods. MFSCBA preparations followed the recommendations provided in Brazilian standard NBR 12653 [17] and international standard ASTM C618-17a [18]. Mortar preparations, the flow value of the control mortar test, the chemical reactivity test for MFSCBA, and the compressive strength analysis followed the guidelines provided by Brazilian standards NBR 5752 [29] and NBR 15895 [30], which directly correlated to international standards ASTM C109/C109M-12 [31], ASTM C1437-7 [32], and ASTM C311-11b [33]. In addition, publications by authors who have addressed the theme were also considered [7, 8, 10, 19, 20, 34].

The percentages of alkalis (Na_2O and K_2O) were calculated based on the NM 25 [35] standard. The pozzolanic studies were conducted using two methods: the first one evaluated the pozzolanic performance of the SCBA with Portland cement by analyzing the axial compressive strength at 28 days, by means of the IDP28 indicator described in the NBR 5752 [29] standard, replacing 25% of the cement mass for SCBA mass. The second pozzolanic analysis method, described in the NBR 15895 [30] standard and referred to as the modified Chapelle method, was used to analyze the chemical reactivity of SCBA with lime and was quantified by means of the $I_{\text{Ca}(\text{OH})_2}$ indicator.

2.4. Determining the Pozzolanic Performance Index with Portland Cement at 28 Days. In general, the NBR 12653 [17]

standard determines that to be classified as a pozzolanic material, the Pozzolanic Activity Index of the material studied with the Portland cement (IDP28) should be at least 90%. This method evaluates the axial compressive strength at 28 days of specimens in which 25% of the cement mass is substituted for the mass of the studied material. As described in the NBR 5752 [29] standard, the mean axial compressive strength of CP II-F-32 cement mortar was evaluated on day 7 for six cylindrical specimens (CSs) (50 mm × 100 mm). Thus, two mortar compositions were prepared (in duplicate): one reference sample without MFSCBA (Mref), and another with MFSCBA (Mmf). For each sample class, six cylindrical specimens (50 mm × 100 mm) were prepared to rupture at 28 days by axial compressive strength in a 100-ton capacity hydraulic press. The materials used in mortar compositions were cement (624 ± 0.4 g), sand (1872 ± 0.4 g), and water (300 ± 0.2 g). As for the Mmf mortar, 25% of the cement mass was replaced for an equivalent mass of MFSCBA, in addition to occasional workability-related corrections made with the use of superplasticizer. These doses represent a water/cement factor of 0.48 and a sand/cement factor of 3.

The following equipment was used in mortar preparation: a 1000 ± 0.05 g analytical balance, an automated mortar mixer operating at low speed at 140 ± 5 rpm and high speed at 285 ± 10 rpm, and a 5-liter flow table used in the consistency index test. Mortar mixtures were performed according to the NBR 7215 [23] standard. The Mmf consistency index (CI) was accepted with a ±10 mm variation in relation to the Mref CI.

For the axial compressive strength results to be accepted in IDP28 calculations, the existence of patterns between the means of both replicate batches is expected. Thus, Student's *t*-test for the mean strength of both samples was performed. The central hypothesis assumed in this test was that the difference of the means of each sample is zero, i.e., $h_0 = 0$. To that effect, the existence of equivalent variances with $2n - 2$ freedom degrees and 95% reliability level [36] were considered. Equation (1) was used to calculate the pozzolanic coefficient.

$$\text{IDP28} = \frac{f_{c_{\text{Mmf}}}}{f_{c_{\text{Mref}}}} \times 100, \quad (1)$$

where IDP28 is the performance index percentage of the studied material with the Portland cement at 28 days; $f_{c_{\text{Mmf}}}$ is the compressive strength mean of Mmf specimens at 28 days, in MPa; and $f_{c_{\text{Mref}}}$ is the compressive strength mean of Mref specimens at 28 days, in MPa.

Finally, a complementary visual microstructural evaluation in areas with cement paste crystallizations was performed by scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDS) at 28 days. The points in the EDS reading came from fragments no larger than 8 mm mainly located in the central area of the ruptured specimens. The samples were metalized with gold ink on a carbon tape.

2.5. Pozzolanic Activity Index Using the Modified Chapelle Method. The method described in the NBR 15895 [30] standard is an adaptation of Raverdy et al. [37]. Generally, the test used to conduct a pozzolanic activity analysis

determines the content of calcium hydroxide ($\text{Ca}(\text{OH})_2$) produced, in milligrams (mg) per gram (g), by the studied material (supposedly pozzolanic).

Equation (2) presents the Chapelle theoretical model for the pozzolanic activity index recommended in the NBR 15895 [30] standard.

$$I_{\text{Ca}(\text{OH})_2} = \frac{28 \times (V_3 - V_2) \times F_c}{m_2} \times 1.32, \quad (2)$$

where $I_{\text{Ca}(\text{OH})_2}$ is the Chapelle pozzolanic activity index, expressed according to the $\text{Ca}(\text{OH})_2$ content of the material studied, in milligrams per gram; m_2 is the mass of the studied material, in grams; V_2 is the HCl 0.1 M volume, in milliliters; V_3 is the HCl 0.1 M volume in blank solution, in milliliters; F_c is the HCl correction factor for a concentration of 0.1 M; and 1.32 is the molecular reaction $\text{Ca}(\text{OH})_2$ per CaO.

According to Raverdy et al. [37], the minimum consumption value of 330 mg of CaO per gram of the evaluated material is expected, and stoichiometry results determined that this value accounts for 436 mg of $\text{Ca}(\text{OH})_2$ per gram.

The analyses were performed in duplicate using two individual readings per test. Thus, the average results and standard deviations of $I_{\text{Ca}(\text{OH})_2}$ indices were calculated considering a total of four results. Lastly, the confidence interval was estimated by Student's *t*-test, with a 5% significance level for reproducibility verification.

3. Results and Discussion

3.1. Characterization of the Materials Used. According to the tests performed to determine the specific mass, the MFSCBA presented values of 2.52 g/cm³. The specific mass of the fly sugarcane bagasse ash reported by Cordeiro et al. [8] of 2.569 g/cm³ was close to the MFSCBA sample. Della et al. [38] emphasize that the lower specific mass of fly sugarcane bagasse ashes results from a greater quantity of pores as compared to the BSCBA. Figure 2 shows the visual images and micrographs of FSCBA and MFSCBA samples.

The FSCBA (Figure 2(a)) and MFSCBA (Figure 2(b)) images were made with an approximation of 100x and 5000x, respectively, in order to provide a clearer image due to the micronization process.

Therefore, Figure 2 shows the efficiency of air jet milling in micronization. The fragments and irregularities of the faces in their most varied forms are characteristics of this type of process, in which collisions and friction are agents that promote grain size reduction. Figure 3 shows the granulometric patterns and X-ray diffraction (DRX) patterns of the FSCBA and MFSCBA categories.

Figure 3(a) shows that both FSCBA samples have the largest particle representation in the range of 0.1 mm to 0.3 mm, which account for at least 70% of them, the majority being smaller than 0.7 mm. The results show the efficiency in reducing ash particle size using air jet milling. As for the MFSCBA sample, 98% of the particles were reduced to a size smaller than 0.02 mm (20 μm). In addition, all particles are smaller than 0.03 mm (30 μm). Therefore, the micronization method using air jet milling, in fact, presented great efficiency in particle size reduction. The granulometry obtained

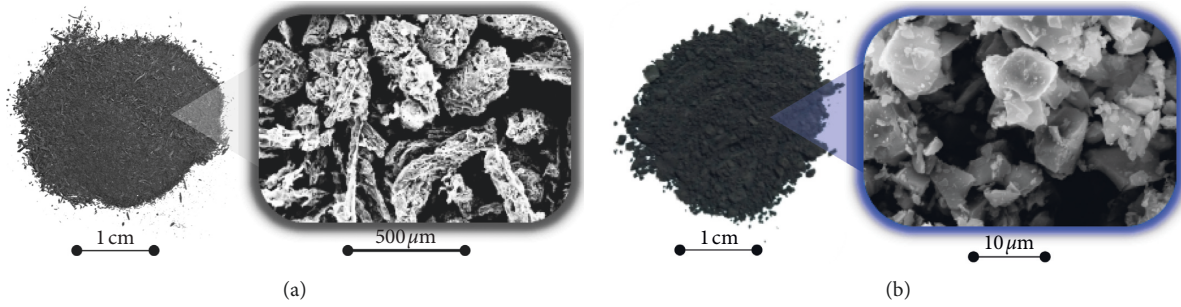


FIGURE 2: Visual aspects and SEM micrographs of fly sugarcane bagasse ash samples. (a) FSCBA and (b) MFSCBA.

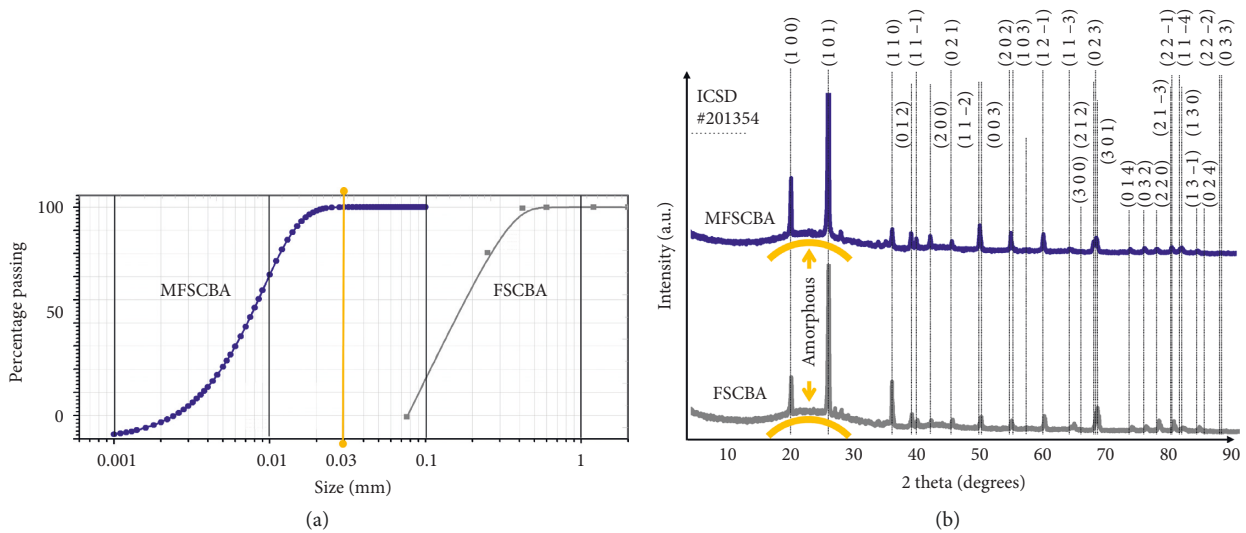


FIGURE 3: Granulometric curves of fly sugarcane bagasse ash samples and X-ray diffraction (DRX) patterns. (a) Granulometric curves of MFSCBA and FSCBA samples and (b) DRX patterns of MFSCBA and FSCBA samples.

in this method is in accordance with the requirements described in Table 1.

Figure 3(b) shows that all samples present diffraction patterns of alpha-quartz structure (standard pattern number #201354–ICSD database) as a priority pattern [39]. This pattern is the same presented by Filho et al. [40] for a sample from a Brazilian plant. The samples presented an elevation of the baseline in a range between 15° and 35°, which also indicates the presence of an amorphous phase.

Table 2 shows the chemical composition of the milled fly sugarcane bagasse ash.

Table 2 shows that the MFSCBA sample presents silicon dioxide (SiO_2) as the compound with higher representativity. In addition to SiO_2 , there are four other oxides with percentages higher than 5% and four other oxides with percentages higher than 1%. Silicon dioxide, iron dioxide, and aluminum dioxide, together with calcium dioxide, can also be observed with good representativity in milling fly sugarcane bagasse ash samples in other studies [7–9, 14].

3.2. Evaluation of MFSCBA as a Pozzolanic Material. Table 1 shows the list of characterization results obtained for the MFSCBA sample. As per Brazilian standard NBR 12653

[17], the milling fly sugarcane bagasse ash is an artificial E class pozzolan resulting from industrial transformation processes.

The MFSCBA sample did not satisfy all prerequisites. However, although the loss on ignition has exceeded standard recommendations, it is lower than the 17.6% found in the sample investigated by Lima and Rossignolo [41] and close to the 11.9% reported by Cordeiro et al. [8].

Lima and Rossignolo [41] used a sample of ash as a pozzolan with 18.02% alkali content. The authors argued that even though this is a significant percentage, it does not represent more than the 0.6% of limit of the total concrete mass. The cement used in this study is estimated to have only 1.1% alkali content. Replacing the cement mass for 25% of MFSCBA would produce a cement compound with 2.18% alkali content. This percentage would represent only 0.48% of the total mass of mortars as the ones dosed in this study. Such alkali percentage would be even less in concretes, given the presence of gravel.

3.3. Determining the Pozzolanic Performance Index with Portland Cement at 28 Days. The result of the mean axial compressive strength at 7 days for Mref was 21.4 MPa with

TABLE 1: Prerequisites, methods, and results of MFSCBA characterization.

	Prerequisites	Class E artificial pozzolan [17]	Methods [17]	Results for MFSCBA
1	SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃ (% min.)	50	X-ray fluorescence spectroscopy (XRF)	74.15
2	SO ₃ (% max.)	5	X-ray fluorescence spectroscopy (XRF)	1.2
3	Moisture content (% max.)	3	Thermogravimetric analysis and moisture control	In accordance ^a
4	Loss on ignition (% max.)	6	Thermogravimetric analysis (TGA)	13.28 ^b
5	Alkalis (% max.)	1.5	X-ray fluorescence spectroscopy (XRF and NBR NM 25 (2003))	5.4 ^b
6	Amount of particles smaller than 0.045 mm (% min.)	80	Air jet milling process and measurement by laser refraction analyzer	100
7	Structural organization with amorphous indication	Indication	X-ray diffraction (XRD)	In accordance ^a

^aThe moisture content was controlled and checked considering mortar production. ^bOff-specification requirements.

TABLE 2: Chemical composition of the milled fly sugarcane bagasse ash sample.

	Elements	MFSCBA (%)
1	SiO ₂	60.833
2	K ₂ O	8.104
3	Fe ₂ O ₃	7.754
4	CaO	7.744
5	Al ₂ O ₃	5.563
6	P ₂ O ₅	3.932
7	TiO ₂	1.771
8	MgO	1.571
9	SO ₃	1.401
10	MnO	0.704
11	Cl	0.355
12	SrO	0.074
13	Na ₂ O	0.058
14	Cr ₂ O ₃	0.048
15	ZnO	0.036
16	ZrO ₂	0.033
17	CuO	0.007
	Others	0.012

maximum relative deviation (MRD) of 2.4%, in accordance with the 6% limit required as per NBR 7215 [23]. All mortars obtained the expected CI of 20 cm ± 1. The Mmf required the use of a superplasticizer (0.5%) in both preparations to correct the CI. The existence of unitary mass values around a level of 2 g/cm³ is observed. Such values are consistent with the mortars used in the research by Filho et al. [40]. The specimens were molded after calculating the Mmf CI.

The axial compressive strength values in MPa described in Table 3 show the results of six specimens. Table 3 also shows the statistical indicators of Student's *t*-test related to the mean of both samples.

The results of the mean compression for Mmf samples are 20% greater than the mean of Mref results. The results of the mean compressive strength at 28 days for Mref (Table 3) are similar to the ones obtained by Filho [42], which was 25.14 MPa, and by Lima and Rossignolo [41], which was 27.19 MPa, using high initial strength cements CP V-ARI. In addition, Filho [42] used sand of the same origin and specifications as the one used in this study.

TABLE 3: Axial compressive strength and statistical indicators of mortars.

Specimens by batches and statistical indicators	Compressive strength indices (MPa)			
	Mref ₁	Mref ₂	Mmf ₁	Mmf ₂
CS1	26.5	25.6	34.1	31.2
CS2	27.1	26.5	31.8	34.6
CS3	27.3	29.7	34.2	30.1
CS4	28.4	25.2	31.5	34.0
CS5	26.0	24.8	29.5	29.7
CS6	25.9	24.9	29.4	29.7
Maximum relative deviation (MRD)	4.9	14.3	7.8	7.4
Variance	0.88	3.46	4.53	4.99
Observations	6	6	6	6
Mean		26.5		31.7
Mean variance		2.17		4.76
Null hypothesis (<i>h</i> ₀)		0		0
Degrees of freedom (DF)		10		10
Stat <i>t</i>		0.88		0.16
<i>P</i> (<i>T</i> ≤ <i>t</i>) one-tailed		0.20		0.44
Critical <i>t</i> one-tailed		1.81		1.81
<i>P</i> (<i>T</i> ≤ <i>t</i>) two-tailed		0.40		0.88
Critical <i>t</i> two-tailed		2.23		2.23

Compressive strength values for Mmf are significantly higher than those obtained by Filho [42], which was 15.65 MPa at 28 days for his mortar with 26% of the cement being replaced for ash. The strength results (concrete specimens) of Delalibera et al. [9], which were about 20 MPa (rich trace) at 28 days, for an estimated trace with 25% of the cement replaced for MFSCBA, are lower than those obtained in this research.

According to Table 3, in all cases, the statistics of the test (Stat *t*) was within the 95% confidence interval both in the one-tailed test (below the critical *t*) and the two-tailed test (ranges between critical - *t* and critical + *t*), assuming the hypothesis *h*₀. The *p* value (*P* (*T* ≤ *t*)) higher than the evaluated significance value (*α*) of 0.05 (5%), in all cases, is another statistical evidence that both batches produce means with repeatability guarantee. Therefore, the results of the means can be used with 95% confidence to estimate the mean result of both test batches. The triplicate was not performed due to the satisfactory results of the duplicates.

Considering the acceptance conditions of compressive strength analyses of the batches, the pozzolanic performance index of the ash with Portland cement (IDP28) was obtained by using equation (1) from the result of the mean compressive strength. The Mmf obtained IDP28 was 120%.

It is noteworthy that the Mmf IDP28 result was satisfactorily reached with a performance that increased the mean mortar strength by 20% when compared to Mref. In addition, this result preliminarily indicates the probable existence of reactivity in oxides with Ca(OH)_2 in the presence of water, especially in regard to the SiO_2 presented in the MFSCBA in the amorphous state, to form hydrated calcium silicate.

Figures 4(a) and 4(b) show the visual aspects of the mortar specimens. Figures 4(c) and 4(d) present the internal structure images obtained via SEM/EDS of the ruptured mortars at 28 days, as well as the points of measurement of the dispersive energy in specimen fragments.

A difference in color among the sample specimens can be observed. The Mmf sample (Figure 4(b)) presented a darker color, similar to the coloration of the MFSCBA sample itself (Figure 2(b)).

As complementary results in Figure 4, EDS data in both Figures 4(e) and 4(f) show a similar Ca and Si ratio, as it was expected. Value deviations regarding the percentages shown in the table occurred because carbon and oxygen percentages were maintained during measurement and are not considered reliable values to be used for comparison purposes. There is no significant difference related to the association of these compounds between mortars; thus, it is possible to assume that Mmf kept at least the same level of formation of the main crystals obtained in the Mref. In the Mref (Figure 4(c)), it is possible to observe the presence of the crystals, which include formations which suggest the presence of calcium hydroxide, calcium silicate hydrate, cristobalite, calcium monosulfate, and ettringite, among others, similar to those reported by Mehta and Monteiro [2], Kazmi et al. [34], and Melo [43]. Moreover, Mref samples presented the largest cracks and voids in the agglomerates when compared to the other samples.

However, the Mmf aspect, especially regarding the formation of continuous plate on large scale with reduction of capillary voids, as mentioned by Mehta and Monteiro [2], was highlighted in the SEM analysis. This could be a probable consequence of the pozzolanic effect attributed to the system, as it was observed by Kazmi et al. [34] and Melo [43]. The reduced amount of capillary voids, the cohesive extension of the plates (Figure 4(d)), and their forms are possibly related to the 20% increase in Mmf strength when compared to the Mref.

3.4. Pozzolanic Activity Indices Using the Modified Chapelle Method. The results obtained for the chemical reactivity indicator were 604 and 596 mg of Ca(OH)_2 per gram for the first batch and 604 and 625 mg of Ca(OH)_2 per gram for the second batch. Thus, the average result of all values in the analysis of the pozzolanic activity index ($I_{\text{Ca(OH)}_2}$) for MFSCBA was 607 mg of Ca(OH)_2 per gram, with $\sigma = 11$ and

confidence interval estimated to be between 587.7 and 627.7 mg of Ca(OH)_2 per gram. It is worth noting that the value obtained with MFSCBA is higher than the minimum recommended by Raverdy et al. [37], which is 436 mg of Ca(OH)_2 per gram.

The results of the statistical confidence intervals obtained show that the reproducibility projections for new results, with 95% confidence provided by Student's *t*-test, reproduced values with satisfactory pozzolanic performances for MFSCBA. As the data were in agreement, a triplicate was not necessary.

The result of the fixation index endorses the hypothesis that the oxides present, in fact, contributed to the formation of calcium silicate hydrate in Mmf. This result is higher than $I_{\text{Ca(OH)}_2}$ obtained by Filho [42], which is 569 mg of Ca(OH)_2 per gram for the fly sugarcane bagasse ash sample, which had amorphous structural characteristics. Cordeiro et al. [8] obtained 736 mg of Ca(OH)_2 per gram with the same ash.

In view of this information, the $I_{\text{Ca(OH)}_2}$ of 607 mg of Ca(OH)_2 per gram for the MFSCBA sample represents a satisfactory result when compared to other results of residues evaluated as pozzolanic, especially in comparisons made with SCBA.

3.5. The Potential Use of fly Sugarcane Bagasse Ash as a Pozzolan in Brazil. The 2018/2019 Brazilian sugarcane harvest alone would have a production potential of up to 0.419 million tons of FSCBA, considering that 11% of all the ashes generated as a result of bagasse burning is estimated to be fly sugarcane bagasse ash [14, 16]. With the use of the air jet milling technology in ash micronization proposed in this study, up to 0.398 million tons of MFSCBA could be obtained. This amount of MFSCBA would reduce up to 0.7% of Portland cement consumption in relation to the 57 million tons consumed in Brazil in 2016, merely by using the MFSCBA as a pozzolanic addition [44]. According to Fairbain et al. [13], this volume of MFSCBA would allow for the effective reduction of 137.919 thousand tons of CO_2 emissions. However, it is important to develop new studies on FSCBA micronization using air jet mills on an industrial scale, and such studies would have to focus on the acquisition of processing lines to meet the estimated demand.

If the prospected volume of fly sugarcane bagasse ashes was prepared as a green pozzolanic material to partially replace 25% of Portland cement mass, this would lead to a production of up to 1.592 million tons of Portland cement of this new composite class in the current sugarcane harvest. This amount of cement dosed in concretes that present reference consumption around 400 kg of cement per cubic meter would provide the production of up to 3.980 million cubic meters of green concrete. This volume of concrete would suffice to build as many as 49 Maracanã stadiums or 127 New World Trade Center complexes [40, 45, 46].

Partnerships in areas such as preparation, commercialization, and distribution of fly sugarcane bagasse ash could be established based on mutual interest between the sugarcane mills and Portland cement composite factories. To that effect, it is recommended that a cost-benefit ratio

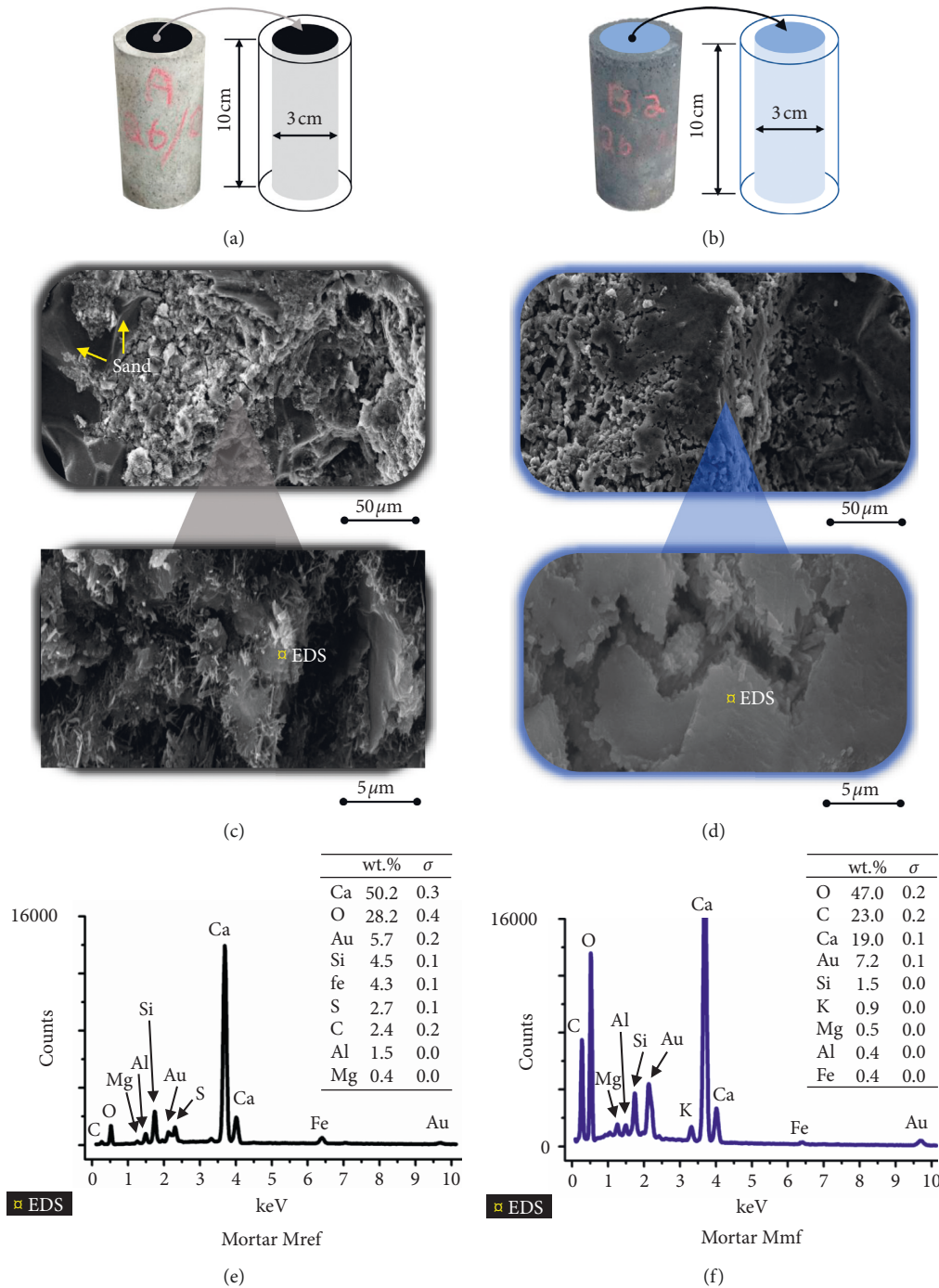


FIGURE 4: Unmolded mortar specimens after curing at 28 days and SEM/EDS images. (a) Mref with indication of sampling point area, (b) Mmf with sampling point area, (c) SEM micrographs for Mref, (d) SEM micrographs for Mmf, (e) SEM/EDS images for Mref, and (f) SEM/EDS images for Mmf.

analysis regarding the sharing of return flows of logistics routes used in the distribution of the respective outputs be made, among other possibilities. The maximum sugarcane bagasse FSCBA projection generated in the mills in the states of São Paulo, Minas Gerais, Goiás, Mato Grosso do Sul, and Paraná, which will represent 88% of the 2018/2019 (2018) national harvest, could be absorbed by the 37 (42%) cement factories located in these states. Kurda et al. [47] report that environmental impacts can be reduced, even considering

long distances between coal power and concrete plants. They emphasize that the increase in the use of coal ash reduces the overall impacts of the production chains involved.

Finally, it is noted that the addition of MFSCBA to Portland cement is an excellent alternative to reduce CO₂ emissions per ton produced. Therefore, the reduction mentioned in this study would result in a great positive environmental impact, given that the cement industry is the second largest CO₂ emitter.

4. Conclusions

The innovative micronization of FSCBA using air jet mills allowed for the development of a solution for the use of milling fly sugarcane bagasse ash to produce green pozzolana. The main highlights were as follows:

- (1) Air jet milling proved to be a technically viable and efficient alternative for the FSCBA micronization to achieve the granulometric requirements established in pozzolanic studies.
- (2) The 120% IDP28 indicator and $I_{Ca(OH)_2}$ of 607 mg of $Ca(OH)_2$ per gram confirmed the existence of pozzolanic activity in MFSCBA, with no use of reburning thermal treatment.
- (3) The expressive result for MFSCBA's IDP28 indicator emphasizes that MFSCBA pozzolanic activity with a maximum dimension of $30\ \mu\text{m}$ increased the mortar axial compressive strength by 20%. SEM images evidenced the suggestive cohesive formation of particles originated from the pozzolanic activity process, as well as oxide hydration of the cement.

All these actions can lead to advances that address social and environmental responsibilities related to the application of agro-industrial residues in the production of Portland cement. Furthermore, this initiative could help Brazil stand out in terms of CO_2 emissions control practices related to Portland composite cement production, complying with national and international legislation.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors herein declare that the publication of this paper will not lead to any conflicts of interest.

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