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Brazilian sugar cane bagasse ashes from the cogeneration industry as active pozzolans for cement manufacture

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ABSTRACT

For proper management of wastes and their possible recycling as raw materials, complete characterization of the materials is necessary to evaluate the main scientific aspects and potential applications. The current paper presents a detailed scientific study of different Brazilian sugar cane bagasse ashes from the cogeneration industry as alternative cementing materials (active addition) for cement manufacture. The results show that the ashes from the industrial process (filter and bottom ones) present different chemical and mineralogical compositions and pozzolanic properties as well. As a consequence of its nature, the kinetic rate constant (K) states that the pozzolanic activity is null for the bottom ash and very low for the filter ash with respect to a sugar cane bagasse ash obtained in the laboratory under controlled burning conditions (reference). The scarce pozzolanic activity showed by ashes could be related to a possible contamination of bagasse wastes (with soils) before their use as alternative combustibles. For this reason, an optimization process for these wastes is advisable, if the ashes are to be used as pozzolans.

1. Introduction

In recent years, the research related to agricultural wastes is intensifying with the aim of evaluating their potential for recycling as well as the elimination of the landfills. In this research line, the works are focused mainly on the sugar cane wastes. Frías et al. [1–3], Villar-Cociña et al. [4–6] and Morales et al. [7] have carried out laboratory studies on the influence of the activation of Cuban sugar cane wastes (straw and bagasse) at 800 and 1000 °C on pozzolanic reactivity. These studies concluded that the resulting ashes presented high pozzolanic activity and their viability to be incorporated as active additions in the production of cements.

Brazil is the biggest sugar producer and exporting country in the world and also with a considerable expansion being expected for the following years. According to the available data, in 2008 more than 568 million tons of sugar cane were generated in Brazil for the production of alcohol (approximately 27,000 million litters) and sugar (approximately 32 million tons) [8,9]. As a result of this industrial process considerable volumes of bagasse wastes are generated, which is estimated to be about 25% of the total production of sugar cane equivalent to approximately 142 million tons annually. However, the projection is about 1000 million tons of sugar cane in 2020 with the consequent increase in the ash generation.

Previously, these bagasse wastes were burned as a means of solid waste disposal but during the last decade these residuals, for their calorific characteristics, are used as the principal raw materials in cogeneration plants to produce electric power in Brazil. Pippo et al. [10] reported the economic and environmental advantages of using bagasse wastes as alternative energy to obtain electricity versus traditional combustibles.

Once used as fuel, the process generates an important amount of ashes around 2.7 million tons, which are accumulated in landfills waiting for alternative ways for its reutilization as raw material in other industrial sectors. In general, the ashes of any industrial process are characterized to be a material highly powdered of low density and high volume. These characteristics cause, due to the effect of the atmospheric conditions, the contamination of adjacent soils, underground water and problems of health, which bears serious social and environmental problems.

These residues obtained in the industrial process of cogeneration are not as homogeneous as their homologous in the laboratory, due to their direct dependence with different factors such as agriculture variables, raw material used as fuel and cogeneration temperature. The resulting ashes can be very different in composition and behavior, which can affect their possible management of reutilization for a certain industrial sector and more concretely in the cement sector. In this sense, the research works published are scarce and only based on technical studies with boiler bagasse ashes [11–15], without analysing scientific aspects, which are the base to understand the technical properties. No bibliographic

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references about the behavior of the cyclons ashes as secondary cementing material have been localized. Therefore, it is necessary to carry out deeper investigations on the correct management of these industrial bagasse ashes in terms of their characteristics.

The aim of the present work is to analyze, to characterize and to evaluate the scientific properties of cogenerated bagasse ashes, obtained in the boilers as well as the ashes obtained in the cyclones. For this, it is necessary to evaluate the pozzolanic activity of the ashes and the measurement of the kinetic parameters by applying a kinetic–diffusive model. Additionally, a bagasse ash was obtained in a laboratory furnace under controlled conditions in order to obtain a reference ash.

2. Experimental

2.1. Materials

Materials used in this research work were different three types of sugar cane bagasse ashes from the same bagasse waste generated in a sugar factory with the following origin: (1) The laboratory bagasse ash (LBA) obtained in an electric furnace with a 10 °C/min heating rate, first at 400 °C for 20 min and then at 800 °C for 60 min. (2) Filter bagasse ash (FBA) obtained from combustion fumes, reaching temperatures of 300 °C and (3) Bottom bagasse ash (BBA) reaching a boiler temperature of approximately 800 °C. Both industrial ashes are originated from the same Brazilian sugar cane mill which uses bagasse wastes as combustible for the cogeneration process. All ashes were sized in order to obtain particles below 90 μm.

2.2. Methods

2.2.1. Pozzolanic method

The pozzolanic activity of ashes was studied by using an accelerated chemical method. The test consists in putting the bagasse ash (1 g) in a lime-saturated solution (75 mL) at 40 °C for 1, 7, 28, 90 and 360 days. The CaO concentration in the solution was analyzed at the end of each period. The combined CaO (mmol/L) was obtained as the difference between the concentration in the control lime-saturated solution (17.68 mmol/L) and the CaO content in the solution in contact with the sample.

2.2.2. Kinetic–diffusive mathematic model

A kinetic–diffusive model published by Villar-Cociña et al. [4–6] is used to describe this pozzolanic reaction in a pozzolan/lime solution system. The model is:

$$C_t = \frac{0.23 \cdot Exp\left(\frac{-3t}{\tau}\right) \cdot \left(-1 + Exp\left(\frac{t}{\tau}\right)\right) \cdot \frac{1}{\tau}}{C_o D_e r_s} + \frac{0.23 \cdot Exp\left(-\frac{t}{\tau}\right) \cdot \frac{1}{\tau}}{C_0 K r_s^2} + C_{corr} \quad (1)$$

where D_e is the effective diffusion coefficient, K is the reaction rate constant, τ is a time constant (the time interval during which the pozzolan radius diminishes until a 37% of its initial radio r_s).

C_t represents the absolute loss of CH concentration with time for pozzolan/lime system and C_{corr} is a correction parameter that takes into account the concentration remainder of CH that is not consumed in the reaction. In some systems the CH is not consumed totally.

It is known that the pozzolanic reaction develops by stages. The resistances of these stages are usually very different and the stages presenting the greatest resistances (i.e. the stages that lapses more slowly) control the process. Accordingly, it is possible in certain cases to have different behavior: diffusive (described by the first term of Eq. (1)), kinetic (second term) and kinetic–diffusive (both terms). Further explanations about the model can be found in Villar-Cociña et al. [5,6].

2.2.3. Experimental techniques of characterization

Different techniques were used for the chemical, physical, mineralogical and morphological characterization. Chemical characterization was carried out by X-ray fluorescence (XRF), using a Philips PW 780 equipment, with an anticathode tube of rhodium of 4 kW. Physical characterization of ashes was carried out with a Sympatec Laser granulometer, with a wet system and isopropyl alcohol as non reactive medium. Mineralogical characterization was studied by X-ray diffraction (XRD) by using the random powder method for the bulk sample and the oriented slides method for the <2 μm fraction. The X-ray diffractometer is a SIEMENS D-500 with a Cu anode, operated at 30 mA and 40 kV using divergence and reception slits of 2 and 0.6 mm respectively. The XRD profiles were measured in 0.04 2θ goniometer steps for 3 s. Concerning thermogravimetric and differential thermal analysis (TG/DTA), a Stanton equipment STA 781 model was used. Samples between 12 and 16 mg of powder were heated at a heating rate of 10 °C/min in an N_2 atmosphere. Morphological characterizations were carried out by using a scanning electron microscope (SEM-EDX) device (PHILIPS XL30, W source, DX4i analyzer and Si/Li detector). The analyzer was previously calibrated with a multimineral sample.

3. Results and discussion

3.1. Physical characterization

The particle size distribution of starting bagasse ashes are shown in Fig. 1. All ashes show similar cumulative size distributions between 0.9 and 175 μm although the BBA has a coarser one than those obtained for the LBA and FBA. So, the 50% of the ashes particles passed by 12 μm for the LBA and FBA, while for the BBA this happened at 18 μm (Fig. 1a).

A more detailed observation of the distribution density curves (Fig. 1b), it is clearly observed that the ashes are formed by three distribution densities at 6, 18 and between 40 and 70 μm. This fact

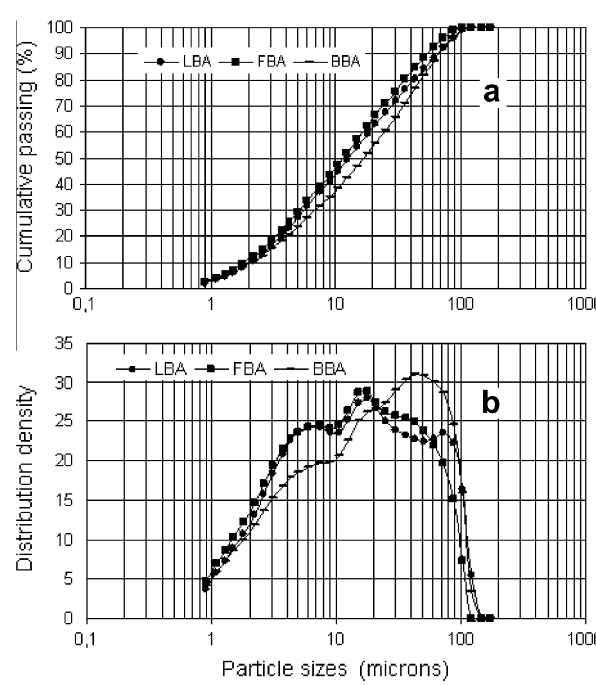

Fig. 1. Particle size distribution curves.

would be related with different mineralogical components present in the ashes. The ash BBA presents a main maximum at 45 μm; while the rest of ashes the maxima distribution densities are localized at 18 μm.

3.2. Chemical characterization

The chemical results (Table 1) show that the three bagasse ashes are composed by SiO_2, Al_2O_3 and Fe_2O_3 whose sum reaches superior values to 75% in the worst of the cases (FBA). The rest of the oxides (K_2O, MgO, TiO_2, P_2O_5 and SO_3) are present in concentrations below to 4%. It is important to highlight that the concentration of Na_2O in the ashes is below 0.2%. The ash FBA shows a smaller content in silica (56%) and a high content of loss on ignition of 18%. This value would be related with the possible presence of non-burned organic particles (coal, bagasse fibers) that are dragged by the combustion gases. The bagasse ash obtained in the laboratory is the more enriched in silica. The BBA shows a different composition to those published previously, mainly in content of SiO_2 (85.6%), Al_2O_3 (5.3%) and Fe_2O_3 (1.3%) [14].

3.3. Mineralogical characterization

3.3.1. X-ray diffraction (XRD)

Fig. 2 shows the XRD patterns corresponding to the analyzed ashes. Mineralogically, the three ashes present very similar spectra, formed by quartz as the crystalline component majority. Also, mullite, iron oxides (Fe_3O_3) and graphite can also be present, but due to its small quantity as well as overlapping bands make very difficult its identification. It is important to note that the cristobalite (at 21.9 2θ) was identified in BBA, fact that can be an indicator that the temperature reached in the boiler was very superior at the 800 °C. For the case of FBA, traces of kaolinite (at 12.33 2θ) and gibbsite (at 18.29 2θ) are also identified. The presence of kaolinite in FBA states that gases combustion temperature is lower than temperature for the process of dehydroxylation of kaolinite (>450 °C).

The mineralogical composition in these bagasse wastes differs considerably to the found in other countries (Cuba, India and Thailand) in whose composition can be calcareous and cristobalite next to the quartz as main components [6,13,16].

From results obtained from mineralogical study, its important to note the high soil contamination (sand and clay minerals as kaolinite), which will have a negative influence on the pozzolanic properties of ashes, mainly in the ashes from boiler and its scarce interest as pozzolans since they will not have a positive influence on the performances of the new blended cements.

3.3.2. Thermogravimetric analysis (TG/DTA)

Thermal analysis (TG/DTA) revealed thermal changes and weight loss when bagasse ashes were subjected to a thermal process. Fig. 4 illustrates these thermal variations for the studied ashes. The TG curves (Fig. 3a) show variable weight losses during heating up to 1000 °C in function of ash origin. So, the LBA presents a minimum and continuous weight loss about 1.69%, followed by the BBA with a loss of 1.98% and finally the FBA shows a weight loss of 9.08%.

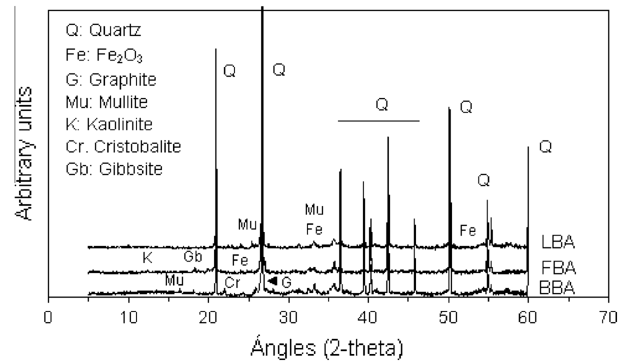

Fig. 2. Mineralogical composition by XRD.

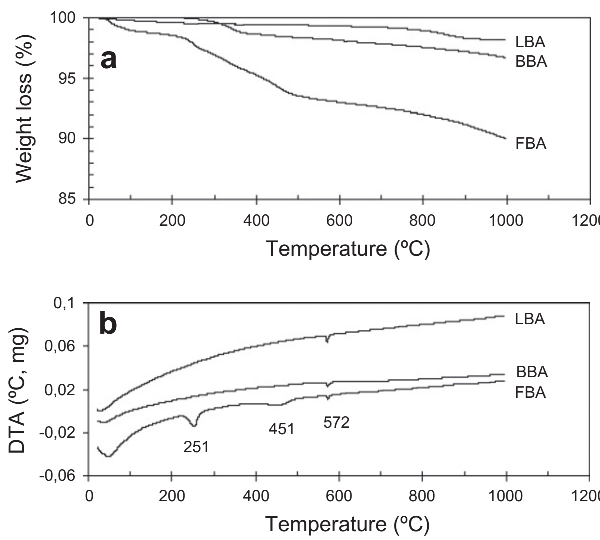

Fig. 3. TG (a) and DTA (b) curves for the ashes.

From DTA curves (Fig. 3b) the following phenomena are observed:

(1) The three ashes (LBA, BBA and FBA) show an endothermic peak localized at 572 °C, which could be attributed to the carbonates decomposition. This is a very weak peak not detected in TG curves that would be indicating the presence of traces. A second peak localized about 50 °C corresponds to the adsorbed water loss.

(2) The ash named as FBA which was obtained from filters or cyclones show important differences with respect to the other ashes:

- An endothermic peak at 251 °C attributed to the dehydroxilation process of gibbsite, which was also identified by XRD and FTIR.
- An endothermic peak at 451 °C assigned to the kaolinite dehydroxilation process. This fact states the presence of this clay mineral in the FB ash.

Table 1
Chemical compositions of ashes by XRF in % by mass.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	TiO_2	P_2O_5	LOI
LBA	69.40	11.26	5.41	2.51	1.28	1.83	3.45	0.09	1.38	1.61	1.56
FBA	55.97	12.44	6.50	0.84	0.48	1.00	0.90	0.00	2.67	0.98	17.98
BBA	66.61	9.46	10.08	1.43	0.92	0.10	3.19	0.22	2.44	1.04	4.27

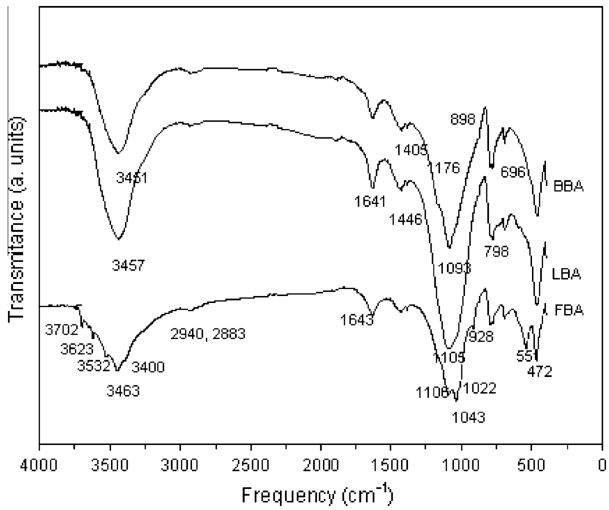

Fig. 4. FTIR spectra of bagasse ashes.

It is important indicate that the possible organic matter present in bagasse ashes is not detected by this techniques because the thermal analysis was carried out in inert conditions (nitrogen gas).

3.3.3. Fourier Transform spectroscopy (FTIR)

The results obtained by means of FTIR supplement the results obtained by the other techniques (DRX, TG/DTA) and they offer the possibility to identify the amorphous or not very crystalline substances. Fig. 4 shows the spectra of the three ashes. In general it can be observed that the spectra of BBA and LBA are qualitatively very similar, but with appreciable differences in relation with FBA.

In all ashes, the wide characteristic bands of vibration of O–H corresponding to the not structurally combined water (3451–3463 cm^{-1}) and the corresponding bands of deformation of the connection H–O–H (1641 cm^{-1}) are observed. The typical bands of the group carbonate $\left(CO_3^{2-}\right)$ are identified, located to 1446 and 898 cm^{-1} in minimum concentrations in the three ashes since this was not identified in XRD patterns.

The bands at 2940, 2883 and 1405 cm^{-1} confirm the presence of coke particles traces. Finally, in the three bagasse ashes are identified clearly the presence of wide bands located between 1093 and 1106 cm^{-1} and other bands of smaller intensity at 1176, 798 (doublet), 696 and 472 corresponding to the vibrations of the bonds Si–O of the quartz.

The FTIR spectrum of the ash obtained from cyclons shows other clearly defined characteristic bands not detected in LBA and BBA. In this type of ash the presence of kaolinite is identified for the vibration bands due to the group hydroxyl (OH^-) to 3702 and 3623 cm^{-1} and for the bands corresponding to the vibrations of the bonds Al–OH and Al–O in octahedrical coordination of kaolinite located to 928 and 551 cm^{-1}. However it is not possible to identify the bands of vibrations corresponding to the metakaolinite, obtained by deshydroxylation of kaolinite. It is due to that their main bands at 472 cm^{-1} corresponding to the SiO_4 and to 800 cm^{-1} referred to the vibrations Al–O of the Al^{3+} in tetrahedrical coordination is overlapped with those of the quartz, which is the main mineralogical component.

On the other hand, in these ashes bands of vibrations of little intensity located to 3592, 3463 and 1022 cm^{-1} corresponding to the presence of gibbsite $(AlOH)_3$ is also identified. These findings by FTIR are in agreement with data obtained by XRD and TG/DTA.

3.4. Morphological composition

It is well known that the morphology of ashes depends directly on the process and burning temperature. A morphological study of the selected ashes carried out by SEM is shown in Fig. 5.

The SEM images show different particles morphologies as a function of the obtaining process. It is clearly observed the presence of coarse particles with respect to the rest of the matrix. The size of these particles differs considerably between the ashes. The particle sizes increase in the following order: FBA < LBA < BBA. The BBA shows a morphology with high coarse particles content; while for the FBA its content had decreased a lot. A semi-quantitative analysis by EDX shows that these coarse particles present in all ashes are formed by silica, which corresponds to the quartz particles identified by XRD patterns.

Apart of quartz, the ashes are formed by different sizes agglomerations (Fig. 6, left) constituted mainly by oxides of silicon and aluminum in a ratio of 2:1 approximately. Only for the case of filter bagasse ashes (FBA) prismatic form particles (Fig. 6, right) were identified with high Si, Al, Ti and Fe contents in a relation of 2 (SiO_2): 1 (Al_2O_3): 1 (TiO_2): 0.5 (Fe_2O_3). This mineral could belong to the fyllosilicate family.

Also, as the temperature reached in the cyclones is very lower than those produced in the laboratory and boiler, prismatic particles of 30 μm long were observed in this kind of ashes (Fig. 7). The EDX analysis show organic nature particles by its high oxygen content (O: 48.78%), which was masked by matrix particles deposited on the surface (Al: 14.07%, Si: 24.16%, S: 3.33%, Fe: 6.20%, K: 1.22% and Na: 1.36%). These findings are in agreement with the observations carried out by previous researchers [12].

3.5. Evaluation of pozzolanic activity of ashes by accelerated chemical method

The pozzolanic activity of different bagasse ashes have been evaluated by the determination of fixed lime ($Ca(OH_2)$) versus

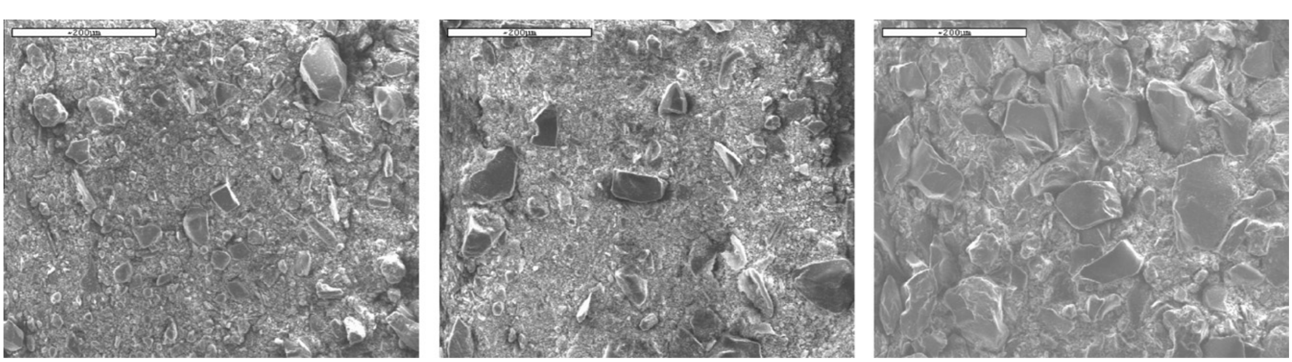

Fig. 5. General aspects of ashes at the same magnification (200 μm): (left) FBA, (middle) LBA and, (right) BBA.

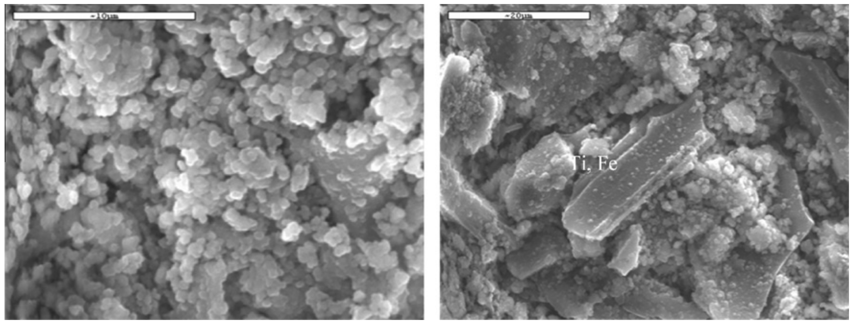

Fig. 6. (left) SEM of LBA (×5000), (right) Prismatic particle enriched in Ti and Fe (×2000).

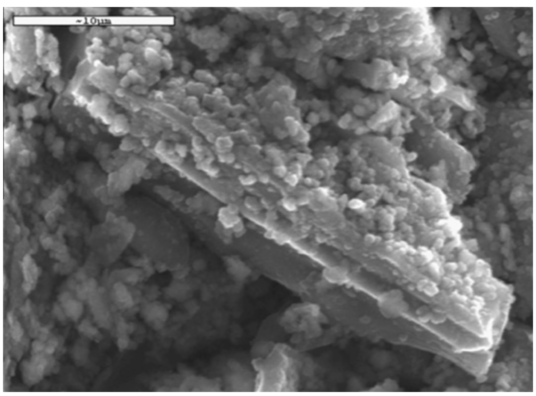

Fig. 7. Prismatic particle in FBA.

reaction times, knowing that the lime-saturated solution has an initial concentration of 17.68 mmol/L. From results summarized in Fig. 8, it is possible to highlight that the pozzolanic behavior is very different for the three ashes.

The ash obtained under controlled conditions in laboratory at 800 °C shows a very high activity according to the values of fixed lime. So, during the first 24 h of reaction, LBA fixed a 42% of the total lime content in solution, passing to 81% at 7 days and a 94% to the end of testing (90 days). For the ash obtained from boiler (or bottom) no pozzolanic activity was showed while the ash coming from cyclones presents a low-medium activity, moving from 19% at 6 h to 42% at 90 days.

These values indicate that the bottom ashes generated in the industrial process, which use bagasse waste as alternative combustible to produce energy do not present enough capacity to react with the lime ($Ca(OH_2)$) to form hydrates phases with hydraulic properties. In a previous paper, Frías and Villar-Cociña [2] reported that the main hydrated phase obtained during the pozzolanic reaction in Cuban bagasse-lime system was gel CSH similar to that

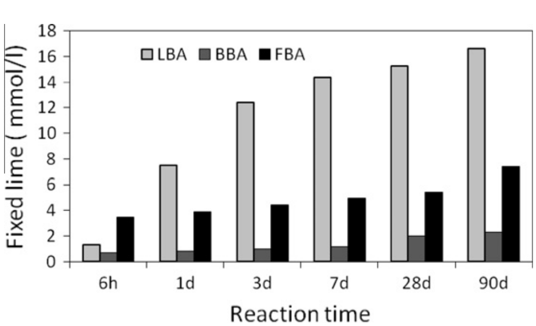

Fig. 8. Pozzolanic activity evolution.

obtained from hydration reaction of Portland cement but with a Ca/Si ratio less than 1.

When BBA is compared with a laboratory bagasse ash, the activity would be similar in both cases since they were calcined about 800 °C. As the behavior is totally different, it is necessary to think that during the burning process different aspects can happen:

- Temperatures inside the boiler should reach values above 1000 °C localized in the regions close to the surface of burning material in order to transform the amorphous silica into crystalline one. In a previous paper (Frías et al. [3]) it was shown that bagasse ashes at calcining temperatures between 800 and 1000 °C are still reactive.
- The bagasse wastes used as combustible in boilers are strongly contaminated with important amounts of agricultural soil, mainly with quartz mineral. If this is the case, the BBA would not be a suitable pozzolan for the cement.
- Finally, the cogeneration process used into industry is not the most suitable to obtain activated bagasse ashes for cement manufacture if considered in the way it is presently conducted.

3.6. Computing of the kinetic parameters of the pozzolanic reaction

The kinetic–diffusive model (Eq. (1)) was applied to all samples by using non-linear regression techniques. Fitting the absolute loss of lime concentration versus reaction time successively to the kinetic control model, diffusive control model and a mixed (kinetic–diffusive) control model and carrying out an exhaustive analysis of the important statistical parameters such as correlation coefficient (r), coefficient of multiple determination (R^2), 95% confidence intervals, residual scatter, residual probability and variance analysis (which constitutes a rigorous evaluation of the fitting process of the model to the experimental data), it can be concluded that kinetic control model shows the best correspondence with the experimental data. Fig. 9 illustrates the absolute loss of lime concentration versus time for the LBA, BBA and FBA samples. The solid line represents the curve of the fitted model.

This means that the chemical interaction speed on the surface of the nucleus of the pozzolan particle is slower than the diffusion speed of the reactant through the reaction product layer formed around the nucleus. This might be due to high porosity of the reaction product layer in these ashes, which facilitates a quick diffusion process.

In the current paper, only a few statistical parameters are shown (r, R^2, SE, RSS) since the rest (mentioned above) are related to the graphic analysis and require large tables better suited for a more specific paper.

The values of the τ parameter and the reaction rate constant K are given in Table 2. In Fig. 9, the correlation and multiple determination coefficients r and R^2 are shown.

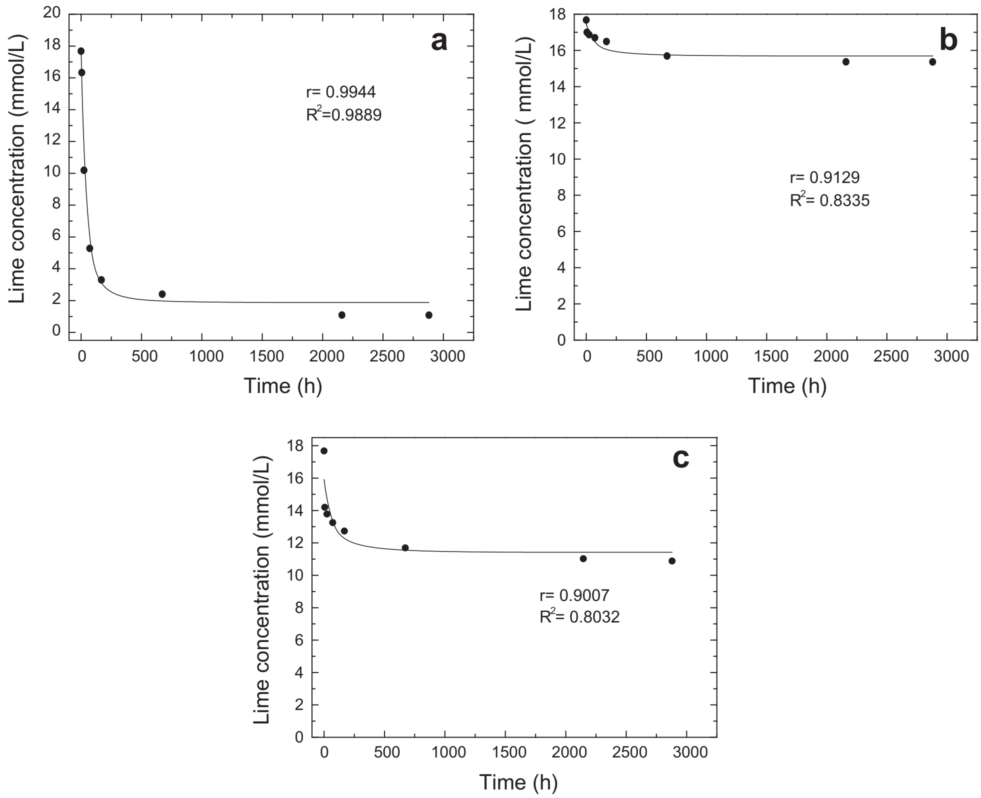

Fig. 9. Absolute loss of CH concentration plotted against reaction time for: (a) LBA, (b) BBA, (c) FBA samples. Black circle, experimental; solid line, model.

Table 2
Reaction rate constants, τ parameter, C_{corr} parameter and statistical parameters for sugar cane bagasse waste samples.

Material	τ (h)	Reaction rate constant, K (h^{-1})	C_{corr}	Correlation coefficient (r)	Coefficient of multiple determination (R^2)
LBA	41.2 ± 5.1	$(6.73 \pm 0.84) \times 10^{-3}$	2.18 ± 0.452	0.9944	0.9889
BBA	78.3 ± 7.9	$(2.80 \pm 0.6) \times 10^{-4}$	15.69 ± 0.22	0.9129	0.8335
FBA	74.4 ± 6.8	$(4.6 \pm 0.7) \times 10^{-4}$	11.42 ± 0.66	0.9007	0.8032

The K values reflect directly the reactivity of the pozzolan and it is a direct index of the pozzolanic activity of the materials. According with the K values, the LBA sample shows more pozzolanic activity (order of 10^{-3} h^{-1}) in comparison with the samples BBA and FBA, which show very low pozzolanic activity (only in the order of 10^{-4} h^{-1}).

4. Conclusions

Based on the scientific studies in the current work, the following conclusions can be drawn:

- Chemically, the three bagasse ashes are formed by the same oxides but with different contents. The main oxides are silica and alumina, although BBA shows a 10% Fe_2O_3. FBA presents a LOI value of about 18% by mass.
- Mineralogically, the ashes are formed by quartz as the main crystalline compound, showing slight differences in the minor minerals. So, cristobalite is only identified in BBA, and kaolinite and gibbsite in ashes from cyclons.
- TG/DTA and FTIR curves confirm the presence of these minor compounds although metakaolinite was not detected by FTIR due to the overlapping of the main characteristic bands with the quartz.
- The three bagasse ashes are morphologically different from each other since it is clearly observed that the calcining temperature has a direct influence in the morphology. So, the quartz particles size and quantity increases substantially from FBA to BBA. The main origin of quartz is the soil contamination in the bagasse wastes.
- A considerable effort is being carried out at the moment by the mills management in order to reduce the soil brought with the sugar cane from harvest operations.
- The fixed lime results show that the boiler bagasse ashes do not show pozzolanic properties while the ashes obtained from cyclones (FBA) has a low-medium activity. The bagasse ashes elaborated under controlled temperature conditions in laboratory showed excellent properties.
- The values of the reaction rate constant obtained in the fitting process of the kinetic–diffusive model show that the LBA has high pozzolanic reactivity (order of 10^{-3} h^{-1}). The FBA and BBA ashes have low pozzolanic activity (reaction rate constant in the order of 10^{-4} h^{-1}), with BBA being the ash with less pozzolanic properties.

As a summary of all exposed, it is important to highlight that the obtained ashes quality is very different from a scientific viewpoint. The two industrial ashes analyzed in the present paper, from boiler and cyclone of sugar cane mill, by its nature could have minimum acceptance for the FBA and null for the BBA as supplementary cementing materials in cement production. For this reason, proper management of these agricultural wastes before their use as combustibles requires good control prior to arrival to the plant, removing all the contaminant materials in order to get only clean bagasse material and controlling the calcining temperature around 800 °C. In these conditions the BBA and FBA will present a pozzolanic behavior more similar to that of ashes produced in the laboratory.

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