# The effect of calcination conditions on the physical and chemical characteristics of sugar cane bagasse ash

## *Influência das condições de queima nas características físico-químicas das cinzas de bagaço de cana-de-açúcar*

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## Abstract

*The effect of calcination temperature and air flow on the content of organic material, morphology of particles, degree of crystallinity and the reactivity with lime solution of the sugar cane bagasse ash is evaluated. The results show that the long fibers of the bagasse and organic material are retained when calcination occurs without sufficient air flow. Calcining with forced air-flow breaks the fibers, removes organic material and produces fine particles at a temperature of 600°C. The non-organic material observed in the ash displays a high degree of crystallinity. Experiments show that the crystalline structure observed in the ashes is due to adhered sand which was not previously washed away. The reduction on the conductivity in lime solution and X-rays diffraction pattern suggest that amorphous silica is formed at temperatures lower than 600ºC and cristobalite is formed at higher temperatures.*

***Keywords:*** *Sugar cane bagasse ash, pozzolanic activity, mineral admixture*

## Resumo

Esse estudo avalia a influência das condições de queima do bagaço de cana-de-açúcar (BCA) nas características físico-química das cinzas geradas. A queima foi realizada nas temperaturas de 400, 600 e 800ºC com e sem circulação de ar no forno. As cinzas obtidas após a queima foram caracterizadas de acordo com o grau de cristalinidade, morfologia, análise granulométrica, ensaios de perda ao fogo e de reatividade em solução saturada de hidróxido de cálcio. Foi observado que a queima com circulação de ar desfaz as fibras, consome o material orgânico e produz partículas finas a uma temperatura de 600°C. O material não orgânico observado nas cinzas apresenta alto grau de cristalinidade. Ensaios em material lavado em laboratório sugerem que a estrutura cristalina observada nas cinzas é areia proveniente da contaminação do solo. A redução na condutividade em uma solução de hidróxido de cálcio e a difração de raios X sugerem que a sílica amorfa é formada em temperaturas inferiores a 600°C e a cristobalita é formada em temperaturas mais elevadas.

**Palavras-chave:** Cinzas de bagaço de cana-de-açúcar, atividade pozolânica, adições minerais.

## 1. Introduction

Sugar-cane bagasse ash (SCBA) is a by-product from the energy co-generation process in the sugar-cane industry. This material is commonly used as a fertilizer, which is not an adequate use due to its high silica content and lack of nutrients. It is thus important to promote another destination for SCBA.

Earlier experiments on rice husk ashes showed that this material exhibits pozzolanic activity (Deepa et al., 2008). It can be anticipated that SCBA might also display a pozzolanic activity due to its high $SiO_2$ content. Therefore, many recent papers have studied the potential pozzolanic properties of the sugar-cane bagasse ashes (Singh et al., 2000; Martinera Hernandez et al., 1998; Payá et al., 2002). Some papers report pozzolanic activity for SCBA (Singh et al., 2000; Martinera Hernandez et al., 1998; Payá et al., 2002). However, some publications report that the chemical or physical characteristics of SCBA are not typical of pozzolanic materials. For example, high unburned carbon content has been reported (Martinera Hernandez et al., 1998; Payá et al., 2002), as well as a high degree of cristallinity of the silica (Martinera Hernandez et al., 1998) and large particle sizes (Cordeiro et al., 2009b).

Experiments have shown that milling conditions affect the pozzolanic properties of SCBA (Cordeiro et al., 2009b; Cordeiro et al., 2008). Recent papers have studied the effect of calcination conditions on the physical, chemical and pozzolanic characteristics of SCBA, but these studies have focused mainly on the temperature (Cordeiro et al., 2009a). It has been shown that ashes burned at different temperatures in an industrial furnace and ashes burned in laboratory exhibit different morphology and pozzolanic activity (Frías et al., 2001). A systematic study has evaluated the influence of the calcinating temperature and has shown that ashes with pozzolanic activity can be produced at an optimum temperature of 600°C. High carbon and volatile compounds were observed at lower temperatures, whereas crystallization of silica into cristobalite resulted from processing at higher temperatures (Cordeiro et al., 2009a). Thus, it is expected that controlling the calcination conditions will improve the properties of SCBA.

SCBA with pozzolanic activity can be produced in laboratory, but experiments with SCBA produced in boilers of the sugar industry revealed a high degree of crystallinity of the silica present and presence of unburned material, which compromises the reactivity of this material (Martinera Hernandez et al., 1998). This discrepancy suggests that calcination conditions play a key role on the development of pozzolanic activity in SCBA. Therefore, the present paper aims at studying the effect of calcination conditions on some chemical and physical properties of SCBA. Focus is on the parameters that might lead to unburned material on the final product and the high degree of cristallinity of silica. It is already known that calcination temperature will affect these characteristics, so experiments were carried out at different temperatures. However, other parameters such as the air-flow during calcination and previous removal of dust by washing the bagasse are also evaluated.

## 2. Experimental procedure

The material used in this study was the sugar-cane bagasse provided by Usina Alcooleira Planalto (Ibiá / Brazil). The bagasse was dried at 60°C for 24 hours in an oven and 90 g portions were calcinated in laboratory at 400°C, 600°C and 800°C. The temperature range for calcination was based on literature (Cordeiro et al., 2009; Morales et al., 2009; Frías and Villar-Cociña, 2007). Calcination was carried out using an electric oven with ~$6.3 \times 10^{-3}$ $m^3$ volume. The heating rate was ~10°C/min, the material was held for 120 min at the temperature and cooled inside the furnace. Experiments were carried out with an enclosed furnace and repeated with an air-pump attached to the furnace in order to force air flow inside the chamber. After cooling, the ashes were analyzed without any milling.

Thermogravimetric analysis was performed using DTG-60 equipment. The morphology of the SCBA was analyzed with a scanning electron microscope (SEM) model Quanta 200F, operating at 15 kV. The carbon content was determined by loss on ignition (LOI) and the chemical composition was determined by X-ray fluorescence. The particle size distribution was determined with a laser diffractometer (model Cilas 1064). The crystalline characteristics were analyzed by X-ray diffraction pattern using Philips PW1710 equipment. CuKα radiation at angular speed of 0.06°/s, over a range of 4°-90° was used. The reactivity of some of the SCBA was determined indirectly by the variation in resistivity in lime saturated solutions, employing a method proposed by Luxan et al., 1989. In this test the variation in conductivity is measured 2 minutes after 5g of the sample material is poured into a lime saturated solution. The solution is kept at 40°C during the test. The larger the variation in conductivity, the more reactive is the material.

## 3. Results

The thermogravimetric analysis of the sugar-cane bagasse is presented in Figure 1 and shows the evolution of the mass as a function of the temperature. It shows that the as-received material loses ~48% of its mass up to a temperature of ~78°C. This is attributed to the humidity of the bagasse. This pronounced loss of mass at low temperature is not observed in the oven-dried material. The evolution of the mass as a function of the temperature is fairly similar in the as-received and in the oven-dried material at higher temperatures. Both exhibit a significant loss of mass between ~250°C and ~360°C followed by a less pronounced loss of mass up to ~530°C in the as-received and ~600°C in the oven-dried material. This loss of mass is attributed to organic matter. The final loss of mass is ~98.5% in the as-received and ~94.3% in the oven-dried material.

The loss on ignition (LOI) and the chemical composition of the sugar-cane bagasse and sugar-cane bagasse ash are summarized in Table 1 for the various calcination conditions studied. It is observed that the bagasse has a high loss on ignition, which decreases with calcination. However, LOI depends on the calcination condition. Higher values are observed after calcination in an enclosed furnace, without forced air flow. For example, loss on ignition over 70% is still observed after calcination at the highest temperature (800°C) in an enclosed furnace while a value of ~2% is observed after calcining at the same temperature in a furnace with forced air flow. It is also observed that increasing calcining temperature tends to reduce LOI.

The chemical composition of SCB and SCBA calcined at various conditions show good agreement, within experimental error. The $SiO_2$ content is typically over 70% of the inorganic matter. The other main components are $Fe_2O_3$, $K_2O$, $Al_2O_3$, CaO and $P_2O_5$.

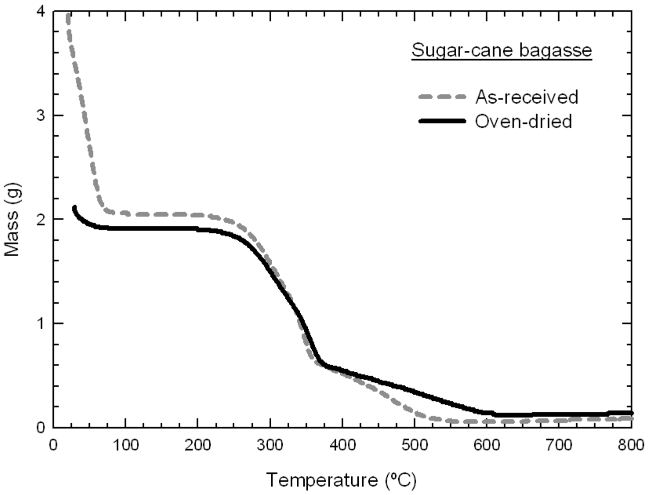

Figure 1
Evolution of mass of Sugar Cane Bagasse as a function of the temperature determined by TGA.

Table 1
Loss on ignition and chemical composition of the sugar-cane bagasse (SCB) and sugar-cane bagasse ash (SCBA) calcinated at various conditions.

| | Calcination condition | | LOI (%) | Composition (%) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Forced Air Flow | Temp. (ºC) | | $SiO_2$ | $Fe_2O_3$ | $K_2O$ | $Al_2O_3$ | CaO | $P_2O_5$ | MgO | $TiO_2$ | $Na_2O$ | MnO |
| SCB | - | - | 94.34 | 4.36 | 0.67 | 0.2 | <0.10 | 0.26 | 0.05 | <0.10 | 0.02 | <0.10 | <0.01 |
| SCBA | No | 400 | 88.8 | 7.79 | 0.47 | 0.97 | 0.37 | 0.42 | 0.23 | <0.10 | 0.06 | <0.10 | 0.02 |
| | | 600 | 73.82 | 20 | 2.35 | 1.23 | 1.53 | 0.61 | 0.3 | <0.10 | 0.18 | <0.10 | 0.02 |
| | | 800 | 74.94 | 15 | 5.42 | 1.22 | 0.7 | 0.59 | 0.29 | <0.10 | 0.19 | <0.10 | 0.04 |
| | Yes | 400 | 41.32 | 44.2 | 2.95 | 4.27 | 2.66 | 1.98 | 1.07 | 0.76 | 0.32 | <0.10 | 0.09 |
| | | 600 | 3.97 | 71.8 | 5.8 | 6.91 | 4.85 | 3.38 | 1.86 | 1.46 | 0.51 | 0.16 | 0.13 |
| | | 800 | 2.27 | 74.47 | 6.78 | 5.67 | 4.43 | 3.24 | 1.56 | 1.27 | 0.51 | 0.2 | 0.13 |

Figure 2 shows the cumulative and frequency distribution of particle size for SCBA produced at different calcination conditions. It is observed that both the temperature and air flow affect the particle size distribution. The cumulative distribution shows that a proportion of finer particles is observed in the SCBA calcined at 600°C with forced air flow while a larger proportion of coarser particles are observed in the samples calcined at 400°C and 800°C without forced air flow.

The frequency distribution of particle size shows that most SCBA display a peak of distribution in the range of tens of microns (~40-80 mm). However, SCBA produced at 600°C with forced air flow exhibits another peak in distribution at ~15 μm. This new peak in distribution, at lower particle size, might lead to the observed larger proportion of finer particles in this calcinating condition.

The X-ray diffraction intensity of the SCBA calcined under different conditions is presented in Figure 3 as a function of 2θ. The curves of the material produced in the enclosed furnace are shown on the top and the material produced with forced air flow at the bottom. Increasing calcining temperatures are presented from top to bottom. It is observed that the ashes produced in the enclosed furnace at 400°C and 800°C exhibit almost no diffraction peaks which indicates the presence of a high volume of amorphous material and impurities. The material calcined at 600°C without forced air flow exhibits small diffraction peaks characteristic of quartz. Some diffraction peaks are observed at 2θ of ~37°, ~44°. ~65° and a strong peak at ~78°. These peaks could not be identified by the diffraction profile of conventional components of SCBA. The material produced by calcination with forced air flow exhibits pronounced diffraction peaks, which agree with the diffraction pattern of quartz. Minor peaks indicate the presence of cristobalite in the SCBA produced at 800°C.

The morphology of the ashes produced at various calcination conditions were analyzed by scanning electron microscopy and some details are shown in Figure 4. The morphology of the ashes calcined in the enclosed furnace or with forced air flow is clearly different. Calcination at different temperatures in the enclosed furnace leads to long fibers (hundreds of microns long), typical of the as-received material. This suggests that calcination in the enclosed furnace is not effective for breaking-up the long fibers of the bagasse. The material calcined at different temperatures under forced air flow displays features in the range of tens of microns indicating an effective break-up of the original long fibers.

As the SCBA, calcinateded at a low temperature (400°C) and without forced air flow, exhibits a large content of unburned matter and impurities, the tests of variation of conductivity in saturated lime solutions were only carried out in the samples calcined at 600°C and 800°C with forced air flow. A reduction of 1.0 mS/cm in the sample calcined at 600°C was observed and no variation was observed when the material calcined at 800°C was poured in the solution.

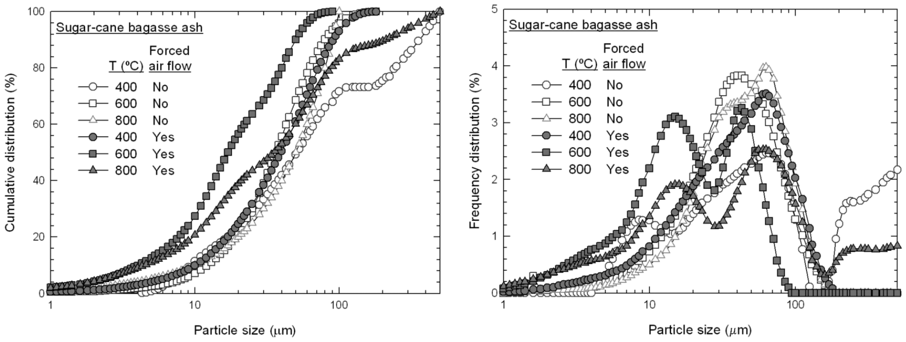

Figure 2
Cumulative and frequency distribution of particle size of SCBA calcined under different conditions.

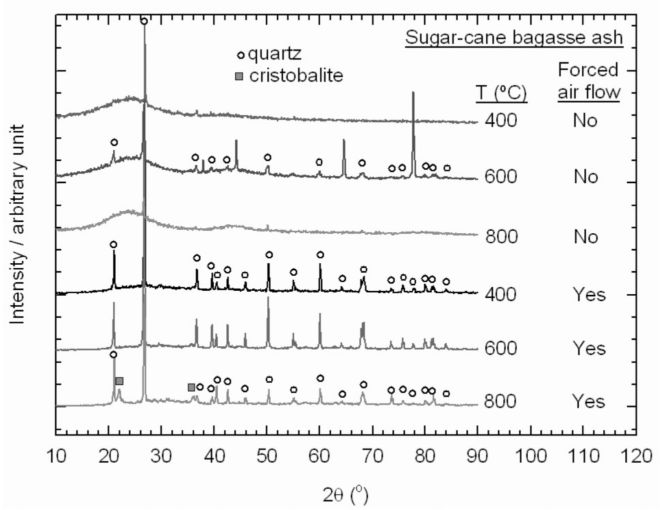

Figure 3
X-ray diffraction pattern of the SCBA calcined under different conditions.

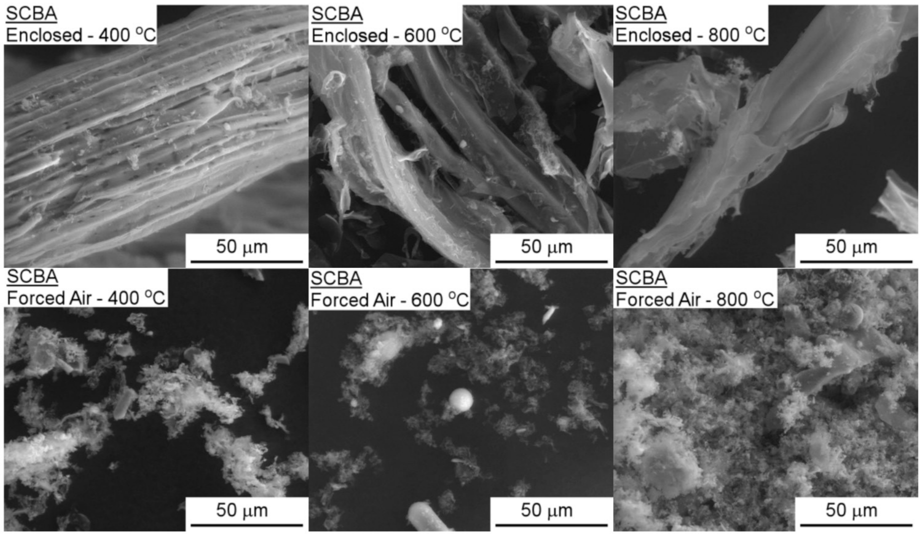

Figure 4
SEM images of the SCB and SCBA calcined under different conditions.

## 4. Discussion

The relation between the present results and the pozzolanic potential of SCBA are discussed below. The analysis will focus on the organic content, crystalline structure, morphology and chemical reaction.

### Organic content

The TGA analysis showed that over 98% of the mass of the sugarcane bagasse is composed of water and organic matter, which are lost during heating. The oven-dried bagasse exhibits ~94% of organic matter. A significant amount of organic matter is still present in the ashes produced by calcining at low temperature (400°C) or by calcinations without forced air flow. As the amount of oxygen inside the furnace is not sufficient for complete combustion of the organic matter, calcination with the enclosed furnace becomes a carbonization of the bagasse. A significant amount of organic matter was observed in SCBA collected from industry (Payá et al., 2002). Thus, increasing calcination temperatures and/or increasing air flow are indicated in order to produce SCBA with low unburned content.

A recent paper (Cordeiro et al., 2009a) reports significant loss on ignition in SCBA calcined up to 500°C which is in agreement with the present findings for the material produced with forced air flow. Therefore, calcining temperatures of ~600°C or higher are suggested in order to produce SCBA with low organic content.

### Morphology

Analysis of the particle size distribution shows that the material calcined at a low temperature (400°C) without forced air flow exhibits a high content of large particles. This is attributed to the difficulty in breaking-up the fibers from the bagasse. Long fibers are observed in the material calcined without air flow (Figure 4) and were reported in the literature for industrial ashes (Payá et al., 2002). Further milling is suggested in order to reduce the SCBA particle size and improve pozzolanic properties (Cordeiro et al., 2009b; Cordeiro et al., 2009a).

It is also observed that the particle size tends to decrease when calcining temperature increases from 400°C to 600°C (Figure 2) under forced air flow, but the particle size increases with further increase in temperature to 800°C. This increase in mean particle size at the higher temperature is believed to be caused by the agglomeration of the particles.

A volume fraction of more than 66% of particles smaller than 45 μm is required for a pozzolanic material, according to ASTM C-618 (2005). The only calcining condition in which such requirement was fulfilled in the present study was at 600°C with forced air flow. SCBA calcined at the same temperature and without forced air flow exhibits ~63% of volume of particles smaller than 45 μm, which is only slightly smaller than the requirement. This shows that the calcining temperature plays a key role on the particle size distribution and that the optimum temperature is ~600°C. The material calcined with forced air flow tends to exhibit a finer particle size than their counterparts calcined in an enclosed furnace, which shows that the air flow is also important for control of particle size distribution. Further milling of SCBA is indicated in order to fulfill the requirement of the ASTM C-618 (2005) standard.

### Crystalline structure

It is known that an amorphous structure is desired in order to achieve the best pozzolanic properties (Mehta and Monteiro, 2006). Amorphous structures are characterized by a continuous distribution of intensities in the X-ray diffraction profile, without pronounced peaks. This is observed in the material calcined at 400°C and 800°C in the enclosed furnace (see Figure 3). The material calcined at 600°C exhibits small peaks, suggesting the presence of some crystalline content. However, the high content of organic matter (>70%) in SCBA calcined without forced air flow makes it difficult to analyze the non-organic content. SCBA crystallinity is best evaluated by the material with low organic content (calcined with forced air flow) and the present results clearly display pronounced peaks characteristic of quartz for all temperatures, which were also observed in industrial SCBA (Payá et al., 2002). This suggests that the $SiO_2$ present in SCBA is mostly crystalline.

However, it is worth noting that earlier experiments on X-ray diffraction of SCBA also showed peaks, suggesting the presence of crystalline quartz, but an analysis of the diffraction pattern revealed the presence of up to ~24% of amorphous material (Cordeiro et al., 2008). The authors suggested that the presence of crystalline quartz in SCBA is due to adhered sand from the soil that was not washed away prior to burning of the bagasse. Experiments conducted in bagasse washed in the laboratory revealed the formation of amorphous material, without peaks of quartz, in SCBA calcinated up to 600°C (Cordeiro et al., 2009a). In order to clarify the source of the crystaline peaks observed in X-ray diffraction SCBA was carefully washed in laboratory prior to calcination in order to remove adhered sand and dust. This SCBA was dried and calcined at the optimum condition of 600°C with forced air flow. Figure 5 shows the x-ray profile of the SCBA produced after washing. The diffraction profile of the as-received, unwashed, SCBA calcinedunder similar conditions is also shown for comparison. It is observed that the crystalline peaks decrease significantly confirming that adhered sand and dust are the major sources of the crystalline peaks observed in the as-received material.

Calcining at a higher temperature leads to the formation of crystalline $SiO_2$. However, the peaks in the X-ray diffraction are attributed to cristobalite. This suggests that the $SiO_2$ forms as amorphous material during calcinations up to 600°C and as cristobalite at higher temperatures. This is in agreement with our results as peaks of cristobalite were observed after calcining at the higher temperature of 800°C. Moreover, a decrease in conductivity in saturated lime solutions due to SCBA calcined at 600°C was observed, which suggests high reactivity for this material. Constant conductivity was observed when pouring SCBA calcined at 800°C which suggests that this material has low reactivity. Thus, the results on the variation of conductivity in saturated lime solution is in agreement with the suggested formation of amorphous $SiO_2$ up to 600°C and crystalline $SiO_2$ at higher temperature.

Earlier papers reported different results concerning the formation of crystalline (Payá et al., 2002; Cordeiro et al., 2008) or amorphous structures in SCBA (Ganesan et al., 2007). A recent paper (Cordeiro et al., 2008) reported peaks of quartz and cristobalite in the SCBA similar to the present results. However, quantitative analysis of the X-ray diffraction pattern indicated ~24% of amorphous material. The authors (Cordeiro et al., 2008) attributed a large fraction of the crystalline material in SCBA to the presence of unwashed sand from the soil. Later it was shown that amorphous $SiO_2$ could be produced from controlled calcination of laboratory-washed SCBA (Cordeiro et al., 2009a).

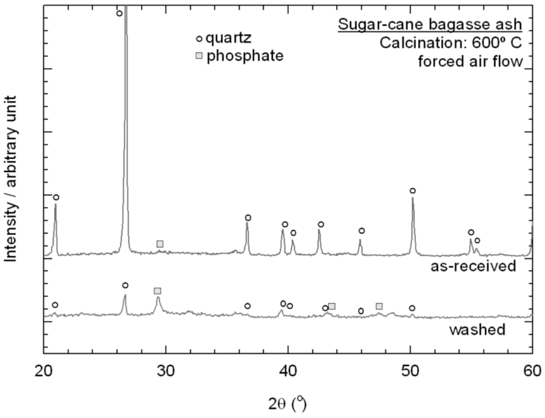

Figure 5
X-ray diffraction pattern of the as-received and laboratory washed SCB.

## Pozzolanic activity potential

The present results are summarized in Table 2. The SEM analysis showed that long fibers are present in the material calcined in the enclosed furnace while the fibers are broken into particles within the range 1 to 100 µm when calcined with forced air flow. There is a high content of organic matter in the material calcined in the enclosed furnace which leads to an amorphous-like X-ray diffraction. The material calcined at high temperature and with forced air flow exhibits low content of unburned material and peaks of crystalline quartz which are attributed to the presence of unwashed sand in the bagasse. Calcination at the highest temperature (800°C) leads to crystallization of the $SiO_2$ and formation of cristobalite, which reduces the reactivity of the material. Thus, the optimum calcination condition is 600°C with forced air flow. The preliminary washing of SCBA is suggested in order to remove crystalline material and to increase the proportion of highly reactive amorphous $SiO_2$. The latter has a potential to be used as a pozzolanic addition to cement based composites. It is worth noting that the SCBA was not milled during the present experiments. The pozzolanic effect was found to increase with fineness of the particles (Cordeiro et al., 2009b) and therefore it is expected that the pozzolanic properties of the present material will improve with grinding.

| Calcination | | Results | | | |
|---|---|---|---|---|---|
| Forced Air | Temp. (°C) | Particle morphology | LOI | X-Ray | Conductivity in saturated lime solution |
| No | 400 | Long fibers | High | Amorphous | - |
| | 600 | Long fibers | High | Semi-amorphous | - |
| | 800 | Long fibers | High | Amorphous | - |
| Yes | 400 | 1 – 100 µm | Moderate | Crystalline | - |
| | 600 | 1 – 100 µm | Low | Crystalline | Pozzolan |
| | 800 | 1 – 100 µm | Low | Crystalline | Non-pozzolan |

Table 2
Summary of results of particle morphology, loss on ignition, X-ray diffraction pattern and conductivity in saturated lime solution of SCBA calcinated at different conditions.

## 5. Summary and conclusion

Sugar-cane bagasse ash (SCBA) was produced in controlled calcining conditions. It was found that air flow conditions play a major role on the morphology of the ashes and on the organic matter content. The following conclusions were drawn:

1. The condition of airflow plays a major role on the consumption of organic matter of the bagasse. Forced airflow reduces significantly the loss-on-ignition of the ashes.
2. The airflow also affects the morphology of SCBA. Calcining without forced airflow cannot break the long fibers of the bagasse in the temperature range of 400°C - 800°C.
3. The non-organic matter in the ashes display crystalline structure due to adhered sand and dust which was not washed away before the experiments.
4. Very high calcining temperatures may compromise the reaction of SCBA with the saturated lime solution due to the crystallization of the $SiO_2$.

## 6. Acknowledgements

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