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Ultrafine sugar cane bagasse ash: high potential pozzolanic material for tropical countries

Cinza ultrafina do bagaço de cana-de-açúcar: material pozolânico de alto potencial para países tropicais



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Abstract

This work describes the characterization of sugar cane bagasse ashes produced by controlled burning and ultrafine grinding. Initially, the optimum burning conditions of the bagasse were determined aiming the maximum pozzolanic activity. In sequence, an ultrafine sugar cane bagasse ash was produced in vibratory mill. Finally, the influence of use of ultrafine sugarcane bagasse ash (10, 15 and 20% of cement replacement, in mass) in properties of high-performance concretes was studied. Rheology (BTRHEOM rheometer), compressive strength (7, 28, 90, and 180 days) and rapid chloride penetrability were investigated. The results indicated that the addition of sugarcane bagasse ash improved the durability characteristics, and did not change the rheological and mechanical properties.

Keywords: *sugar cane bagasse ash, pozzolan, concrete, grinding, calcination.*

Resumo

Este trabalho descreve a caracterização de cinzas do bagaço de cana-de-açúcar produzidas a partir de queima controlada e moagem ultrafina. Inicialmente, as condições ótimas de queima do bagaço foram determinadas com o objetivo de alcançar a máxima atividade pozolânica. Em seguida, uma cinza ultrafina de elevada reatividade foi produzida em moinho vibratório. Por fim, estudou-se a influência do emprego de cinza ultrafina do bagaço de cana-de-açúcar (10, 15 e 20% de substituição de cimento, em massa) nas propriedades de concretos de alto desempenho. Foram avaliadas a reologia (reômetro BTRHEOM), a resistência à compressão (7, 28, 90 e 180 dias) e a penetração acelerada de íons cloro. Os resultados indicaram que a cinza do bagaço não altera as propriedades reológicas e mecânicas e possibilita a obtenção de concretos mais duráveis.

Palavras-chave: *cinza do bagaço de cana-de-açúcar, pozolana, concreto, moagem, queima.*

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1. Introduction

Pozzolanic materials are siliceous or aluminum-siliceous compounds that separately possess little or no cementitious properties. However, these materials can react to the calcium hydroxide in ambient temperature to form compounds with agglomerative properties when finely ground and in water presence [1]. The use of pozzolans is a common practice in worldwide. Materials as fly ash, silica fume, metakaolin and rice husk ash, amongst others, are used to produce concretes, mortars and pastes with differentiated properties. Several works show the technological advances, as increases of compressive strength and durability, and environmental benefits from the use of the described materials [1-4], normally produced from industrial or agro-industrial by-products.

In recent years, a new mineral admixture has been observed by research boards in Brazil [5-7] and abroad [8-12]: the sugar cane bagasse ash. The bagasse ash is the main agro-industrial by-product generated in Brazil and one of the most important by-product in the world [5]. It is generated during the production process of sugar and alcohol. Large quantities of bagasse are used as combustible for boilers after the extraction of the sugar cane juice in the plants and small distilleries. With the technological advances of the sugar/alcohol sector and the necessity of more electric energy, the bagasse has been valued as primary source of energy in Brazil. Nowadays, the bagasse has supplied the energy necessities of the sugar/alcohol sector and it also generates a excess that can be commercialized by concessionaires of energy or other industrial sectors [5]. In 2008, 650 million tons of sugar cane were produced in Brazil [13]. Thus, Brazil presents a potential of about 4,5 million tons of raw material for the mineral admixture production, distributed in all domestic territory, considering that about 0.7% of the mass of the sugar cane remains as ash after the combustion processes [5].

The bagasse ash presents pozzolanic activity when used with Portland cement [6-9]. Thus, it is possible to produce concrete with bagasse presenting positive mechanical and durability characteristics in comparison with the reference concretes [5-11], since the particles of the ash present ultrafine size. It is important to emphasize that the ashes used in the cited works were produced without control of the burning conditions. The production of the bagasse ashes with controlled burning could prevent the formation of crystalline phases and, consequently, increase its pozzolanic activity, as Payá *et al*. [10] and Cordeiro [5] show. This behavior is similar to the rice husk ash [1-12,14].

This work presents the results of the physical-chemical characterization of the ashes produced in controlled systems of burning and grinding. Thus, initially the results of a study on the influence of the burning temperature of the bagasse in the physical-chemical characteristics of the ash are presented. After that, the optimum conditions of vibratory grinding of a residual ash to the production of an ultrafine pozzolan are described. The results of the comparison between different properties (yield stress, plastic viscosity, compressive strength and rapid chloride-ion permeability) of concrete with ultrafine residual ash and a reference concrete are also presented in this work.

1.1 Justification

Several studies indicate the potential of the sugar cane bagasse ash for use as pozzolan in pastes and mortars [5-8,10,12]. However, there are few researches on the application of the ash in concrete [5.11]. This work presents different methods of production of the pozzolanic sugar cane bagasse ash. The bagasse ash is the main by-product of the sugar-alcohol industry, which presents a period of great growth. Thus, this work expects to contribute to the use of this by-product as pozzolan in different parts of the world, mainly in tropical regions, where the sugar cane production is very expressive.

2. Materials and experimental program

2.1 Materials

The bagasse and bagasse ash were collected in stock area and the boiler, respectively, of a sugar and alcohol factory situated in the city of São João da Barra/RJ, Brazil. The collection of the ash was carried out during the boiler cleaning operation throughout three months. After that, 180 kg of ash were homogenized and partitioned for the grinding tests. Table 1 presents the chemical composition of the bagasse and the ash, beyond the composition of the ordinary Portland cement (without mineral additions) used for evaluation of the pozzolanic activity of the ash in set with standard sand [15]. Cement, coarse aggregate of crushed syenite with 19 mm maximum size, siliceous river sand (fineness modulus of 2.12), water-reducing high-range admixture polycarboxylate-based (32.6% solids content) and deionized water were also used for the concrete mixtures.

Table 1 – Chemical composition (% in mass) of the bagasse residual ash and Portland cement

Compound	Bagasse residual ash	Portland cement
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
P_2O_5	1.07	–
Loss on ignition	0.42	1.05

2.2 Controlled burning

In laboratory, the bagasse was washed with distilled water and dried at 80 °C for 48 hours in a ventilated oven. After that, the bagasse was submitted to the burning in an electric oven without forced circulation of air, however with renewal of the gases during the burning by two orifices (10 and 40 mm of diameters) in the internal chamber. The adopted conditions of burning were: burning with two steps [5,16]; temperature of 350 °C in the first step;

Table 2 – Mixture proportions of concretes (in kg/m^3)

Materials	Reference	Content of bagasse ultrafine ash		
		10%	15%	20%
Portland cement	478.0	430.2	406.3	382.4
agasse ultrafine ash	–	47.8	71.7	95.6
Coarse aggregate	860.0	860.0	860.0	860.4
Fine aggregate	905.3	905.3	905.3	905.8
Superplasticizer	1.43	1.43	1.43	1.20
Water	164.4	164.4	164.4	164.4

temperature between 400 and 800 °C other step, with 100 °C of variation; heating rate of 10 °C/min; and time of residence in each step of 3 h. The ratio of sample to internal chamber volumes was maintained constant in all burning processes.

After burning, the samples were subjected to dry grinding using a Restch PM-4 planetary mill operating with 300 rpm, jar and grinding media of alumina (spheres of 10 mm diameter), 25% filling, 66% porosity and 2 min grinding time. Particle sizes were obtained by using a Mastersizer 2000 laser diffraction analyzer, Malvern Instruments. X-ray diffraction patterns (powder method) were taken by using a Rigaku Miniflex diffractometer with Cu-Kα radiation with Bragg angles between 5 and 50°, 0.05°/step and 2 s acquisition time. The chemical composition was determined by X-ray fluorescence (Phillips PW2400 spectrometer) with tube of 3 kW and rhodium target. The loss on ignition was determined in accordance with the NBR 5743 [17] standard. The specific surface area was obtained according to the N_2 adsorption (BET method) using a Gemini 2375 V5. The density was determined by using an Accupic Micromeritics gas (He) picnometer. The pozzolanic activity was determined in accordance with NBR 5752 [18] with the calculation of the pozzolanic activity index with Portland cement.

2.3 Ultrafine grinding

The dry grinding of the sugar cane bagasse ash was performed in a vibratory mill (Aulmann & Beckschulte Maschininfabrik) with cylindrical vase (internal diameter of 19 cm) of steel of 33 liters. 16.5 liters of cylindrical grinding bodies (13 diameter mm and height 13 mm) of alumina and 8 liters of sample were used in each batch. The grinding times used were 8, 15, 30, 60, 120 and 240 min. The characterization of the ashes was made with the tests: particle size distribution, scanning electron microscopy (Jeol JXA840-A microscope), Blaine specific surface area and pozzolanic activity index.

2.4 Mix-design, mixture and characterization of high-performance concretes

The concretes were designed within a computer code Betonlab Pro2 [19], when numerical simulation of different compositions are made based on Compressible Packing Model developed by De Larrard [20]. The compressive strength was stipulated in 60 MPa and the consistency, in accordance with slump test, kept in ranging between 130 and 170 mm with specific contents of superplasticizer. Four mixtures were designed: reference and concrete with partial replacement of 10, 15 and 20% (in mass) of the Portland cement for ultrafine bagasse ash, as shown in Table 2.

Besides the slump tests, the characterization of the concretes in fresh state was performed in BTRHEOM rheometer with determination of yield stress (τ_0) and plastic viscosity (μ) after 10 minutes of the mixture in planetary mixer. It was assumed that the concrete behaves as a Bingham fluid. Thus, there is a linear relationship between torque (Γ) and rotation speed (Ω) – Equation 1 – that permit the calculation of τ_0 and μ from equations 2 and 3, respectively [21].

$$\Gamma = \Gamma_0 + \frac{\partial \Gamma}{\partial \Omega}.\Omega \qquad (1)$$

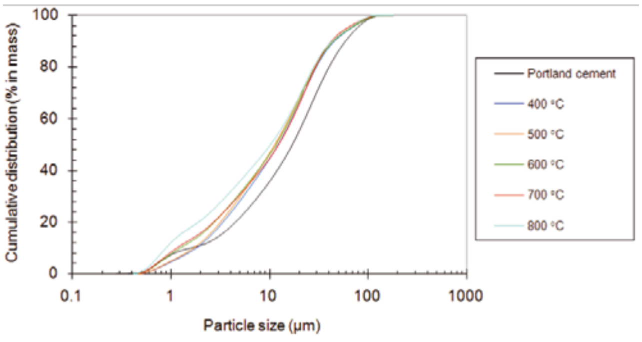
$$\tau_0 = \frac{3.\Gamma_0}{2\pi.\left(R_2^3 - R_1^3\right)} \qquad (2)$$

$$\mu = \frac{2.h.\dfrac{\partial \Gamma}{\partial \Omega}}{\pi.\left(R_2^4 - R_1^4\right)} \qquad (3)$$

Where:
Γ_0: liner coefficient of Equation 1;
$\frac{\partial \Gamma}{\partial \Omega}$: angular coefficient of Equação 1;
R_2: external radius of concrete in the rheometer (120 mm);
R_1: internal radius of concrete in rheometer (20 mm);
h: height of concrete in rheometer (100 mm).

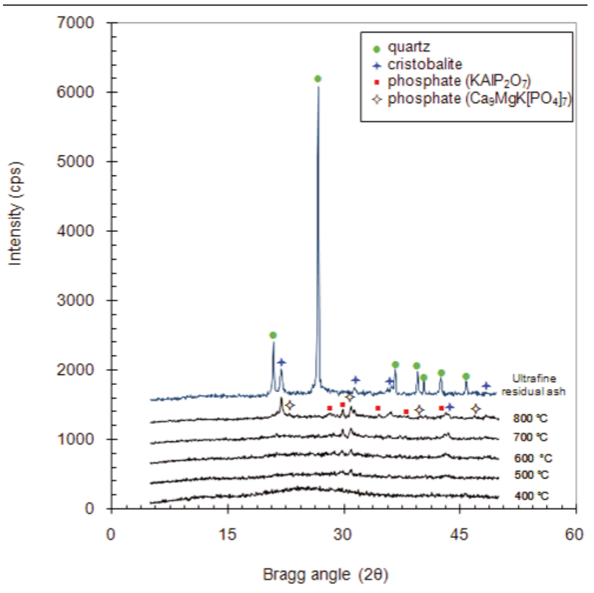
The compressive strength of concretes was determined by failure

Figure 1 –Particle size distributions of Portland cement and bagasse ashes produced in different burning temperatures



of cylindrical specimens (100 mm diameter and 200 mm height) in a Shimadzu UH-F1000kNI servohydraulic machine, after 7, 28, 90 and 180 days of curing in a moisture-controlled room (21 °C temperature and 100% relative humidity). The tests were performed in accordance with the NBR 5739 standard [22] with velocity of 0.0075 mm/min. Four specimens were used for each age. The durability behavior was investigated based on rapid chloride penetrability in 28 days, according to ASTM C1202 standard [23]. In this case, duplicate specimens were tested.

Figure 2 – X-ray diffraction patterns of bagasse ash produced in different burning temperatures and residual ultrafine ash produced in vibratory grinding during 120 min



3. Results and discussions

3.1 Production of pozzolanic ash with controlled burning

In this stage, the grindings were performed objecting to equalize the particle size distribution of ashes produced at different temperatures, as can be observed in Figure 1. Such procedure is important, since that the pozzolanic activity varies with the particle sizes of the material. The different products of grinding present not uniform granulometry and average particle sizes between 7 and 12 μm, which is a range of particle size similar to the granulometry of the Portland cement used in this work. The X-ray patterns of the sample, shown in Figure 2, indicate the variation of crystallinity of the silica as a function of the burning temperature. The ashes produced at 400 and 500 °C are amorphous, with a characteristic diffuse halo between angles 2θ of 20 and 30°. With temperatures above 600 °C, the ashes exhibit an incipient crystallization of phosphates ($Ca_9MgK[PO_4]_7$ and $KAlP_2O_7$). The ash produced at 800 °C presents silica as cristobalite, which is an indicative of the superior burning temperature for the production of a material with adequate pozzolanic activity, for the adopted conditions of grinding.

Figure 3 presents the values of pozzolanic activity index, calculated from the compressive strength of reference of 37.81 MPa. It is possible to observe that there is variation of the pozzolanic activity of the ash in function of the burning temperature. With the exception of the ash produced at 600 °C, which presents 77% activity index, the other samples do not reach the minimum value (75%) suggested by NBR 12653 [24] for a material can be classified as pozzolan. The reduced reactivity of the ash produced at 400 °C to be attributed to the dilution of the active phases due to high carbon content in the sample (85%). In accordance with the investigated parameters, the burning temperature of 600 °C is most appropriated to produce pozzolan from sugar cane bagasse ash considering the studied conditions of burning.

Figure 3 – Pozzolanic activity index values of sugar cane bagasse ash produced in different burning temperatures

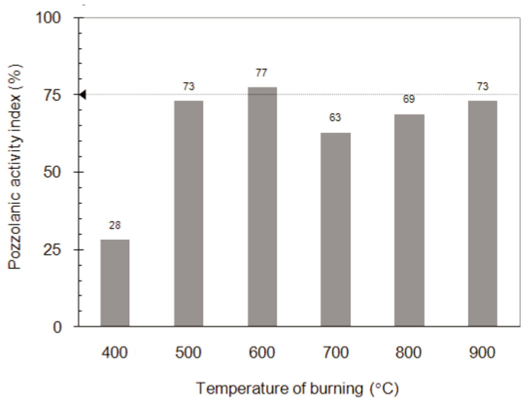


Table 3 – Chemical composition and physical characteristics of sugar cane bagasse ash produced at 600 °C

Compound	Content (% in mass)	Characteristic	Value
SiO_2	60.96	Density	2569 kg/m^3
Al_2O_3	0.09	BET Specific surface area	11887 m^2/kg
Fe_2O_3	0.09	Residue in 325 mesh	8.3%
CaO	5.97	Average particle size	11.6 µm
Na_2O	0.70		
K_2O	9.02		
MnO	0.48		
MgO	8.65		
P_2O_5	8.34		
Loss in ignition	5.70		

Table 3 presents the chemical composition and some important physical characteristics of the ash produced at 600 °C. The sample presents, in the adopted grinding conditions, 8.27% of the restrained mass in the 325 mesh (45 µm) and average particle size of 11.6 µm. The ash presents typical density for siliceous materials and the specific surface area (BET) of the ash is characteristic for ashes of cellular origin, which is similar to the fineness of rice husk ash [5].

3.2 Production of ultrafine residual ash with vibratory grinding

Figure 4 presents the particle size curves of the bagasse residual ashes produced in different times of vibratory grinding. After 60 min of grinding, for example, the produced ashes present maximum particle sizes of 40 µm and the particle size curves present narrower ranges of particle size. With 240 min of grinding an average size of 1.7 µm is reached. The increase of the values of specific surface in function of the increase of the grinding time, observed in Figure 5, confirms the faster breakage kinetics due to the ultrafine grinding. The approximately linear relation (R^2 = 97%) between the specific surface area and grinding time suggests the validity of the Law of Rittinger for the conditions used up to 120 min of grinding. This law establishes that the new specific surface area, produced for the grinding process, is directly proportional to the useful work consumed in this operation [25]. However, for the time of 240 min a divergence occurs between the obtained value and the Law of Rittinger, probably due to the extreme grinding in this period of test.

Figure 4 – Particle size distribution of bagasse residual ashes produced in different grinding times and as-received ash (no grinding)

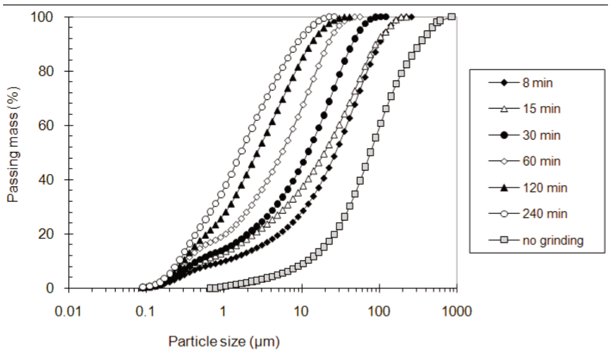


Figure 5 – Relationship between grinding time and Blaine specific surface area of bagasse residual ashes

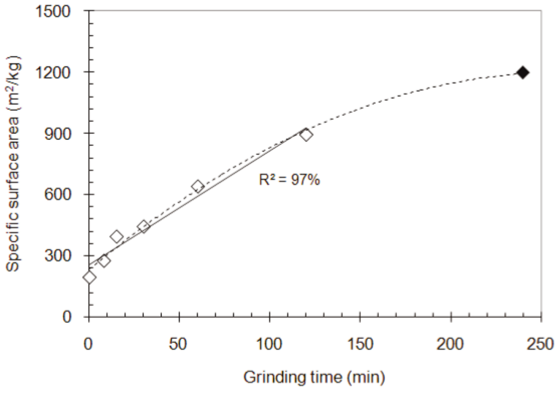
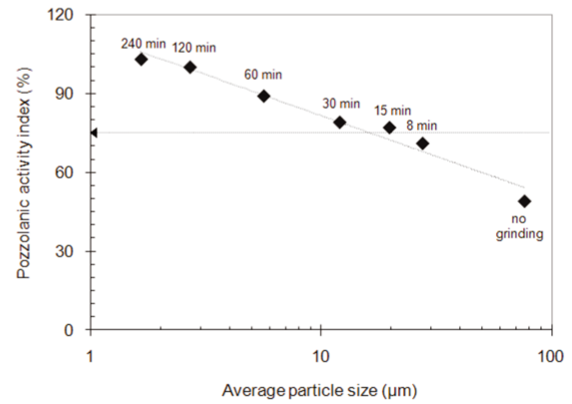


Figure 6 – Pozzolanic activity index values of sugar cane bagasse ash with different average particle sizes



The results of the pozzolanic activity tests are presented in Figure 6 and indicate an increase of the reactivity of the ash in function of the reduction of the average particle sizes, which is inversely proportional to the grinding time. All ashes present pozzolanic activity index higher than to the minimum value established by standard [24] after 15 min of grinding, with prominence for the values reached by the ashes produced with 120 and 240 min. However, it does not have an expressive difference between the values of activity index reached by 120 and 240 min ashes. After 120 min of grinding an ash with equal index the 100% was produced and the Law of Rittinger is applicable, enabling the calculation of the energy used for grinding process [5]. Thus, the grinding time of 120 min was adopted for the production of the sugar cane bagasse ash to be used in high-performance concretes. Figure 7 presents the morphologic details of the bagasse ash produced after 120 min of grinding, where it is possible to observe the presence of quartz as contamination for sand not removed after the washing of the sugar cane in the plant [5]. This evidence confirms the presented results of X-ray diffraction shown in Figure 2. It is important to note that the presence of the quartz does not compromise the use of the ash as pozzolan, mainly due to the filler effect provided by the ultrafine particles of the residual ash [6].

Figure 7 – Morphological aspects of sugar cane bagasse ash produced in vibratory grinding during 120 min. In details, particles of quartz (a) and bagasse ash (b)

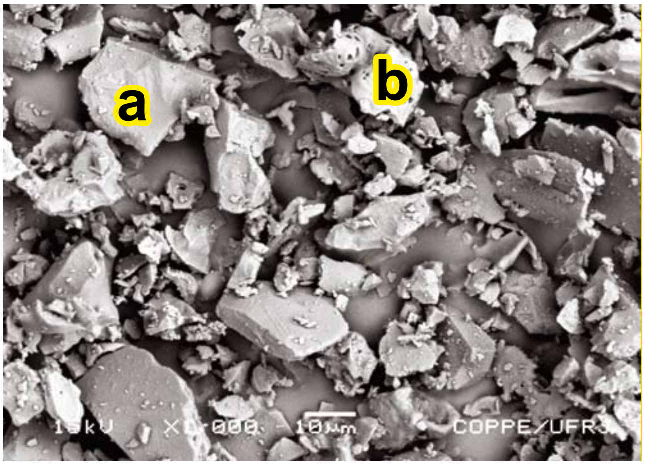
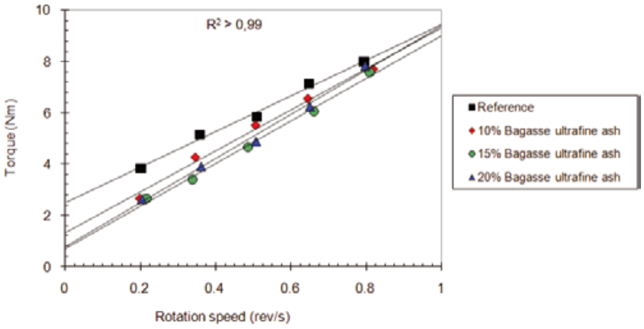


Figure 8 – Relationship between rotation speed and torque of different concretes in BTRHEOM rheometer



3.3 Application of ultrafine residual ash in high-performance concretes

The ultrafine ash of the bagasse (120 min of milling provides different characteristics of concrete), when in partial replacement to the Portland cement, as in fresh as in hardened state. The use of the ultrafine ash proportionates an increase of the slump values. In particular, the mixture made with 20% ultrafine ash presents consumption of the superplasticizer (dosage of 1.20 kg/m^3) lesser than the other mixtures. As observed in a test, the slump value would be 190 mm, superior value to the maximum limit of the adopted slump range (150 ± 20 mm) in case that the superplasticizer dosage were equal to the other concretes (1.43 kg/m^3). For the analysis of the rheology results in the BTRHEOM, presented in Figure 8, it is possible to note that the consideration of the concrete in fresh state as fluid of Bingham is adequate for the studied mixtures. The determination coefficients (R^2) for the linear adjustments between the torque and angular speed values are higher than 99% for all the mixtures. Table 4 shows the slump, yield stress and plastic viscosity values for the evaluated concretes. It is observed that the plastic viscosity is not expressively modified due to the incorporation of the ultrafine bagasse ash. However, the yield stress is lesser in the concretes with ultrafine ash, which indicates the positive effect of the chemical admixture in the concrete rheology. The best of the mixtures with ultrafine ash can be attributed to the reduced carbon content (0.42% loss on ignition) and presence of quartz particle with regular morphology (see Figure 7).

Table 4 – Properties of concretes in the fresh state

Mixture	Slump (mm)	τ_0 (Pa)	μ (Pa.s)
Reference	130	693	306
Bagasse ultrafine ash – 10%	150	362	353
Bagasse ultrafine ash – 15%	170	196	363
Bagasse ultrafine ash – 20%	170	211	380

The results of compressive strength tests performed after 7, 28, 90 and 180 days of curing are indicated in Figure 9. After 7 days, there are no significant differences between the average values of compressive strength of the reference concrete (53.8 MPa) and 10% of ultrafine bagasse ash concrete (55.0 MPa) according to the Duncan average test to the probability level of 5%. The 15 and 20% ultrafine ash concretes present values of compressive strength significantly inferior (50.7 and 47.1 MPa, respectively). After 28 days of curing, the reference concrete reaches a value similar to the strength established in the mix-design (60.9 MPa). The concretes with ultrafine bagasse ash, at this same age, do not present significant differences between itself and the reference mixture – strengths of 61.6, 59.0 and 57.8 MPa for the 10, 15 and 20% ultrafine ash concretes, respectively. There are no significant differences between the four investigated mixtures after curing of 90 and 180 days of curing, in accordance with the Duncan average test of the probability level of 5%. The strength final value observed for the reference concrete is equal the 71.2 MPa; for the ultrafine ash concretes are obtained 74.3 (10% of ash), 72.1 (15% of ash) and 70.5 MPa (20% of ash). It is important to note the reduced dispersion of the results of each composition in the investigated ages of evaluation. The greatest value of standard deviation (3 MPa), for example, is verified for the 15% ultrafine ash concrete after 28 days and corresponds to a coefficient of variation of only 5%.

The compression behavior of the concretes is in accordance with the value of the pozzolanic activity index of the selected ultrafine ash (Figure 6). In fact, the method used for the evaluation of the pozzolanic activity contemplates the physical and chemical effects of the ash, since it is calculated from values of compressive strength of mortars. As the ash presents 100% pozzolanic index, the cement replacement by the ultrafine bagasse ash does not cause reduction of the compressive strength, since the replacement level is not very elevated (above 20%).

In relation to accelerated chloride-ion penetration tests, the ultrafine bagasse ash provides reductions of about 30% in the electric charge values of the concretes when compared with the reference mixture. In this case, the reference concrete can be classified as having "low" penetration, while the ultrafine ash concretes are classified as having "very low" ionic penetration, with electric charge lesser than 1000 C, as shown in Figure 10. Similar results were observed by Ganesan *et al*. [11] when comparing a reference concrete of 40 MPa with a concrete with cement replacement by 20% sugar cane bagasse ash from India. The reduced ionic penetration in the concretes with ash can be attributed to the physical and pozzolanic effects of the ultrafine bagasse ash [6].

4. Conclusions

In accordance with the presented results, it is possible to conclude that:

- The sugar cane bagasse can be an important raw material for the pozzolan production, mainly in tropical countries, where

Figure 9 – Average compressive strength values of different concretes against curing time (dispersion of results indicated by standard deviation)

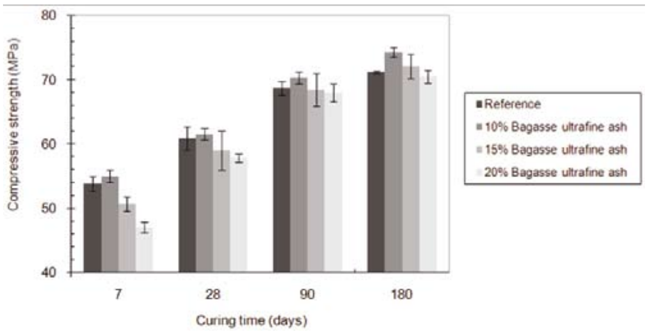
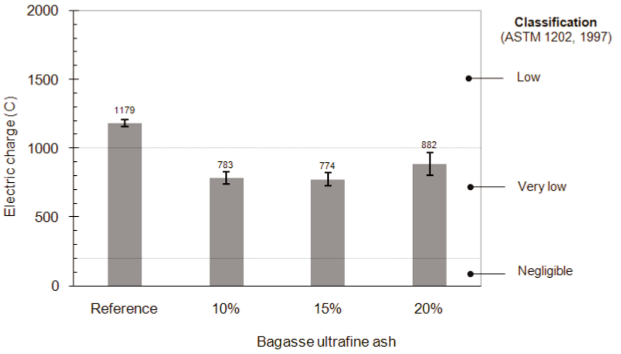


Figure 10 – Chloride-ion accelerated penetration values of different concretes (dispersion of results indicated by standard deviation)



the culture of sugar cane is very important. Specifically, an amorphous ash with high specific surface area and reduced loss on ignition can be produced with burning at 600 °C in muffle oven. The pozzolanic activity of the bagasse can be attributed to the amorphous silica;

- Pozzolans can be produced with vibratory grinding of the sugar cane bagasse residual ash. In this case, the grinding in vibratory mill for 120 min enables the production of an ash with pozzolanic activity index of 100%;
- With the produced residual ash in ultrafine grinding during 120 min the cement replacement up to 20% is possible in high performance concrete with the improvement of the rheological properties, non-reduction of the compressive strength and with “very low” chloride-ion penetration.

5. Acknowledgments

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