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Characterization and properties of blended cement matrices containing activated bamboo leaf wastes

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A B S T R A C T

The worldwide production of bamboo generates large volumes of leaf wastes, which are deposited in landfills or burned in an uncontrolled manner, with negative effects in the environment. The ash obtained by calcining of the bamboo leaf waste, shows good qualities as supplementary cementing material for the production of blended cements.

The current paper shows a detailed scientific study of a Brazilian bamboo leaf ash (BLA) calcined at 600 °C in small scale condition, by using different techniques (XRF, XRD, SEM/EDX, FT–IR, TG/DTG) and technical study in order to analyse the behaviour of this ash in blended cements elaborated with 10% and 20% by mass of BLA. The results stated that this ash shows a very high pozzolanic activity, with a reaction rate constant K of the order of 10^{-1}/h and type I CSH gel was the main hydrated phase obtained from pozzolanic reaction. The BLA blended cements (10% and 20%) complied with the physical and mechanical requirements of the existing European standards.

1. Introduction

For decades, the cement industry has been incorporating significant amounts of industrial byproducts (silica fume, fly ash, blast-furnace slag, metakaolin) for its economic, energetic and environmental benefits [1–5], which are clearly specified in the existing standards [6].

In recent years, either by the scarcity of natural resources or by economic crisis, there has been a clear trend toward investigating alterative additions for the manufacture of eco-efficient blended cements. Recent research has focused on wastes generated in different agro-industrial sectors [7–13], which are generating serious environmental and social problems, by their accumulation in landfills and uncontrolled burning.

One of these sectors is the bamboo with an annual production estimated in 20 million tons all over the world, mainly in Asia and Latin America. Brasil is one of the main producers of bamboo in Latin America with plantations estimated in 10 million of hectares [14], and according to the available estimated data of 25 t ha^{-1} [15], it would be producing about 250 million of bamboo annually, generating high volume of leaves, disposable wastes but with suitable properties to use in new applications.

The use of bamboo leaf ashes (BLA) for industrial applications is, at present, a novelty and little-known research line as reflected by the scarcity of available literature [15,16]. As supplementary cementing materials, the only referenced works were published by Singh et al. [17] and Dwivedi et al. [18] in which they reported the good pozzolanic properties of an Indian bamboo leaf ash, obtained under controlled calcination conditions at laboratory scale. Subsequently, Villar et al. [19] determined for the first time, the kinetic parameters of the pozzolanic reaction, using the conductimetric method to assess the pozzolanic behavior of a Brazilian BLA.

In view of the limited information, the main objective of this research is to deepen the scientific aspects of the pozzolanic reaction in the BLA/Ca $(OH)_2$ system, and the subsequent behavior in blended cements made with addition of BLA (10% and 20% by mass) and the correspondent fulfilment with existing standards for the elaboration of commercial type II/A cements.

2. Experimental

2.1. Materials

- The bamboo leaves were recollected in the campus of the University of Sao Paulo in Pirassununga (latitude 21°59′46″ S and longitude 47°25′33″ W), state of Sao Paulo, Brazil. The bamboo leaf ashes (BLA) were obtained in a laboratory electric furnace at 600 °C calcining temperature for 1.20 h of retention, which represents a 5% of total mass of leaves. Once calcined, the ashes were ground in a agate mortar and pestle and sieved below 45 μm.

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Table 1
Chemical compositions of BLA and OPC 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
BLA	78.71	1.01	0.54	7.82	1.83	1.00	3.78	0.05	0.08	0.99	3.83
OPC	20.16	4.36	2.52	63.41	2.21	3.57	0.91	0.35	0.21	0.14	1.99

- A Spanish commercial ordinary Portland cement (OPC) type CEM I 52.5 R according to the existing European standard was used. All cement particles were below 63 μm. This cement was supplied by Lafarge cement Company (Villaguenga de la Sagra, Toledo). The chemical composition shows in Table 1.
- The blended cements were prepared by replacing 10% and 20% by mass of the cement by an activated bamboo ash. The preparing, curing and specifications of BLA blended cements were carried out in accordance with current European standards [6,20].

2.2. Methods

2.2.1. Pozzolanic activity method

The pozzolanic activity of bamboo leaf ashes was studied by using an accelerated chemical method. The test consists in putting the bamboo leaf 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.

To quantify the kinetic parameters (D_e – effective diffusion coefficient, K – reaction rate constant and τ – time constant) of this pozzolanic reaction in pozzolan/$Ca(OH)_2$ system, a kinetic–diffusive model was used. Further explanations about the model can be found in previous works [11,21,22].

2.2.2. 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. 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 [23]. 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 analysis (TG/DTG), 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. Fourier transformed infrared analysis (FTIR) was performed using a spectrometer Thermoscientific NICOLET 6700 (Waltham, USA). Specimens were prepared by mixing 1 mg of the sample in 300 mg of KBr. The spectral analysis was performed in the range 4000–400 cm^{-1}.

3. Results and discussion

3.1. Chemical and mineralogical composition of bamboo ashes

3.1.1. Chemical composition

Table 1 shows the chemical compositions by XRF corresponding to the BLA and OPC samples. The activated bamboo ash analysed for this study was mainly formed by SiO_2, followed by CaO, MgO and K_2O. The other oxides (Al_2O_3, Fe_2O_3, Na_2O, TiO_2, P_2O_5 and SO_3) were present in concentrations equal or below to 1.01%.

The chemical values are not in agreement with those published previously with an Indian bamboo ash [16], even some differences can be found in bamboo ashes coming from the same Brazilian region, mainly in the content of SiO_2 (80.4%), MgO (0.99%), CaO (5.06%) and K_2O (1.33%) [18].These differences would be related to the different species of bamboo, soil, climate and age.

3.1.2. Mineralogical composition

Fig. 1 illustrates the XRD pattern corresponding to the bamboo ash analysed. This ash showed clearly a practically amorphous nature, which corresponds with the broad band localised between 15° and 40° 2θ. Mineralogically, calcite ($CaCO_3$) as main crystalline component was identified by characteristics peaks at 29.8°, 39.4°, 43.2°, 47.4° and 48.5° 2θ, followed by traces of cristobalite (SiO_2) with reflections at 21.9°, 28.3° and 36.1° 2θ and calcium sulphate ($CaSO_4$) at 25.4° and 31.3° 2θ. Also, traces of graphite (26.6° 2θ) and quartz (26.7° 2θ) can be present in starting sample, but their reflection peaks are overlapped (neither of them indentified in XRD pattern). This mineralogical composition varies substantially of the Indian bamboo ash analysed by Singh et al. [17], reporting quartz as the main crystalline compound and traces of calcite. The FTIR spectrum of the starting bamboo leaf ash (Fig. 2) showed two main zones of infrared absorption: a zone between 3600 and 3000 cm^{-1} and other zone at lower frequencies between 1700 and 500 cm^{-1}. The absorption band of vibration of O—H (3436 cm^{-1}) and the deformation band of H—O—H (1635 cm^{-1}) confirmed the presence of not structurally combined water. The FTIR frequencies at 1443 and 875 cm^{-1} (this last one was not identified in the figure) are typical bands assigned to C—O bonds of carbonates (calcite). Two bands centred at 1100 and 469 cm^{-1} were assigned to stretching and bending vibrations (Si—O) in the SO_4 tetrahedra, which are due to the presence of traces of crystalline mineralogical phases such as quartz and cristobalite and, mainly of amorphous silica present in the BLA. As a consequence of this, the band at 801 cm^{-1} can be assigned mainly to amorphous silica [24].

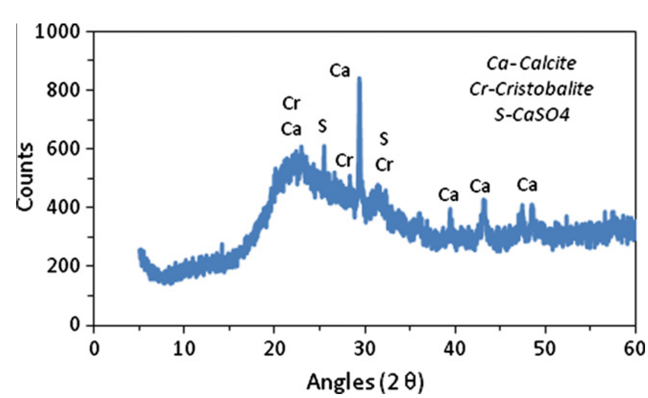

Fig. 1. XRD pattern of bamboo leaf ash (BLA).

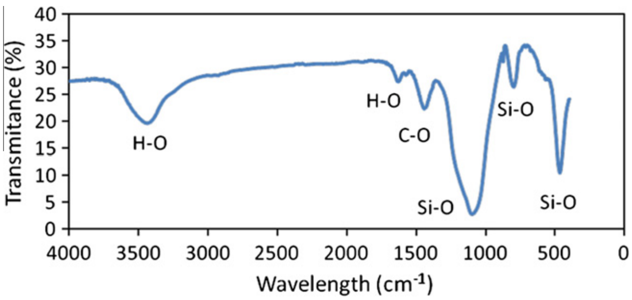

Fig. 2. FTIR spectrum of BLA.

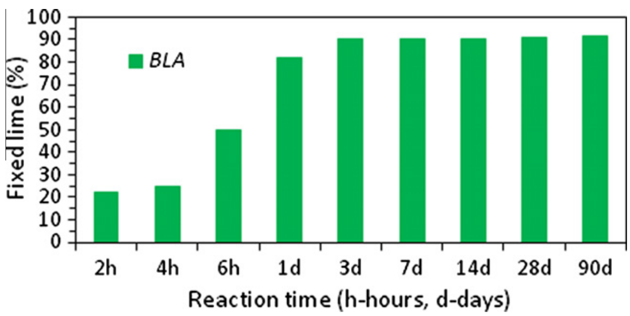

Fig. 3. Evolution of fixed lime in BLA/$Ca(OH)_2$ system.

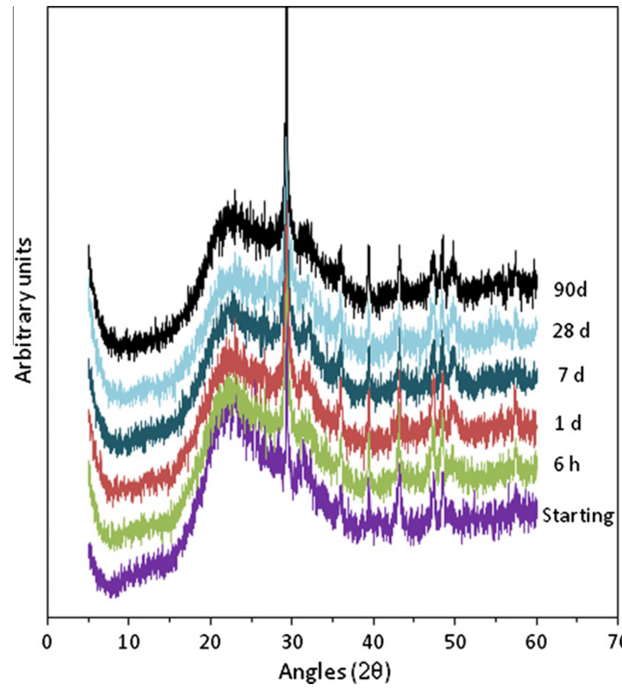

Fig. 4. Pozzolanic reaction evolution by XRD.

3.2. Pozzolanic behaviour of bamboo ash

The pozzolanic activity of the bamboo ash was evaluated by the determination of fixed lime percentages with respect to the initial total $Ca(OH)_2$ content (17.68 mmol/l) up to 90 days of reaction (Fig. 3).

The bamboo ash obtained under controlled conditions in laboratory showed a very high reactivity according to the values of fixed lime. At 6 h of reaction, BLA fixed a 50% of the total lime content in solution, passing to 82% and 90% at 1 and 3 days, respectively. There is virtually no change in lime content after 3 days, so the pozzolanic reaction had practically finished, with only little change thereafter (91.4% at 90 days).

These values indicate that the bamboo ash is a very reactive pozzolan during the initial 24 h of reaction, with similar pozzolanic behaviour to the silica fume used in the commercial cement manufacture [25] and with higher activity than the ashes from activated sugar cane wastes [12,26].

The quantification of the kinetic parameters in BLA/$Ca(OH)_2$ system was determined by using a kinetic-diffusive model, according to the described by Villar et al. [27]. The values of the τ parameter (constant of time), reaction rate constant (K), free energy of activation (ΔG),correction term to account for the remaining concentration of $Ca(OH)_2$ that is not consumed in the reaction (C_{corr}), correlation coefficient (r), coefficient of multiple determination (R^2) and residual sum of squares (RSS) are given in Table 2. According with the values of the kinetic parameters, it was possible to highlight that bamboo leaf ash calcined at 600 °C had a very high reactivity (order of 10^{-1}/h and free energy of activation around 80 kJ/mol). This reactivity is one order of magnitude greater than those obtained for rice husk ash (RHA) and sugar cane straw ash (SCSA) and two orders greater (in the value of the reaction rate constant) than sugar cane bagasse ash (SCBA), which are considered in the technical literature to be highly pozzolanic materials [10,11,28].

This pozzolanic behaviour of the bamboo ash is related to a rapid reaction between the amorphous SiO_2 of the BLA and the calcium hydroxide, forming CSH gels as unique hydrated phase. By its noncrystalline nature this hydrated phase was not identified by XRD patterns (Fig. 4). A part of the mineralogical compounds identified in starting BLA, a decrease in the band intensity localised between 15° and 30° 2θ (amorphous material) was observed with the increasing hydration reaction. However, the use of the other techniques such as TG/DTG and SEM/EDX are suitable for the identification of this kind of gels:

(a) Thermogravimetric analysis (TG/DTG) carried out in BLA/$Ca(OH)_2$ system showed an important weight loss between 100 and 300 °C of temperature (Table 3), main zone of dehydroxilation of the main hydrated phases produced during pozzolanic reaction such as silicates, aluminates and aluminium silicate hydrates [29]. According to DTG curves (Fig. 5), a wide band was observed in the first 6 h, increasing its intensity with hydration time. This band localised at 110 °C was attributed to CSH gels.

(b) The combination of SEM and EDX techniques afforded information on the morphology, texture and chemical composition of the hydrated phases. As Fig. 6 for 28 days shows, it confirmed the presence of CSH gels in BLA/$Ca(OH)_2$ system (Fig. 6 right), showing irregular forms not well defined and with very small dimensions, only observed at high

Table 2
Reaction rate constants, τ parameter, C_{corr} parameter, free energy of activation and statistical parameters for bamboo leaf ash calcined at 600 °C.

Material	τ (h)	K (h^{-1})	ΔG (kJ/mol)	C_{corr}	r	R^2	RSS
BLA	7.8 ± 0.4	(1.26 ± 0.08) × 10^{-1}	78.65 ± 1.42	1.75 ± 0.13	0.9981	0.9974	0.0352

Table 3
Evolution of weight losses (%) between 100 and 300 °C with hydration time.

	Starting	6 h	1 d	7 d	28 d	90 d
BLA	0	1.22	2.04	2.64	2.79	3.41

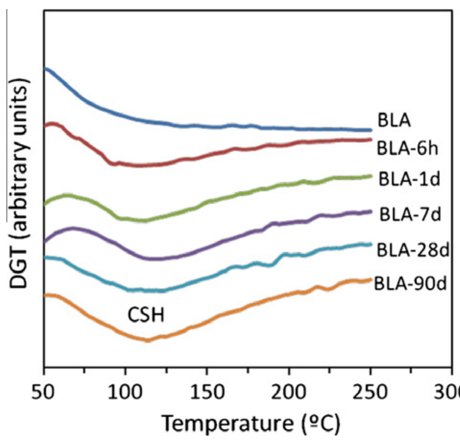

Fig. 5. DTG curves in the 50–300 °C interval.

magnifications. A CaO/SiO_2 ratio of 0.92 was identified, which corresponds to type I CSH gels, according to the Taylor classification (between 0.8 and 1.5) [30].

3.3. BLA-blended cement behaviour

3.3.1. Rheological properties

The studies of rheological properties focused on water content at normal consistency and the determination of setting times, according to European standards [31]. The blended cements selected for this study were elaborated with 10% and 20% replacements of OPC by BLA.

According to the results obtained (Table 4), the incorporation of BLA modified substantially the water content in blended cement pastes with respect to the control paste necessary to reach a normal consistency. The water demand increased with the increasing BLA content. Assigning the 100% for the OPC water content, the addition of 10% and 20% BLA increased the water demand by 19% and 46%, respectively. This behaviour was similar to other pozzolans such as: silica fume, metakaolin, bagasse sugar cane, rice husk ash, paper sludge, ceramic residues [1,32–34].

With respect to the results obtained for setting times (±15 min), it was clearly detected that the incorporation of BLA to cement pastes did not modify initial and final setting times substantially. Compared to the control paste, a slight delay of setting times was detected when 20% BLA ash was incorporated to the cement paste. This fact could be related to the presence of minimum contents of ZnO and PbO in BLA (0.07% and 0.01% respectively), oxides well known by their retarding effect in the setting times.

Table 4
Evolution of water content (g), setting times (±15 min) and volume stability (mm).

	W content	Initial set	Final set	Expansion
OPC	149	140	245	0
OPC + 10% BLA	178	155	245	0
OPC + 20% BLA	218	175	275	0

3.3.2. Volume stability

The purpose of the volume stability test is to estimate the potential risk of delayed expansion due to hydration of free calcium oxide (CaO) and magnesium oxide (MgO) in form of periclase. The expansion test was carried out according to the standard EN 196-3 [35]. Table 4 shows the results obtained for the different cements selected (average of three measurements), what indicated that the BLA blended cements showed volume stability similar to the control paste. Therefore, these expansive compounds (quicklime and periclase) are not present or they are in insignificant amounts in the bamboo leaf ashes. According to Taylor [1], if the MgO content exceeds about 1.5%, this will form small crystals of periclase, which is an expansive phase at long term.

3.3.3. Compressive strength evolution

The effect of the percentage of BLA on compressive strength of blended cement mortar (average of six specimens) is depicted in Fig. 7. These mortars were prepared with a sand/binder ratio of 3/1 and a water/binder ratio of 0.5 for the preparing of $4 \times 4 \times 16$ cm specimens, according to the existing EN standard [35]. At 7 days of reaction, BLA blended cement mortars showed a slight decreasing of the compressive strength values with respect to the control mortar. The strength loss was of 1.2% and 6.7% for 10% and 20% of BLA respectively, representing minimum values if compared with the magnitudes of substitution percentages of BLA. For longer hydration times, compressive strength values were very similar to the value corresponding to the control mortar, independently of the added percentage. At 28 and 90 days of curing respectively, the strength losses for blended cements elaborated with 10% and 20% of BLA were only of 1% and 2.8% with respect to the control mortar.

This favourable behaviour of blended cements at different ages was related to the high pozzolanic rate of bamboo ashes, transforming the portlandite generated during the OPC hydration into CSH gels with hydraulic properties. These results did not match with those reported by Singh et al. [17] who studied the strength behaviour of 20% BLA blended cement mortars up to 28 days of reaction, showing lower values than control mortar. This different behaviour found for this kind of ash was due to the lower silica content (75.90%) and pozzolanic activity (only 80% by mass of fixed lime at 28 days) and, probably a less fineness of BLA used.

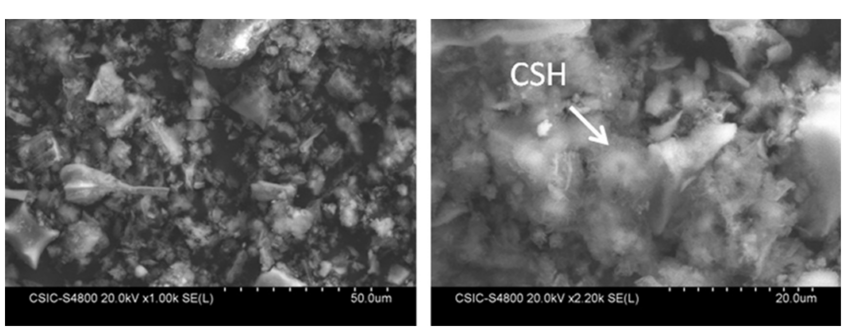

Fig. 6. Micrographs showing a general aspect (left) and CSH gels (right) at 28 days.

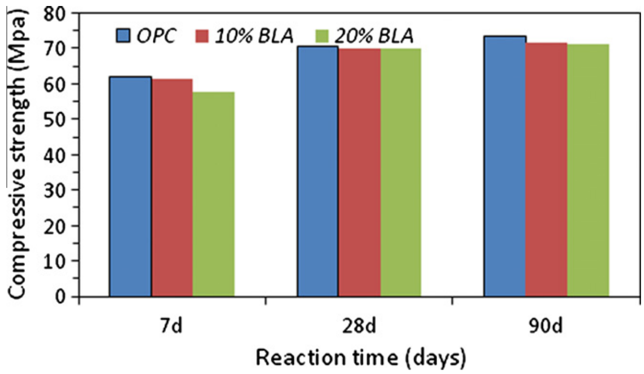

Fig. 7. Compressive strength evolution in BLA blended cement mortars.

4. Conclusions

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

- The bamboo leaf ash was formed by 78.7% of SiO_2 as the main oxide.
- A mineralogical study of BLA by XRD revealed the presence of amorphous silica mainly and, traces of calcite, cristobalite and calcium sulphate.
- According to the results obtained from pozzolanic activity method, the BLA was a very reactive pozzolan comparable to the commercial silica fume. At 3 days of reaction, the ash had consumed practically all the available $Ca(OH)_2$ in dissolution. The reaction rate constant K was of the order of 10^{-1}/h, value greater than other agricultural wastes (such as RHA, SCSA and SCBA) considered highly pozzolanic materials. The reactivity is one order of magnitude greater than that of RHA and two orders greater than that of SCBA.
- TG/DTG and SEM/EDX analysis confirmed the presence of type I CSH gels as unique the hydrated phase produced during the pozzolanic reaction in BLA/$Ca(OH)_2$ system.
- The elaboration of blended cements with 10% and 20% of BLA increased the water content to get a normal consistency, the setting times were similar to the control paste and finally, these cements did not show volume instability, due to absence of expansive compounds such as quicklime and periclase in BLA.
- The BLA blended cement mortars showed compressive strength values practically equal to the control mortar from 28 days of curing, in spite of the fact the BLA showed high water demand.
- Portland cements elaborated with bamboo leaf ashes (10% and 20%) comply with the physical and mechanical requirements, according to existing EN 197-1 standard.
- The results obtained in the present work highlighted that the use of bamboo leaf activated at 600 °C and 1.20 h of retention is a suitable pozzolan for the manufacture of future type II/A commercial cements. However, this good behaviour of LBA as pozzolan will be related to the different factors, including origin of bamboo leaves, chemical composition, calcining conditions, and amorphous nature of the ash.

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