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Characterization of sugar cane bagasse ash as supplementary material for Portland cement

Caracterización de ceniza de bagazo de la caña de azúcar; como material suplementario del cemento portland

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ABSTRACT

Sugar Cane Bagasse is a by-product of the sugar agro-industry; it is partly used as fuel. However, bagasse ash (SCBA) is considered waste, which creates a disposal problem. Furthermore, if sugar cane bagasse is burned under controlled conditions, the SCBA can be potentially reused. This paper considers the technical viability of using SCBA as a partial replacement for cement. Two samples of SCBA from a Colombian sugar industry were characterized. The chemical composition of the samples shows high percentages of silica, 76.3% and 63.2%. The mineralogical and morphological characteristics of the waste were determined by X-ray diffraction patterns (XRD), thermal analysis (TG/DTA) and scanning electron microscopy (SEM). The pozzolanic activity of SCBA was evaluated using the Frattini test and the strength activity index test (SAI). The ASTM C618 defines an SAI of at least 75% as a requirement for classifying material as a pozzolan. This condition was achieved in the experiments performed. The results indicate that SCBA produced in the manufacture of commercial cements can be recycled for use as pozzolanic material. This supplementary material can partially replace cement and therefore reduce CO_2 emissions.

Keywords: Sugar cane bagasse ash, Portland cement, pozzolan, blended cement.

RESUMEN

El bagazo de caña es un subproducto de la agroindustria azucarera; el cual es utilizado particularmente como combustible. Sin embargo, la ceniza de bagazo (CBA) ha sido considerada como un residuo que causa problemas de disposición. Por lo tanto, si el bagazo de caña, fuera quemado bajo condiciones controladas, la CBA tendría un potencial para su re utilización. Este artículo considera la viabilidad técnica para el uso de la CBA como reemplazo parcial del cemento Portland; en los ensayos se caracterizaron dos muestras de CBA de una industria azucarera colombiana, en los cuales la composición química presentó altos porcentajes de sílice en un 76.3% y 63.2%. Las características mineralógicas y morfológicas fueron determinadas por las técnicas de difracción de rayos X (DRX), análisis térmico (TG/ATD) y microscopía electrónica de barrido (MEB). La actividad puzolánica de la SCBA fue evaluada utilizando el ensayo de Frattini y el índice de actividad por resistencia mecánica (IAR). La norma ASTM C618 define un índice IAR de al menos el 75% para clasificar un material como una puzolana; esta condición fue alcanzada en estos ensayos. Los resultados indican que la CBA puede ser reciclada como un material puzolánico en la fabricación de cementos comerciales así, dicho material suplementario puede reemplazar parcialmente el cemento y por lo tanto reducir las emisiones de CO_2.

Palabras clave: ceniza de bagazo de caña, cemento Portland, puzolana, cementos adicionados.

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Introduction

In any process carried out by commercial industries, principles on conservation and the use of natural resources must be taken into account. Therefore, it is necessary to consider a new approach to materials and production processes that is based on the reduction of natural resource extraction and the waste generated. "The cement industry is a key industry for sustainable development of any country, can be considered the backbone for development" (El-Haggar, 2007). With increasing industrial activities around the world, it is imperative to search for materials that can replace some of the current components in commercial cement, with oth-

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ers that have a similar chemical composition and that can reduce production costs without affecting the quality of the concrete (Chusilp et al., 2009a).

An important alternative found in research on this topic, is the use of an agro-industrial byproduct of the sugar industry, sugar cane bagasse ash (SCBA), as a mineral additive for cement. As a part the productive process of the sugar industry, sugar cane stalks are ground to extract the juice, the remaining fibrous residue, called bagasse, is one of the most valuable products in the sugar industry as its net calorific value is about 8000 kJ/kg (Batra et al., 2008). It is common practice to burn bagasse in boilers operating at temperatures around 700 °C and 900 ° C to produce steam and useful energy for the processes and needs of the factory (Souza et al., 2011). The efficiency of boilers used in the mills is typically 60-70% because the bagasse is significantly fibrous, has low density, high moisture and requires fuel such as coal for its combustion (Rasul, 2000). Thus, it contains high amounts of unburned carbon (Martirena et al., 1998; Chusilp et al., 2009). The ashes resulting from this process are characterized as highly pulverized material of low density and high volume. These features mean that if transported to landfills and dispersed in the environment, SCBA can contaminate air, soil, adjacent waters, and groundwater, resulting in effects to human health (Frias et al., 2011; Ortiz et al, 2009).

Recently, there have been several studies concluding that these ashes have a high reactivity towards lime and cement, supporting its use as a partial replacement for Portland cement or OPC (Chusilp et al., 2009; Cordeiro et al., 2008, 2009, 2012; Paya et al., 2002; Fairbairn et al., 2010; Martirena et al., 1998; Jimenez-Quero et al., 2013; Ganesan et al., 2007). SCBA can potentially replace cement for up to 20% (Ganesan et al., 2007; Cordeiro et al., 2009). The use of this material as an additive to produce clay bricks and roof tiles has also been reported (Souza et al., 2011; Faria et al., 2012). It should be noted that the chemical, mineralogical and pozzolanic properties of SCBA depend on the industrial process from which it has been obtained (Frias, 2011; Cordeiro, 2009a). Lower quality SCBA, particularly ash with high levels of unburned coal, are suggested to be used as inert material, especially as a replacement fine aggregate in concrete (Sales et al., 2010).

In 2010, Colombia harvested 20.3 million tons of sugarcane; by 2011 the production reached 22.7 million tons (ASOCAÑA, 2012). In Valle del Cauca, a sugar producing region in Colombia, there are about 13 mills that contribute to the domestic production of sugar. Such large volumes of processed sugarcane during the year generate considerable amounts of SCBA; the transformation of this waste and its proper use is a topic of current interest in sugarcane production regions.

Currently, approximately 70% of byproducts are disposed in landfills and only 30% is used for composting. According to the information provided by some sugar mills in the region, regional bagasse ash is not being used in the manufacture of products that benefit communities. This paper focuses on a preliminary study on the use of SCBA as a partial replacement for Portland cement (OPC). The study includes chemical and mineralogical characterization, and determination of the pozzolanic activity of two ash samples obtained from burning bagasse produced by the Colombian sugar production industry.

Experimental Procedure and Materials

The ashes used for the study were obtained from a sugar mill located in the Valle del Cauca, two samples were collected from the bottom of the multicyclone and precipitator of a boiler that works at temperatures between 600 °C and 900 °C. The samples will be referred to as SCBA1 and SCBA2. These samples presented a loss on ignition (LOI) of 10% and 14% respectively. ASTM C 618 limits the LOI of pozzolanic materials to less than 6%; a high LOI value is indicative of unburned carbon, a compound that interferes with the hydration reaction and increases the demand for water. This affects the final properties of the concrete (Chusilp et al., 2009). The difference in chemical composition, between the two samples, is the result of variability in the combustion process.

For both samples, a separation process of unwanted materials by sieving was applied, using sieves N° 140, 170 and 200. This reduced the LOI for SCBA1 and SCBA2 to 3.7% and 11% respectively. Table 1 shows the chemical and physical characteristics of the ash used, demonstrating that ash components with the highest percentages correspond to silica (SiO_2) and alumina (Al_2O_3), coinciding with reports by other authors (Payá et al., 2002, Rincón et al., 2010; Cordeiro et al., 2008). A high alkali content is also observed (see table 1), this limits the percentage of ash that can be added to the cement without compromising durability and causing problems such as alkali-aggregate reactivity or efflorescences. It should be noted that the alkali content in cement is limited to 0.6% (ASTM C150 standard); so it will be a factor to consider in future research.

Table 1. Chemical and physical characteristics of SCBA and cement (OPC)

Characteristics, (%)	SCBA1	SCBA2	OPC
SiO_2	72.8	61.3	19.4
Al_2O_3	6.4	5.6	4.0
Fe_2O_3	5.5	5.6	3.6
CaO	3.8	3.9	64.5
MgO	2.3	3.2	1.5
K_2O	2.7	5.0	0.4
Na_2O	1.2	0.9	0.3
Loss on Ignition	3.7	11.0	2.6
Average Particle Size (μm)	79.8	41.5	16.1

The average particle size was determined for the two ash samples in a Malvern granulometry Mastersizer Laser 2000; the results are presented in Table 1 and the particle size distribution is shown in Figure 1. It is appreciated that SCBA1 presents an average particle size of about two times that of the corresponding figure for SCBA2.

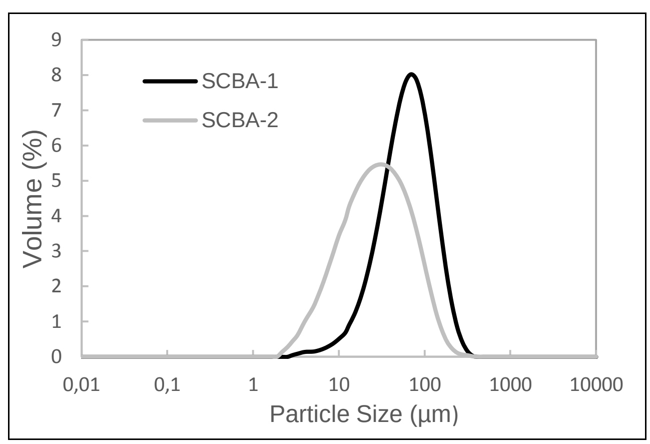

Figure 1. Particle size distribution of SCBA

The mineralogical and morphological characteristics were determined by X-ray diffraction (XRD), thermal analysis (TG / DTA) and scanning electron microscopy (SEM).

To determine the viability of using the SCBA as an addition to OPC, the pozzolanic activity of SCBA was evaluated using the Frattini test and the strength activity index test (SAI). The Frattini test is a commonly used direct method that involves chemical titration to determine the dissolved Ca^{2+} and OH_{-} concentrations in a solution containing OPC and the test pozzolan. SAI is an indirect testing method that measures the effect of the test pozzolan on the compressive strength of the blended mortar. The Frattini test was carried out according the procedure described by Colombian Standard NTC 1512. The pozzolanic activity index was evaluated according to ASTM C311.

Using the Frattini method (NTC 1512), test samples (20 g) were prepared consisting of 80% OPC and 20% of the test pozzolan and mixed with 100 ml of distilled water. These samples were left for 7 and 28 days in sealed plastic bottles in an oven at 40°C. After each time cycle, samples were vacuum filtered. The filtrate was analyzed for $[OH^{-}]$ and $[Ca^{2+}]$.

The ASTM C311 standard indicates that mortar mixtures should be elaborated with 2.75 parts of sand for 1 part of cement with a water to cement ratio of 0.485, which can be modified to achieve a flow of 110 ± 5%. These parameters were used in order to prepare the reference sample (without SCBA). To ensure that the flow of the blended mortar mixture (SCBA 20%) complies with the flow set by the standard, the water ratio was fixed at 0.56. The characteristics of the OPC used are shown in Table 1. The samples were cured by submersion in an aqueous solution of $Ca(OH)_2$ at room temperature for 7 and 28 day periods. Having completed these periods, the samples were tested for compressive strength to calculate the SAI.

Results and discussion

Analysis by X-ray Diffraction (XRD)

A study of the mineralogical composition of the SCBA through the X-ray Diffraction technique was carried out. The equipment used for this analysis was an X'Pert Pro defractometer with an X-Ray tube containing a copper anode, a scan rate of 5 ° / min, step of 0.020 degrees, range at 3.0 to 70.0 degrees, voltage at 40kV and current at 30 mA. Figure 2 shows the corresponding diffractograms.

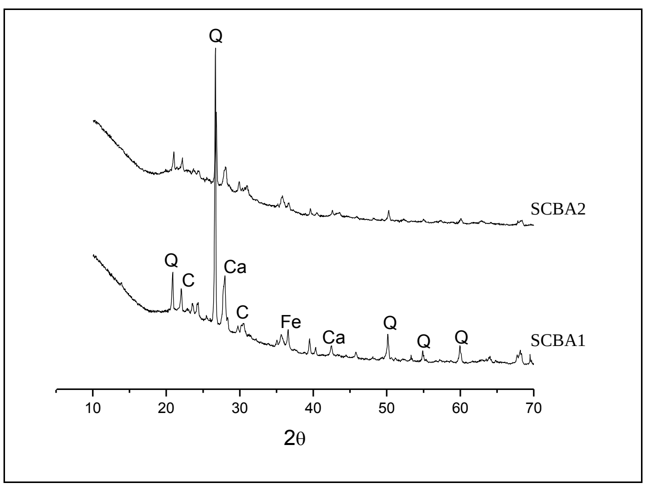

Figure 2. X-ray diffractograms. Q: Quartz, C: Cristobalite, Ca: Calcite, Fe: Ferric Oxide (Fe_2O_3)

Figure 2 shows the XRD patterns with a broad band or lifting of the baseline between 15 and 35 (2Θ), indicating the presence of amorphous or vitreous materials in both SCBA samples. This amorphous character can contribute significantly to the pozzolanic activity of the material to be added to OPC. The presence of cristobalite was confirmed in the range 10-30 (2Θ) for both cases, indicating that the combustion temperature of the bagasse was higher than 800 °C, coinciding with findings by different authors (Payá et al., 2002; Morales et al., 2009; Chusilp et al., 2009; Cordeiro et al., 2009a, 2012; Frías et al., 2007, 2011).

For SCBA samples, quartz is observed as the main crystalline compound as shown by the XRD pattern (see Fig. 2) and confirmed by chemical analysis (see Table 2). The presence of quartz can be attributed to the sand adhering to the sugarcane at the time of harvest, due to the characteristics of soil origin. This is true even after the sugarcane has been washed. Cordeiro (2008) reports that even after washing the harvested sugarcane, sand can represent as much as 2% in weight of the material that is processed and that after the calcination of organic material during the burning of the bagasse, the proportion of quartz increases considerably (Cordeiro et al., 2009). Other minor compounds such as iron oxides and calcite were also detected (Morales, 2009).

Thermogravimetric analysis (TG/DTA)

Thermal analysis (TG/DTA) of samples was performed on a TA Instruments Model SDT Q-600 at a heating rate of 10 °C/min in a nitrogen atmosphere. Figure 3 shows the weight loss curves (TG) and thermal analysis (DTA). The figure demonastrates that SCBA1 has the lower weight loss of the two samples, as temperature increased to 1000°C, 3.6 and 11.2 % respectively. This is due to lower LOI as seen in the chemical composition listed in Table 1.

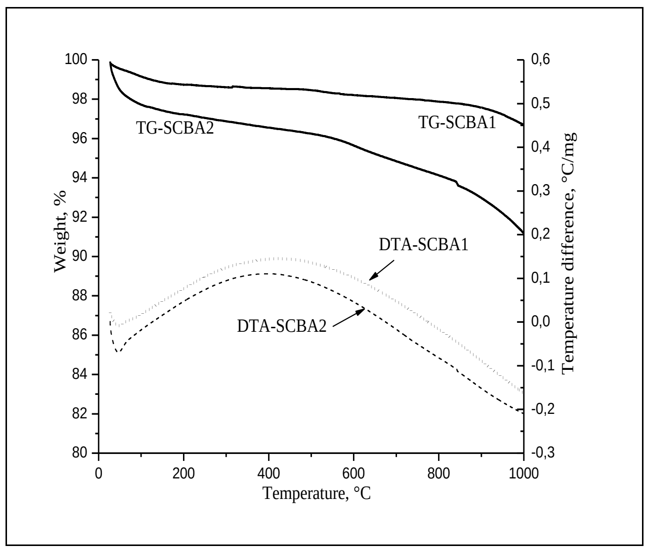

Figure 3. Thermal Analysis for SCBA1 and SCBA2 under inert condition (nitrogen gas)

In the case of the DTA, the figure shows a single endothermic peak at around 50°C, which corresponds to loss of water absorbed by the samples. Some authors, including Frias et al. (2007, 2011), have reported the presence of endothermic peaks associated with compounds such as carbonates (between 600-700°C), kaolinite (≅ 500-650°C) or gibbsite (≅ 250°C). In this study, these compounds are not observed; this is in accordance with the results obtained by XRD. It is important to indicate that the possible organic matter present in bagasse ash is not detected by this technique because the thermal analysis presented in Figure 3 was carried out under inert conditions (nitrogen gas). For this reason, a thermal test was performed in an oxygen atmosphere, the results are presented in Figure 4. In this figure, the presence of an exothermic peak located at approximately 430 °C is observed, which corresponds to the organic matter (unburned carbon) present in the SCBA samples. TG curves indicate that the weight loss takes place mainly between 300 and 500 °C.

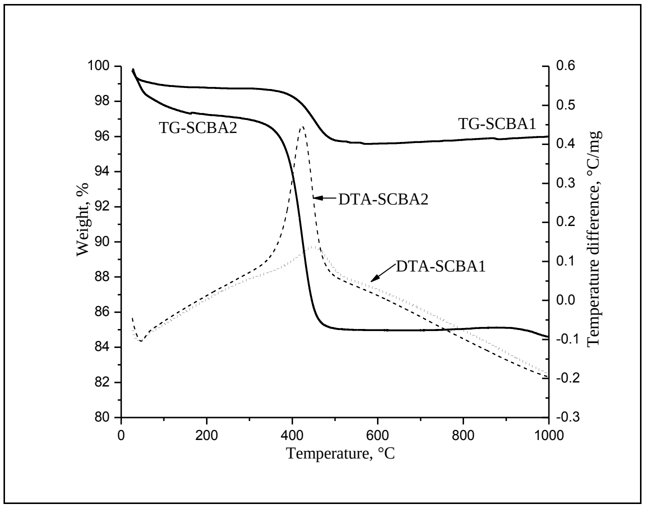

Figure 4. Thermal Analysis for SCBA1 and SCBA2 under oxygen

Scanning Electron Microscopy (SEM)

The samples were observed using the SEM technique on a FEI QUANTA 200 device. Figure 5 shows the corresponding images. These images show different morphologies, with particles of different shapes and sizes. The presence of coarse and porous particles is observed; these are typical organic materials present in SCBA. In general, prismatic, spherical and fibrous particles are shown. These morphologies are similar to those observed by Paya et al. (2002), Cordeiro et al. (2009) and Batra et al. (2008). These figures confirm that the particle size of SCBA1 is greater than SCBA2, which is in accordance with the data reported by laser granulometry (see Table 1).

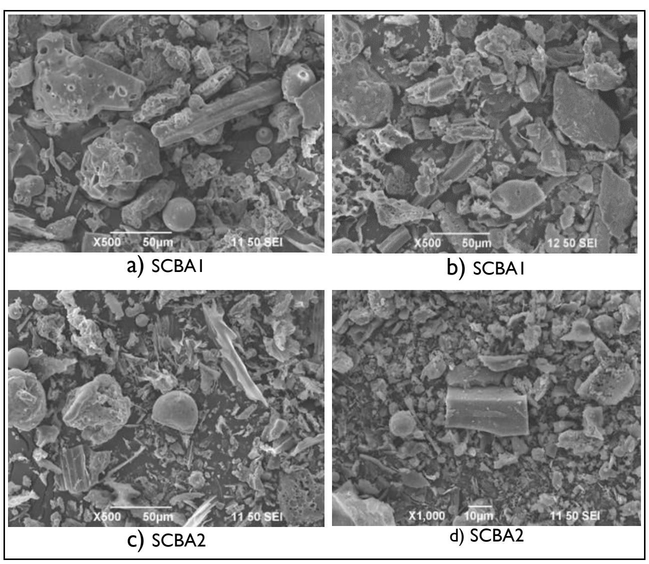

Figure 5. SEM images: SCBA1 and SCBA2.

Pozzolanic Activity

According to Massazza (2003), the term 'pozzolanic activity' considers all reactions occurring among the active constituents of a pozzolan, lime (released by the OPC hydration) and water. This concept includes the maximum amount of lime that a pozzolan can combine with and the rate at which such combination occurs. The overall amount of combined lime depends essentially on the nature, quality and quantity of the active phases present in the pozzolan sample and the lime combination rate depends on the particle size and specific surface area of the pozzolan, the water/solid ratio and the curing temperature.

The Frattini test can accurately define the pozzolanic activity of blended OPCs measuring the CH (Portlandite) consumption released during OPC hydration. In this sense, the Frattini test compares [Ca^{2+}] and [OH^-] contained in the aqueous solution that covers the hydrated sample at 40°C for a given time (7 and 28 days) with the solubility curve for CH in an alkaline solution at the same temperature. The Frattini test results are presented in Figure 6 as points in the graph with axes values representing [Ca^{2+}] versus [OH^-]. In this graph, the calcium isothermal curve demarcates the pozzolanic area below this curve and the non-pozzolanic area above it. At 7 days, the results of the cement pastes containing SCBA1 are above the solubility curve and at 28 days are lying closely below the solubility line indicating its poor pozzolanic activity. By contrast, the results for SCBA2 are below the solubility line, this indicates that this material presents higher pozzolanic activity than SCBA1. This may be because the SCBA1 sample has a larger particle size than SCBA2; it is also observed by XRD, that the SCBA1 contains more crystalline material.

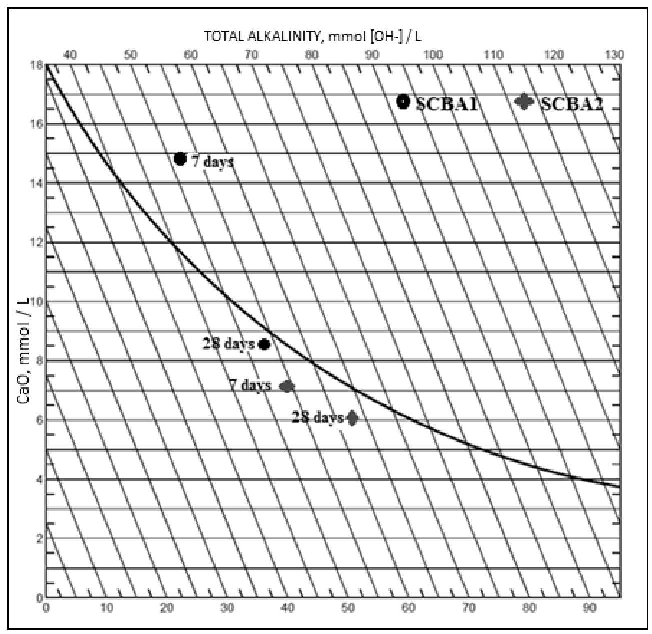

Figure 6. Frattini test results for SCBA1 and SCBA2, after 7 and 28 days of curing

The calcium concentration at 7 and 28 days may be compared to the theoretical [CaO] and the reduction is expressed as a percentage of the theoretical maximum removed, as is seen in Table 2. These results indicate that the pozzolanic activity of SCB2 is higher than that of SCBC1. The negative value in the table (% reduction [CaO]) is associated to the position above the solubility curve, indicating a non-pozzolanic performance of SCB1 at 7 days of curing. In this case, it can be said that although the material performs as such, the reaction rate is very slow.

In order to quantify the results from the Frattini tests, it is possible to use the equation (Eq. 1) mentioned in EN 196 standard (Donatello et al., 2010):

$$\text{Max [CaO]} = 350/([OH^-] - 15) \quad (1)$$

Table 2. Frattini test results at 7 and 28 days of curing

Sample	7 days				28 days			
	[OH-] mmol/L	[CaO] mmol/L	Theoretical max. [CaO] mmol/L	[CaO] Reduction%	[OH-] mmol/L	[CaO] mmol/L	Theoretical max. [CaO] mmol/L	[CaO] Reduction%
SCBA1	52.21	14.72	9.40	-56.50	52.82	8.52	9.25	7.89
SCBA2	54.04	7.06	8.96	21.20	62.36	5.95	7.39	19.48

The calcium concentration at 7 and 28 days may be compared to the theoretical [CaO] and the reduction is expressed as a percentage of the theoretical maximum removed, as is seen in Table 2. These results indicate that the pozzolanic activity of SCB2 is higher than that of SCBC1. The negative value in the table (% reduction [CaO]) is associated to the position above the solubility curve, indicating a non-pozzolanic performance of SCB1 at 7 days of curing. In this case, it can be said that although the material performs as such, the reaction rate is very slow.

Figure 7 presents the results of the pozzolanic activity index (SAI) test at 7 and 28 days of curing for the heat-treated samples of SCBA. Both ash samples show similar strength activity index (SAI = 65%) at 7 days, counter to the results attained using the Frattini test. In general, SCBA1 y SCBA2 reported high levels of pozzolanic activity at 28 days of curing, 91 y 97 % respectively; these values exceed those reported by other authors, which have presented an index of about 83% (Oliveira et al., 2010). Lin et al. (2012) report a SAI of 104% at 91 days of curing. It should be noted that by this test, no significant effects are seen between the two samples due to their remarkable difference in the average particle size. ASTM C618 standard requires a SAI greater than 0.75 (75%) for fly ash and natural pozzolans at a replacement of 20%. By this standard, the SAI value of the SCBA samples at 7 days or 28 days indicatespecification compliance. In this sense, the ashes studied have potential use as an addition to Portland cement.

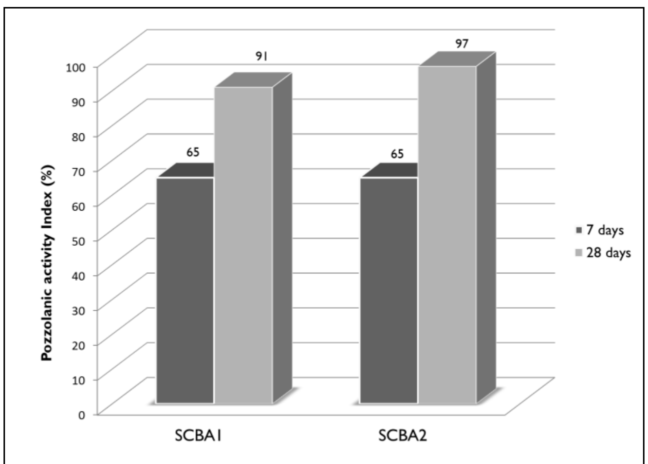

Figure 7. Pozzolanic activity index (SAI) for thermal treated SCBA

Conclusions

According to the results of this study, the following conclusions can be asserted:

The bagasse ash studied has high silica content and significant amorphous content, making them subject to consideration as supplementary material for partial replacement of Portland cement (OPC).

The pozzolanic activity index (SAI) of SCBA exceeds the minimum specification set by ASTM C618 and those reported by other researchers for samples 28 days cured; however the Frattini test demonstrates the importance of particle size, suggesting that mill processing of the material is required.

It is worth noting that 20% replacement of OPC by SCBA with a particle size of 41 microns, allows for a cementitious material with mechanical properties similar to cement starting.

In conclusion, SCBA shows potential as a pozzolan, yet more studies are recommended in order to evaluate the optimal proportion of addition, the rheological effect on the cement mixture and the long-term performance of blended cement in terms of its mechanical and durability properties.

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