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Ultrafine grinding of sugar cane bagasse ash for application as pozzolanic admixture in concrete

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ABSTRACT

Sugar cane bagasse ash, a byproduct of sugar and alcohol production, is a potential pozzolanic material. However, its effective application in mortar and concrete requires first the controlled use of grinding and classification processes to allow it to achieve the fineness and homogeneity that are required to meet industry standards. The present paper investigates the role of mill type and grinding circuit configuration in grinding in laboratory- and pilot plant-scale on the particle size, specific surface area and pozzolanic activity of the produced ashes. It was observed that, although different size distributions were produced by the different mills and milling configurations, the pozzolanic activity of the ground ash was directly correlated to its fineness, characterized by its 80% passing size or Blaine specific surface area. From a low pozzolanic activity of less than 50% of the as-received ash, values above 100% could be reached after prolonged grinding times. Electric power requirements to reach the minimum pozzolanic activity were estimated to be in the order of 42 kWh/t in an industrial ball mill. Incorporation of an ultrafinely-ground ash in a high-performance concrete in partial replacement of Portland cement (10, 15 and 20% by mass) resulted in no measurable change in mechanical behavior, but improved rheology and resistance to penetration of chloride ions.

1. Introduction

The use of pozzolanic materials as admixtures both in the production of clinker and in partial replacement of Portland cement in mortars and concrete increased significantly in the last 25 years. Investigations demonstrated the validity of using these materials from both technical and environmental reasons and, at times, even economical. Some traditionally used pozzolanic materials, such as silica fume, fly ash, metakaolin and rice husk ash, are by-products of industrial and agroindustrial processes, which further contribute to reduce both environmental and economical problems that would be associated with their disposal.

Only a few industrial operations are required to transform some of the cited industrial by-products in pozzolans. These operations typically correspond to grinding and classification, which are responsible for the controlled reduction in particle size and increase in specific surface area. Such increase in specific surface area is directly responsible for the kinetics of their pozzolanic reactions, which can be limited in the case of poorly reactive pozzolans. The use of pozzolans in the fine and ultrafine size range along with Portland cement can also allow reaching greater packing density of the mortar or cement mixture, due to the so-called microfiller effect [1]. The other physical effect that becomes potentially important with the reduction in particle size is the heterogeneous nucleation. In this case, pozzolan fine particles settle in between the clinker crystals, allowing a nucleation of hydrates on foreign fine particles by reducing the energy barrier [2].

Preliminary investigations with sugar cane bagasse ash (SCBA) demonstrate that it presents appropriate chemical composition for application as a pozzolan, mainly in regard to its high silica content and presence of amorphous silica [3–8]. However, it typically presents variable and coarse particle size distribution, so that the production of pozzolanic ash from SCBA requires the use of ultrafine grinding to transform this industrial residue in a mineral admixture [7].

The paper analyzes the influence of different mechanical grinding configurations, carried out in laboratory and pilot-scale mills, on the physical characteristics of SCBA. Detailed measurements of particle size distribution, specific surface area, pozzolanic activity have been carried out and the specific grinding energy required to obtain the different comminution products has been estimated. The feasibility of using a selected sample of produced ultrafine SCBA in high-

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performance concrete has also been demonstrated, and a number of properties of the concrete were assessed both in fresh (rheology) and hardened states (compressive strength and resistance to chloride ion penetration).

2. Materials

The as-received SCBA sample was collected at an agroindustry that produces sugar cane and alcohol in the State of Rio de Janeiro (Brazil). Samples were collected during the cleaning operation of the boilers in the factory. In the boiler, the sugar cane bagasse is burnt at temperatures varying from 700 to 900 °C, depending on the moisture content and feed of the bagasse. Table 1 shows a summary of the physical characteristics and the chemical composition of both SCBA and Portland cement (PC) used later in the investigation in preparing mortars and concrete. Of particular interest in Table 1 is the high silica content (78.3%) of SCBA and the low loss on ignition (0.4%). Fig. 1 presents the X-ray diffraction (XRD) pattern of SCBA, where the predominance of silica as cristobalite and quartz is evident. The quantitative XRD analysis was performed using Bruker's Topas v. 3 software [9], which is based on the Rietveld method [7]. The mass percent values of calculated phases from analysis are 59% of quartz, 16% of cristobalite and 24% of amorphous with an estimated error of ±4%. This high content of quartz is ultimately due to sand adhered to the sugar cane and that is harvested along with it. Even after washing of the sugar cane, the factory reports [8] that sand can represent as much as 2% in weight of the material that is processed. After the loss of organic matter during burning the bagasse, this proportion increases significantly, reaching values as high as those found for quartz (the main constituent of the sand in the region) observed in the present study. The presence of cristobalite is associated to the high temperature in the boiler. The reactivity of this SCBA was recently investigated by Cordeiro [8] using ^{29}Si nuclear magnetic resonance spectroscopy of hardened cement pastes. The results indicated that the intensity of the Q^4 peak, characteristic of amorphous silica (chemical shift of 110 ppm), decreased with the hydration development, which suggests its pozzolanic activity.

In the preparation of mortars, standard natural sand [10] and deionized water were used. Coarse aggregate (19–6.3 mm) composed predominantly of crushed syenite, fine aggregate of siliceous river sand (with D_{50} of 0.9 mm and a fineness modulus of 2.12), and polycarboxylate-based superplasticizer were used in formulating the concrete mixtures.

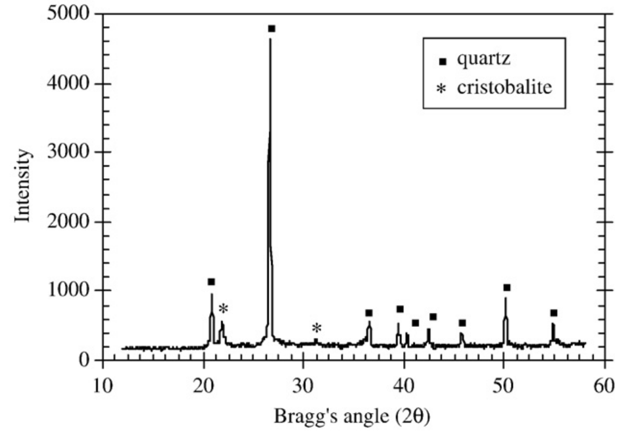

Fig. 1. X-ray diffraction pattern of as-received SCBA.

Table 1
Physical characteristics and chemical composition of as-received SCBA and Portland cement (PC)

Physical properties		
Characteristic	SCBA	PC
Density (kg/m^3)	2530	3170
Blaine specific surface area (m^2/kg)	196	308
Median particle size, D_{50} (µm)	76.3	16.9
% Passing 45 µm	67.4	8.4
Chemical composition (wt.%)		
	SCBA	PC
SiO_2	78.34	20.85
Al_2O_3	8.55	4.23
Fe_2O_3	3.61	5.25
CaO	2.15	63.49
Na_2O	0.12	0.16
K_2O	3.46	0.40
SO_3	–	2.38
MnO	0.13	<0.05
MgO	1.65	–
P_2O_5	1.07	–
L.O.I[a]	0.42	1.05

[a] Loss on ignition.

3. Methods

3.1. Grinding of SCBA

Grinding tests were carried out in either open circuit or closed circuit, using tumbling mills with steel and alumina balls, a vibratory mill with short alumina and a hammer mill, all operating in dry mode. In the case of the tumbling mill two types of tests were conducted, both using batch mills (Table 2). In one set of experiments, the mill was run in a fully batch mode, thus simulating continuous open circuit operation, and milling experiments were conducted for up to 960 min. In the second type of experiment, the mill was run in batch mode, but simulating closed circuit operation by classifying the product of each batch using a 45 µm sieve and replacing the amount of passing material with a fresh batch of feed for a new cycle, until reaching steady-state conditions. Mill type and milling conditions corresponded exactly to those described in the standard Bond grindability test [11] used to determine grindability in a ball mill. Vibratory grinding experiments were carried out using a mill manufactured by Aulmann & Beckschulte Maschininfabrik (Germany) that was operated in batch mode (also simulating continuous open circuit operation) for grinding times of up to 240 min (Table 2). Grinding in a hammer mill was performed in a pilot-scale mill operating continuously in closed circuit with a dynamic air classifier. The mill operates at 2200 rpm with the classifier set for a product finer than about 30 µm. The circuit production rate was about 0.15 t/h.

Bond [11] proposed an expression that allows estimating the power consumption (W, in kWh/t) of an industrial ball mill operating under standard conditions. This expression is based on the

Table 2
Summary of mill characteristics and grinding conditions used in tumbling mill and vibratory mill tests

		TM	BM	VM
	Type	Tumbling mill	Tumbling mill	Vibratory mill
Mill	Diameter (mm)	240	300	190
	Volume (L)	10	20	33
	Speed (rpm)	35 (40% of critical)	70 (75% of critical)	–
	Type	Alumina balls	Steel balls	Alumina cylinders
Grinding media	Size (mm)	10–30	15–40	13
	Volume (L)	5	4	16
	Filling (%)	50	20	48
Sample	Volume (L)	2.5	0,7	8

Table 3
Mixture proportions of concretes (kg/m^3)

Materials	Proportion of cement replaced by SCBA in the mixture			
	0%	10%	15%	20%
Portland cement	478.0	430.2	406.3	382.4
SCBA	–	47.8	71.7	95.6
Fine aggregate	860.0	860.0	860.0	860.4
Coarse aggregate	905.3	905.3	905.3	905.8
Water	167.4	167.4	167.4	167.4
Superplasticizer	1.43	1.43	1.43	1.20

measurement of the work index [11,12], which is a parameter that expresses the resistance of the material to grinding. In SCBA grinding, where size reduction is carried out invariably dry, in either open (approximated in batch tests) or closed circuits and resulting in products that could reach significant fineness, Bond's law is given by

$$W = 13Wi\left(\frac{1}{\sqrt{P_{80}}} - \frac{1}{\sqrt{F_{80}}}\right)\left(\frac{P_{80} + 10.3}{1.145P_{80}}\right)k \tag{1}$$

where Wi is the Bond ball mill work index (kWh/t), P_{80} is the sieve opening which 80% of the product passes (in μm), A_{80} is the sieve opening which 80% of the feed passes (in μm) and k is a correction factor equal to 1.2 for open circuit and 1.0 for closed circuit grinding [13].

3.2. Characterization of ashes

Particle size distributions were measured using a laser diffraction particle size analyzer (Malvern Mastersizer) in liquid mode with analytical-grade ethyl alcohol as dispersant and ultrasound agitation for 60 s. The fineness of the ashes was characterized using the 80% passing size (D_{80}) in the cumulative distribution. Particle morphology was characterized qualitatively with the aid of scanning electron microscope (SEM) images, obtained using a Jeol 840-A.

The index of pozzolanic activity with Portland cement, described in the Brazilian standard NBR 5752 [14], was calculated from the ratio between the 28-day compressive strengths of mortars with SCBA, and a reference, represented by mortars prepared using only cement, sand and water. Mortar mixtures were all prepared using a constant 1:3 (weight basis) cement–sand ratio and the amount of water required to achieve a consistency index [15] in the range of 225±5 mm. In the mixtures with pozzolan, 35% in volume of the Portland cement was replaced by the pozzolanic material. Preliminary tests were conducted with different ashes with the aim of identifying a water/cementitious material ratio that resulted in consistency indices within the specified range. This was achieved with a water/cementitious material ratio of 0.52. After mixing and molding, cylinders of 50 mm diameter and 100 mm height were maintained in a moist chamber during the first 24 h at a temperature of 22 °C and 100% relative humidity. Then these specimens were demolded, sealed with plastic film and stored in hermetically closed containers at a temperature of 38 °C±2 °C until aging for a total of 28 days. At the end of the curing process, specimens were tested until fracture in a servohydraulic press (Shimadzu UH-F1000kNI) operating at 0.1 mm/min.

3.3. Mix proportions, preparation of specimens and test method of concretes

High-performance concretes were formulated within the framework of the Compressible Packing Model [16] using the computer code Betonlab Pro2 [17]. The water/cementitious materials ratio was maintained constant at 0.35. The incorporation of SCBA was carried out by replacing 10%, 15% and 20% of the cement mass. The SCBA concretes were compared with the reference mixture. Slump was kept constant at 150±20 mm with specific contents of superplasticizer. Table 3 summarizes the mix proportions based on a constant mixture volume of 1 m^3. For each mixture, specimens were left to cure for 24 h, and then demolded and cured in a moisture-controlled room at 100% relative humidity of 21±1 °C of temperature. Rheological analyzes were carried out using the BTRHEOM rheometer [18]. It was assumed that the fresh concrete behaves as a Bingham fluid, for which there is a linear relationship between shear stress and shear velocity gradient. In the 28-day compressive strength (f_c) tests, the average value of two diametrically opposed linear variation displacement transducers was used to control the servohydraulic machine with displacement rate of 0.0075 mm/min. The modulus of elasticity (E_c) was calculated considering linearity of the stress-strain curve for stresses in the range $50.10^{-6}E_c$ to 0.4 f_c. The results of the mechanical response tests were validated by statistical testing using analysis of variance (ANOVA) and Duncan's Multiple Range Tests [19] (probability≤0.05). Results are expressed as average of 4 specimens±standard deviation. In addition, the rapid chloride permeability test was carried out using the procedures described in the ASTM C 1202-05 [20].

4. Results and discussion

4.1. Production and characterization of the ashes

Table 4 presents a summary of particle size and specific surface area data of the various experiments. In the case of the grinding tests performed in batch mode (TM and VM), the expected reduction in particle size and increase in specific surface area with the increase in milling time is evident. At the longest milling time investigated in vibratory milling a reduction from a feed representative size (D_{80}) of 199.0 μm to 5.4 μm was achieved. Fig. 2 shows a comparison between SCBAs produced after 8 min and 240 min of vibratory grinding. Fig. 2-a shows that SCBA ground for 8 min presented an heterogeneous mixture of coarser quartz particles and particles with porous cellular structure. After 240 min of grinding, SCBA also presents some coarser quartz particles; however, the cellular grains were totally broken down (Fig. 2-b) by the mechanical action of the grinding media. From Fig. 3 it is evident that grinding resulted in a reasonably smooth and continuous shift of the particle size distributions to finer sizes with milling time. A comparison between data for the mills operating in fully batch mode shows that significantly finer products are obtained in the vibratory mill (VM) when compared to the tumbling mill (TM) after grinding at comparable times, which confirms the faster breakage kinetics reached in the former. The reasonably wide size distribution of the as-received SCBA, shown in Fig. 3, indicates the need for grinding techniques to reduce and control its particle size distribution.

Table 4
Characteristic parameters of particle size distributions of SCBA produced under different grinding types and configurations

Grinding configuration	Grinding time (min)	D_{80} (μm)	Blaine specific surface area (m^2/kg)
Sample as-received	–	199.0	196
Tumbling mill in batch mode (TM)	30	113.2	201
	60	81.3	218
	120	69.7	260
	240	52.1	319
	480	34.5	409
	960	27.2	512
Vibratory mill in batch mode (VM)	8	73.4	277
	15	61.8	395
	30	31.8	444
	60	15.6	640
	120	8.5	893
	240	5.4	1197
Tumbling mill in closed circuit (BM)	–	47.2	295
Hammer mill in closed circuit (HM)	–	19.3	523

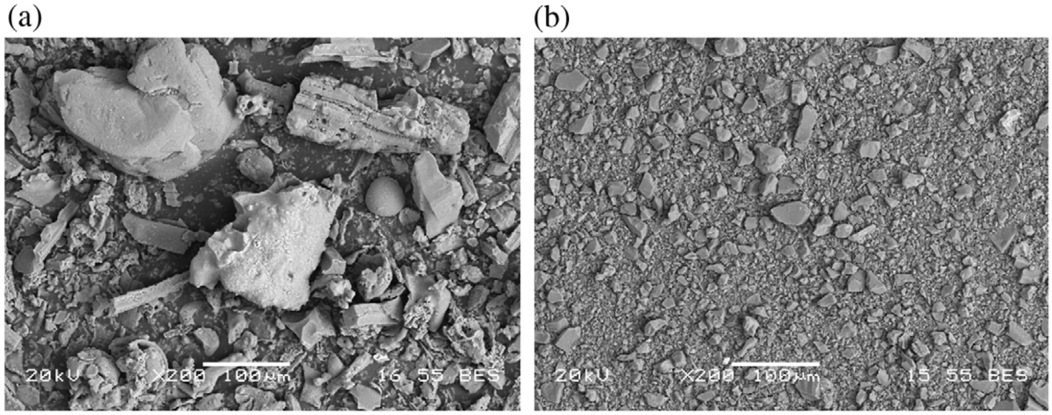

Fig. 2. SEM images of SCBAs produced after 8 min (a) and 240 min (b) of vibratory grinding.

Bond ball mill (BM) and the hammer mill (HM) tests, both conducted in closed circuit with classification, generate products that are characterized by even greater uniformity (range of the particle size is reduced) when compared to the original material (Fig. 3-b). This can certainly be attributed to the mode of operation, since coarser particles produced often return to the mill after being classified. The fineness of the products from these tests was directly associated to the setting of the classification stage. Fig. 4 demonstrates that an inverse relationship exists between representative particle size (D_{80}) and Blaine specific surface area. This behavior is relatively independent of mill type and milling configuration, including even the as-received SCBA.

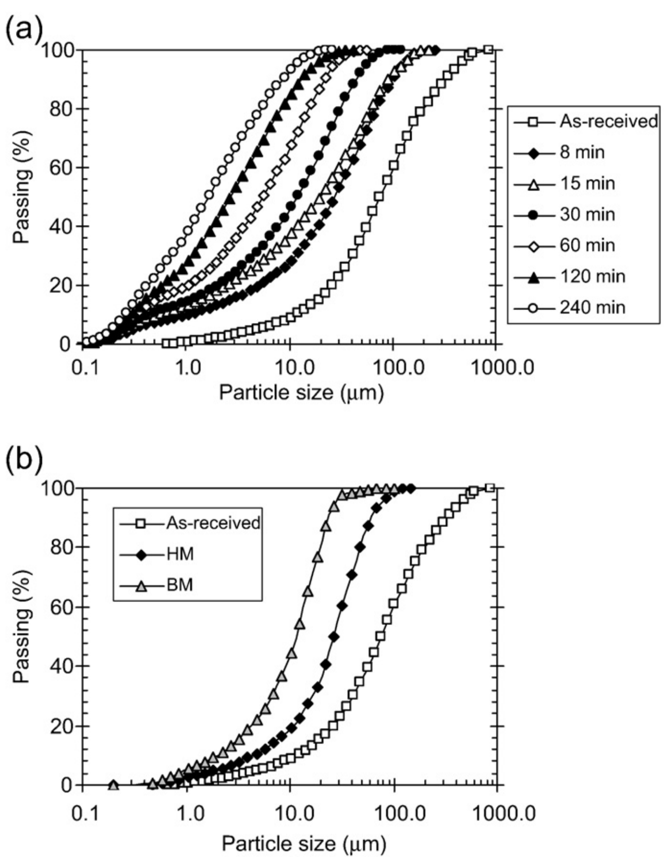

Fig. 3. Selected particle size distributions of ground SCBAs conducted in vibratory mill (a) and closed circuit (b).

Table 5 summarizes data on compressive strength and consistency index for the different mortars. It is observed that the consistency of the mortars containing SCBA does not significant change as compared with that of the reference mortar (all indices are in the range of 225 ± 5 mm). No measurable difference in mortar water demand was detected either for SCBA ground in closed or open circuit configurations. This behavior contrasts with the one observed for fly ash [21] and Portland cement [22], where grinding in closed circuit results in not only a steeper size distribution, but also a higher water demand. The modest change of water demand of SCBA mortars can be attributed to the increase in the proportion of regularly-shaped particles (Fig. 2-b).

Fig. 5 compares results on the pozzolanic activity index of the different products of ultrafine grinding, along with data for the as-received SCBA. In general, it is evident that the values of pozzolanic activity index increase as specific surface increase and particle size (D_{80}) of the ash decreases. Fig. 5 also confirms the need for ultrafine grinding of SCBA, since the as-received material presents an index of pozzolanic activity of only 49%. The Brazilian standard NBR 12653 [23], in analogy to the American standard ASTM C 618-05 [24], defines as 75% the minimum value to classify a material as pozzolan. This condition has been achieved in all milling experiments where the specific surface area was larger than about 300 m^2/kg and D_{80} finer than about 60 μm, with the only exception of the grinding experiment in the tumbling mill for 240 min. Therefore, products generated from both closed-circuit grinding tests (HM and BM), from all vibratory milling (VM) tests of at least 15 min and conventional milling (TM) tests of at least 480 min were classified as pozzolans.

It is also worth noting that the product of 240 min of vibratory grinding presents a pozzolanic activity index that is higher than 100%.

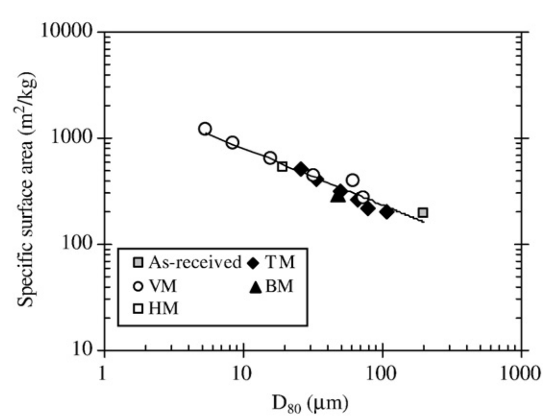

Fig. 4. Relationship between D_{80} and Blaine specific surface area of ground SCBAs.

Table 5
Summary of experimental results from mortar tests

Cementitious material	Grinding configuration	Grinding time	Compressive strength[a] (MPa) – (standard deviation, MPa)	Consistency index (mm)
PC	–	–	37.81 (1.48)	226
0.35 SCBA+ 0.65 PC	As-received	–	18.71 (0.19)	220
	Tumbling mill in batch mode (TM)	30	23.27 (0.63)	220
		60	24.14 (0.13)	222
		120	24.51 (0.13)	222
		240	27.77 (0.70)	225
		480	30.32 (0.34)	230
		960	31.80 (0.25)	230
	Vibratory mill in batch mode (VM)	8	26.96 (0.66)	220
		15	29.08 (0.09)	223
		30	29.93 (0.44)	225
		60	33.63 (0.76)	230
		120	37.65 (0.96)	228
		240	38.83 (0.36)	230
	Tumbling mill in closed circuit (BM)	–	30.61 (0.89)	228
	Hammer mill in closed circuit (HM)	–	34.29 (1.86)	228

[a] Average of 4 measurements.

Data in Tables 4 and 5 show that in order to present the a pozzolanic activity index of 100% the SCBA had to present D_{80} of about 9 μm whereas the cement presents a D_{80} equal to 50 μm. Fig. 5 demonstrates that for the SCBA pozzolanic activity is primarily determined by specific surface and particle size, so that effects of mill type and even grinding configuration (open or closed circuit) are only of secondary importance.

While Fig. 5 demonstrates that increases in pozzolanic activity will result from reductions in particle size, it is also important to estimate the associated electric power required in grinding. Bond ball milling experiments resulted in a Bond work index Wi equal to 36.7 kWh/t for SCBA. Fig. 6 presents data on pozzolanic activity plotted as a function of the equivalent specific energy consumption W (Eq. (1)). In spite of the scatter and considering an interpolation of the data, it shows that SCBA requires as least 50 kWh/t of grinding power to make it a pozzolanic material when grinding in a ball mill operating in open circuit in industry. This value may be further reduced (down to about 42 kWh/t) by grinding in closed circuit (Fig. 6), which is more energy-efficient.

In addition to that, it is also relevant to compare the energy required for SCBA to reach the pozzolanic activity index of 100%. Fig. 6 shows that as much as 300 kWh/t would be required to reach this product specification in ball milling in open circuit. This could be reduced to about 250 kWh/t by operating in closed circuit (although the energy expenditure of classifier that should be added is not computed here and could be significant) and perhaps even down to about 125 kWh/t if other more energy-efficient ultrafine grinding mills are used. Although significant, this is very close to the typical electrical energy consumed in producing ordinary Portland cement, estimated in about 110 kWh/t [12].

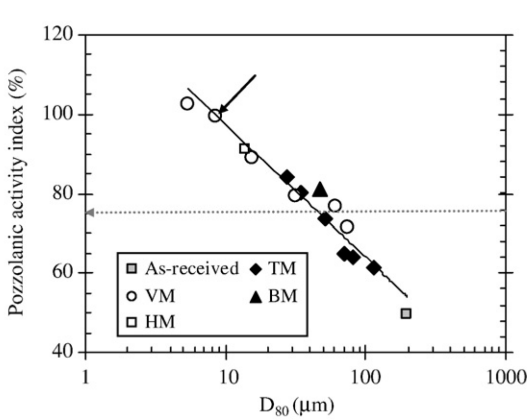

Fig. 5. Relationship between D_{80} and pozzolanic activity index for the SCBAs. The dotted line represents the minimum value that characterizes a material as pozzolanic according to the Brazilian standard NBR 12653 [23].

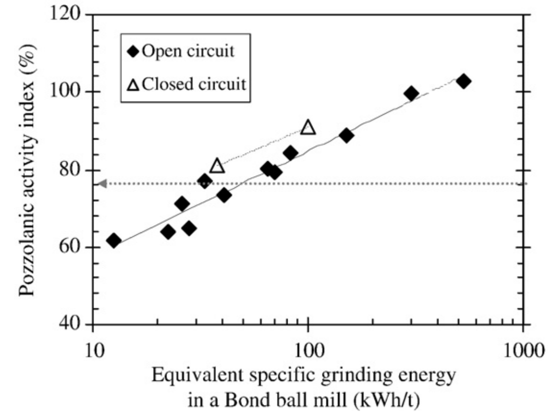

Fig. 6. Relationship between pozzolanic activity index and equivalent specific grinding energy in a Bond ball mill estimated using Eq. (1). Trend line plotted using data for open circuit operation and the dotted line represents the minimum value of pozzolanic index.

4.2. Application of an ultrafine SCBA in high-performance concrete

In order to demonstrate the feasibility and explore the benefits of applying the ultrafinely ground SCBA in concrete, the material ground in the vibratory mill for 120 min in batch (open circuit) mode was used in the experiments, since this was the product that more closely matched the strength of cement in the mortar test (pozzolanic index of 100%). Data on this grinding test is pointed out with an arrow in Fig. 5. Table 6 presents the values of yield stress and plastic viscosity calculated from data obtained using the BTRHEOM rheometer, as well as slump values. Replacement of cement by the ultrafinely ground SCBA results in an increase in slump values. In fact, in order to maintain the slump value of the concrete where 20% of cement was replaced by ground SCBA within the established values it was possible to reduce by 16% the superplasticizer content in comparison to the concrete where no SCBA was used. It is also observed that plastic viscosity increases marginally as a result of incorporation of the SCBA in the mixture. However, the yield stress is reduced for concretes using SCBA, which suggests a positive effect of the ash on the rheology of concrete. This change in the yield stress is probably due to the reduction of the particle interlocking and internal friction, which may be explained by the presence of regular-shaped particles in the SCBA, as previously discussed. Additionally, the negligible carbon content (Table 1) of the SCBA contributes to this trend, since the concretes with high carbon content mineral admixtures require more superplasticizer for the same workability [25].

Table 7 presents a summary of data on compressive strength, peek deformation and elastic modulus at 28 days, when it is possible to observe that the replacement of up to 20% of cement by SCBA did not

Table 6
Properties of concretes in the fresh state

Cement replaced by SCBA (%, in mass)	Slump (mm)	Yield stress (Pa)	Plastic viscosity (Pa s)
0	130	693	306
10	150	362	353
15	170	196	363
20	170	211	380

Table 7
Properties of concretes in the hardened state (standard deviation is indicated within parentheses)

Cement replaced by SCBA (%, in mass)	Compressive strength (MPa)	Modulus of elasticity (GPa)	Peak strain (με)	Electric charge (C)
0%	60.86 (1.72)	34.20 (0.73)	2425 (75)	1179 (25)
10%	61.56 (0.89)	32.70 (1.20)	2595 (85)	783 (42)
15%	59.01 (3.03)	32.73 (0.24)	2590 (125)	774 (48)
20%	57.83 (0.64)	33.90 (0.54)	2585 (55)	882 (32)

change significantly the mechanical behavior of the reference concrete. No statistically significant changes were detected on the mechanical properties, at 95% significance. This behavior is consistent with the result of pozzolanic activity index (Fig. 5), once the ultrafine SCBA presents pozzolanic index of 100%. Average values of compressive strength were about 60 MPa, values of modulus of elasticity approximately equal to 33 GPa, whereas peek deformations were of about 2500 με.

Accelerated chloride-ion penetration test results, presented in Table 7, indicate that SCBA allows a reduction of about 30% in the electric charge that passes through the concrete samples, when compared to the concrete with no addition of SCBA. The incorporation of the ultrafine ash allowed the concrete to change from being originally classified by ASTM as having “low” penetration (charge ranging from 1000 to 2000 C) to “very low” (charge ranging from 100 to 1000 C) after incorporation of SCBA. No significant change is observed of the penetration of ions in relation to the amount of SCBA used in the concrete. Ganesan et al. [6] observed a significant reduction of the charge passed in 20% SCBA concrete in comparison with the reference. Similar behavior was also observed for fly ash concretes [26]. The chloride-ion penetration results suggest pore refinement due to the pozzolanic reaction of the ultrafine SCBA and demonstrate the significant potential of ash as a mineral admixture in concrete, as long as an appropriate grinding strategy is used and product fineness is achieved.

5. Conclusions

In general, grinding to values of D_{80} (80% passing size) below about 60 μm and Blaine specific surface areas above 300 m^2/kg resulted in products that can be classified as pozzolans. Particle size, characterized by the 80% passing size, and Blaine specific surface area were found to correlate well with the pozzolanic activity index of the mortars, irrespective of mill type and grinding configuration.

Power consumptions required to render SCBA a pozzolanic material were estimated to be in the order of 42 kWh/t in an industrial ball mill operating dry and in closed circuit with a classifier. On the other hand, it is estimated that about 250 kWh/t of specific grinding energy would be required in the same mill to reach the fineness of SCBA to make it capable of replacing (although partially) Portland cement with no loss in compressive strength.

The application of an ultrafinely ground SCBA produced by vibratory grinding allowed the production of high-performance concrete with the same mechanical response up to a 20% replacement as the concrete prepared solely using Portland cement. Such addition of SCBA also resulted in improvements in rheology of concrete in the fresh state and resistance to penetration of chloride-ions.

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