



# Effect of mechanical processing on sugar cane bagasse ash pozzolanicity



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## ABSTRACT

This work intends to contribute to the understanding about the effect of sugar cane bagasse ash (SCBA) on the hydration of cement-based pastes by focusing on the influence of different particle size (or specific surface area). Isothermal calorimetry, thermogravimetry, chemical shrinkage, and strength pozzolanic index tests were performed to compare the hydration and chemical evolution of pastes containing SCBA with different fineness and pastes with inert (quartz) and pozzolanic (rice husk ash) materials. The results showed that a clear change in the kinetics of hydration and portlandite content by comparing the SCBA with different particle sizes. In addition, the different SCBAs had a marked effect on the chemical shrinkage and portlandite, with an intermediate behavior between the pastes with quartz and rice husk ash. Finally, with increasing in specific surface area and soluble fraction of SCBA, its pozzolanic activity was progressively enhanced due to both portlandite consumption and physical effects.

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## 1. Introduction

There has been much recent interest in the use of sugar cane bagasse ash (SCBA) as a supplementary cementitious material (SCM), especially in countries with low demand for materials traditionally used in combination with Portland cement, e.g., fly ash, blast-furnace slag, and natural pozzolans. Searching for new SCM sources is an urgent matter in some countries [1]. Meanwhile, numerous studies have highlighted the positive effects of SCBA on rheology [2–3], mechanical properties [2–5], and durability of concrete [2,4,6], besides reduction of anthropogenic carbon dioxide emissions due to the partial replacement of Portland cement [7].

In sugar cane plants, boilers are usually fired by pure bagasse, which generate SCBA in an amount around 0.3% of the total mass of processed sugar cane [8]. Due to burning conditions of bagasse and the presence of contaminants, SCBA with several different chemical compositions can be found. In general, silica ( $\text{SiO}_2$ ) – present in an amorphous form or in a variety of crystalline polymorphs – is the main component of SCBA. With proper burning, SCBA with predominantly amorphous silica can be produced [9]. However, most SCBA is produced from industrial burning where partially crystalline silica-based SCBA is produced through of the crystallization of the original hydrated silica ( $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ) in crystalline forms (normally cristobalite) or contamination of SCBA with quartz from soil [4,10–13], or both.

Ultrafine grinding has been commonly used to remedy the adverse effects of the presence of crystalline phases in SCBA and in order to increase its homogeneity [4,11,13–15]. Grinding affects the pozzolanic

activity of partially crystalline SCBA by increasing the specific surface area [10,11] and introducing imperfections or active centers on the surface of mineral particles [16–18]. More recently, a two-stage classification grinding circuit was proposed to reduce the content of quartz in SCBA, increasing significantly its reactivity [14].

Although it seems intuitive that the pozzolanic activity of SCBA enhanced by reducing its particle size, there are no studies researching the effect of different particle sizes of SCBA on the kinetics and mechanism of hydration of cement-based pastes. Based on the above considerations, kinetics of hydration at early ages by calorimetry, portlandite consumption by thermogravimetry, and chemical shrinkage were applied in this study to evaluate the different SCBA samples – obtained from the same source – concerning the effect of particle size on their properties (specific surface area, soluble fraction, and pozzolanic activity) and hydration of cement-based pastes. Additionally, the SCBA behavior is compared with those of amorphous (rice husk ash) and inert (quartz) materials.

## 2. Materials

SCBA was obtained from a sugar mill located in the State of Rio de Janeiro, Brazil. This ash was generated after the burning of bagasse used as biomass at temperatures around 800 °C. The grinding of SCBA was performed in batch dry-mode using a 33-L vibratory mill (Aulmann & Beckschulte Maschinenfabrik, Bergneustadt-Germany) filled with 16.5 L of steel balls (diameter ranged from 18 to 38 mm in equal proportions) and 8 L of feed sample. From this single sample, four different SCBA samples were produced through use of grinding times of 8, 30, 120, and 960 min such that their particle size varied. By means of

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these grinding times, the products present  $D_{50}$  values varying from 29.6 to 4.4  $\mu\text{m}$  and are designated as SCBA $x$  ( $x = 1, 2, 3, 4$ ), where  $x$  increases as the SCBA  $D_{50}$  decreases. Fig. 1 compares the particle size distribution curves of as-received SCBA ( $D_{50} = 76.3 \mu\text{m}$ ) to those produced by grinding, evidencing the progressive particle size reduction. The details of SCBA grinding, including different mills, grinding configurations, and grinding energy consumption, are given elsewhere [11]. Fig. 2 shows that the SCBA is a mixture of quartz and porous cellular particles from bagasse.

$\text{SiO}_2$  is the most abundant constituent detected in SCBA (80.8%), as indicated in Table 1. Minor additional oxides are found in SCBA sample, especially  $\text{K}_2\text{O}$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{CaO}$ . The loss on ignition of 0.4% is indicative of the complete burning of sugar cane bagasse. X-ray diffraction patterns of SCBA1 and SCBA4 (Fig. 3) reveal two crystalline (quartz and cristobalite) and amorphous (from Rietveld refinement analysis with Panalytical HighScore software) phases. Quartz is likely to be contamination from soil [11] and has been observed in several SCBA samples [2,4,10–13,19]. The original hydrated silica is converted to cristobalite due to the long time burning at high temperature into the boilers [9]. It is worth noting that the typical baseline deviation associated with 24 wt% amorphous phase cannot be visually recognized in XRD patterns due to the highly crystalline nature of quartz. It is important to stress that the presence of amorphous compounds was not increased by the ultrafine grinding of SCBA, according to quantitative XRD. After grinding, the chemical composition and crystalline phases of different SCBA remain the same of the as-received SCBA. This is in disagreement with earlier reports on the potential for mechanochemical effects on silica caused by grinding [16,18].

A rice husk ash (RHA) produced by controlled burning and grinding procedures was used as a model pozzolanic material. The burning of a water-washed rice husk was performed under oxidizing conditions in a laboratory muffle furnace at 600 °C for 3 h with a 10 °C/min heating rate. The grinding was performed using the same procedure used for SCBA samples with a grinding time of 120 min. Fig. 4 shows the particle size distribution of RHA sample, which presents a large amount of amorphous  $\text{SiO}_2$  (93.1%) according to chemical composition (Table 1) and XRD scan (Fig. 3), which is desirable for a highly reactive pozzolan.

A highly pure ultrafine quartz (named QTZ) was used as an inert material. The commercially available sample ( $D_{50}$  around 28  $\mu\text{m}$ ) was made finer with a laboratory PM100 planetary ball mill (Retsch, Haan-Germany) to particle size distribution approximately equal to the particle size distribution of RHA and SCBA3. The grinding was conducted at 250 rpm stirrer speed using 5 g of quartz in a 50  $\text{cm}^3$  tempered tool-steel bowl

with 10 10-mm balls. The grinding was run in batch dry-mode for 30 min. Detailed chemical composition (Table 1) and XRD scan (Fig. 3) confirm the high purity of the sample, while Fig. 4 displays that the particle size distribution of SCBA3, RHA, and QTZ are relatively similar.

Pastes were prepared using an ASTM Type I Portland cement [20], Brazilian Class G cement [21] and a standard quartz sand sized from 0.15 to 2.40 mm [22] were used to cast mortars for the pozzolanic activity index tests based on the compressive strength. The oxide composition and the calculated Bogue potential phases of both types of cements are summarized in Table 1. Type I cement had about 3% of calcium carbonate (quantified by thermogravimetry) in its composition (ASTM C150/C150M-16 [20] allows up to 5% by mass of the final cement to be finely ground limestone in Type I cement). The particle size distribution of the cements is displayed in Fig. 4. Polycarboxylate-based superplasticizer (32.3% oven-dried residue and 1.18  $\text{g}/\text{cm}^3$  density) and deionized water were also used for paste production.

### 3. Methods

The specific surface area of all mineral additions was assessed by means of BET method from adsorption of nitrogen gas analyses at 77 K. These experiments were conducted by using an ASAP 2020 physisorption analyser (Micromeritics, Norcross-USA) and all samples were prior degassed at 200 °C for 6 h under a vacuum of 0.1 mm Hg.

The soluble fraction of silica experiment consisted of 5.0 g of mineral addition in 100 mL of 10% NaOH solution at 90 °C for 6 h. After that, the solution with material was filtered (Whatman quantitative filter paper, Grade 589-WH), rinsed with 800 mL of deionized water, and subsequently oven-dried at 800 °C for 2 h. Finally, soluble silica fraction in each mineral addition was expressed by the quantity in percentage of material dissolved in the solution. This test was done using three replicate samples and the measurements were corrected by subtraction of mass loss of material submitted to the same drying protocol.

All pastes were produced at a constant water to cementitious material ratio (w/cm) of 0.35 with a constant superplasticizer content of 0.1% in relation to the mass of solid material and samples were prepared for the evaluation of isothermal calorimetry, thermogravimetry, and chemical shrinkage. Besides the reference mix (named P-REF), containing cement, water, and superplasticizer (premixed with the mixing water), six pastes were mixed with four different SCBAs, RHA, and quartz added on 20% of the mass of cement used in the reference based on previous studies [4–6,11,13,15]. These pastes were designated by P-SCBA $x$  ( $x$  equals to 1, 2, 3, and 4 in accordance with the SCBA type), P-RHA, or P-QTZ when each mineral addition was used. Each paste was mixed by adding solids to mixing water and superplasticizer in a 100-mL beaker and stirring with a spatula by hand for 30 s. After that, pastes were mixed using an electric hand mixer for more 2 min.

Isothermal calorimeter tests were performed at 25 °C  $\pm$  0.02 °C using an eight-channel conduction calorimeter (Tam Air TA Instruments, New Castle-USA). Duplicate samples containing approximately 10 g from each mix were put in 20-mL HDPE ampoules. Before mixing, all constituents were kept at 25 °C ambient for 24 h. A time lag existed between the contact of water, superplasticizer, and solid constituents, and ampoule putting into the calorimeter (typically 5 min). Data were recorded for a period up to 72 h of hydration.

The reactivity of different mineral additions was evaluated by means of thermogravimetry by using a STA7300 analyser (Hitachi, Tokyo-Japan). Immediately before each age of analysis, a sample of approximately 20 g of paste cured in a sealed plastic bag was ground using an agate mortar and pestle, and was placed into a platinum crucible in nitrogen atmosphere at a 100 mL/min flow rate. The samples were dried inside the analyser at 10 °C/min from 25 to 40 °C and 45-min hold time at 40 °C to eliminate the evaporable water. Then, another heating step was performed from 40 to 950 °C at 10 °C/min. Thermogravimetric tests were performed at 1, 3, 7, 28, 56, and 120 days. Differential thermogravimetry curves were used to determine the onset and the

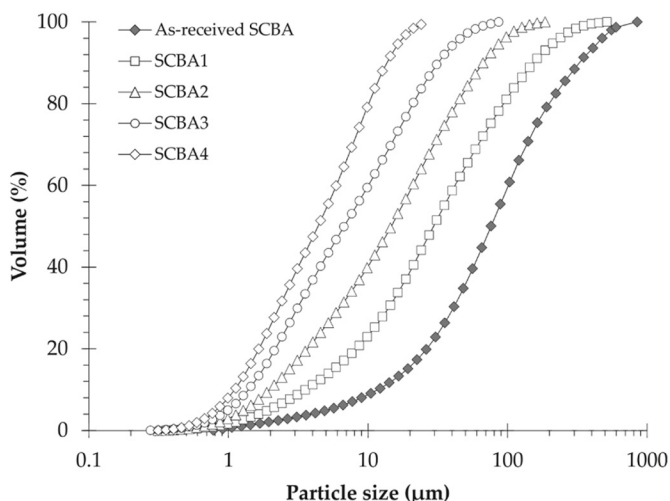


Fig. 1. Particle size distribution curves of different SCBA samples by light scattering analysis using a Mastersizer 3000E analyser (Malvern Instruments, Malvern-UK) considering Fraunhofer approximation model.

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