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Pozzolanic behavior of bamboo leaf ash: Characterization and determination of the kinetic parameters

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ABSTRACT

The paper presents a characterization and study of the pozzolanic behavior between calcium hydroxide (CH) and bamboo leaf ash (BLAsh), which was obtained by calcining bamboo leaves at 600 °C for 2 h in a laboratory electric furnace. To evaluate the pozzolanic behavior the conductometric method was used, which is based on the measurement of the electrical conductivity in a BLAsh/CH solution with the reaction time. Later, the kinetic parameters are quantified by applying a kinetic-diffusive model. The kinetic parameters that characterize the process (in particular, the reaction rate constant and free energy of activation) were determined with relative accuracy in the fitting process of the model. The pozzolanic activity is quantitatively evaluated according to the values obtained of the kinetic parameters. Other experimental techniques, such as X-ray diffraction (XRD) and scanning electron microscopy (SEM), were also employed.

The results show that this kind of ash is formed by silica with a completely amorphous nature and a high pozzolanic activity. The correlation between the values of free energy of activation ($\Delta G^{\#}$) and the reaction rate constants (K) are in correspondence with the theoretical studies about the rate processes reported in the literature.

1. Introduction

Natural pozzolans by themselves possess little or no cementing value, but finely ground in the presence of moisture, they will chemically react with calcium hydroxide at ordinary temperature to form hydrated phases possessing cementing properties [1,2]. Nowadays, some industrial by-products and wastes are attracting much research because of their high silica and/or alumina content for the use as additives in commercial Portland cements. It is well-known that hydrated phases formed during pozzolanic reaction commonly improve the performance of concrete [3–7].

The use of waste materials with pozzolanic properties in concrete production is a worldwide practice. It is known that the incessant generation of solid waste materials represents serious environmental and technical problems. Also, the assessment of the pozzolanic activity of cement replacement materials is becoming increasingly important because of the need for more sustainable cementing products.

In recent years, the use of solid waste derived from agriculture as pozzolans in the manufacture of blended mortars and concrete has been the focus of new research [8–12]. In fact, the addition to concrete of ashes from the combustion of agricultural solid waste is, at present, a frequent practice because of the chemical reactivity of the ashes with the portlandite generated during the cement hydration reaction. Some of these agricultural wastes such as rice husk ash, coconut pith, sawdust, cork granules, wheat straw ash, sugar cane bagasse ash and sugar cane straw ash, are being tested for mortar and concrete production [13–17].

It is known that the use of vegetable fibre reinforcement of cementing materials provides useful results. The bamboo fibres are exceptional in this respect, not only because of their eco-friendly nature but also because they provide the most economic and socially useful outlet for bamboo chips or clipping [18–22]. Fibre reinforcement of cementing materials still remains an exciting and innovative technology because of the basic engineering properties of crack resistance, ductility and energy absorption that it imparts to concrete—properties that promote a long, trouble-free service life [23].

Bamboo is probably the fastest-growing and highest yielding natural resource and construction material available to mankind. However, the use of bamboo generates other residues not used as fibres, such as the bamboo leaf. In some countries, significant amounts of bamboo are processed, generating high volumes of

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solid waste. Brazil, for example, is the main bamboo producer country in Latin America and in the Amazonia region alone the bamboo growing area is close to 180,000 km^2. In Brazil, paper production alone consumes about 500 ktonnes/year of cultivated bamboo, which generate 190 ktonnes/year of agricultural wastes. These wastes are often burnt in open landfills, negatively impacting the environment.

In the literature, the studies about the pozzolanic properties of bamboo wastes are scarce. Little research has been carried out to study the bamboo leaf waste as a pozzolanic material [24,25]. Dwivedi et al. [24] reported the reaction between calcium hydroxide (CH) and bamboo leaf ash for 4 h of reaction, using the differential scanning calorimetry (DSC) technique, while Singh et al. [25] studied the hydration of bamboo leaf ash in a blended Portland cement. These studies concluded that bamboo leaf ash is an effective pozzolanic material. When 20 wt.% of bamboo leaf ash was mixed with OPC the compressive strength values of mortars at 28 days of hydration were found to be quite comparable to those of OPC.

Nevertheless, the methods for evaluating pozzolanic activity used in the aforementioned research were, in the majority of the cases, focused on the qualitative aspect of the behavior of the pozzolanic materials rather than the quantitative aspect of the pozzolan-lime reaction (computation of kinetic coefficients). At present, researchers are beginning to focus on known kinetic coefficients as an acceptable and rigorous criterion for evaluating the pozzolanic activity of the materials. Recently, Villar-Cociña et al. [26,27] proposed a new kinetic-diffusive model that allows characterizing the pozzolanic activity of sugar cane waste mixed with clay for all ages of the reaction by computing the kinetic coefficients (reaction rate constant, fundamentally) of the CH/sugar cane-clay ash reaction. The results obtained showed a good correlation between the experimental and theoretical data.

The present paper shows the chemical and mineralogical characterization (XRF, XRD, SEM-EDX) of bamboo leaf ash and for the first time, the kinetic parameters are calculated for the bamboo ash/lime system. The electrical conductivity is recorded experimentally which can be easily determined and correlated with the $Ca(OH)_2$ concentration. The kinetic-diffusive model fitted by mathematical methods makes it possible to determine the kinetic parameters (reaction rate constant and free energy of activation) and the pozzolanic activity of these materials in a rigorous way.

2. Experimental

2.1. Materials

The bamboo leaves were recollected in the vicinity of the Faculty of Animal Science and Food Engineering of the University of São Paulo, Pirassununga, Brazil. The leaves were dried in the sun. Fig. 1 shows a photograph of a bamboo leaf, which contains two parts: the mesophyl cell and the parallel veins. The bamboo leaf ashes were obtained in a laboratory electric furnace at 600 °C calcining temperature for 2 h of retention. Once calcined, the ashes were ground and sieved below 90 μm, fineness similar to that of Portland cement. The ash showed a gray color. Fig. 2 shows the ash used in the current study. The particle size distribution was determined using a Shimadsu Mod. SALD-201V particle size analyzer. According with the granulometric curve (Fig. 3), the ashes have a fine granulometry with grain size between 1 and 100 μm and average size (D_{50}) of 58.1 μm.

Fig. 1. Appearance of the bamboo leaf.

Fig. 2. Appearance of the bamboo leaf ash.

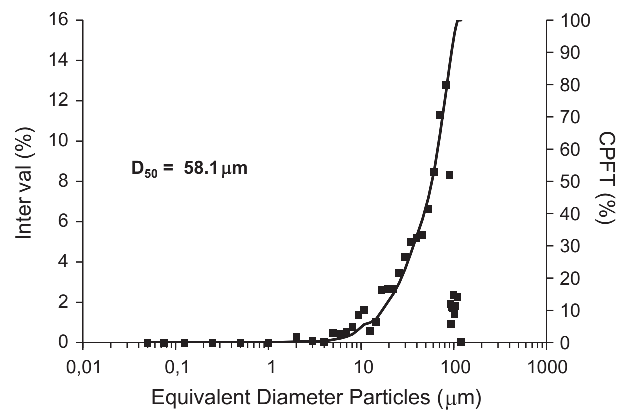

Fig. 3. Particle size distributions for BLAsh samples.

A saturated solution of calcium hydroxide, prepared with deionized water and $Ca(OH)_2$ (95% minimum purity), was used. To obtain the solution, the $Ca(OH)_2$ in excess was mixed with deionized water and stirred for 2 h, after which the solution was maintained at rest for 24 h. Thereafter, the solution was filtered and it was valued with hydrochloric acid (HCl). The obtained concentration was of 0.040 mol/L. The initial conductivity obtained was 7.33 mS/cm.

2.2. Test methodologies

2.2.1. Pozzolanic activity

To carry out a qualitative or quantitative determination of pozzolanic activity many experimental methodologies have been developed [17,28,29]. In this work, as in other studies carried out by the authors [26,30], a conductometric method was used in order to study the pozzolanic activity of these materials. This method follows the conductivity of the pozzolan–calcium hydroxide solution with reaction time.

Hundred milliliter of saturated $Ca(OH)_2$ solution were mixed with 2.10 g bamboo leaf ash (which is the proportion commonly found in the literature for similar experiments) and magnetically stirred. Immediately after the ash was mixed with the CH solution, the conductivity measurements began. The measurements of conductivity were made in a Digimed (DM-32) microconductimeter at room temperature (∼26 ± 1 °C) at different times. To correlate the CH concentration with the conductivity of the CH solution a calibration curve was applied [26].

2.2.2. Mathematical model

A kinetic-diffusive model [13,26] is used to describe this pozzolanic reaction in a pozzolan/CH solution system. The model is:

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

where D_e is the effective diffusion coefficient, K is the reaction rate constant, C_0 is the initial conductivity of the solution and τ is a constant of time (time interval in which the radius of the nucleus of pozzolan diminishes to 37% of the initial radius of the average size particle, r_s). The correction term (C_{corr}) was added to the model to account for the remainder concentration of CH that is not consumed in the reaction. In some systems the CH is not consumed totally.

The dimensionless magnitude $\xi = (C_0 - C_t)/C_0$ represents the relative loss of conductivity and C_t represents the absolute loss of conductivity with time for the pozzolan/CH system.

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 slowest) control the process. Accordingly, it is possible in certain cases to have different behaviours: Diffusive (described by the 2nd term of Eq. (1)), kinetic (3rd term) and kinetic-diffusive (both terms). Further explanations of the model can be found in the Refs. [26,27].

3. Results and discussion

3.1. Bamboo leaf ash characterization

The chemical composition of the ash was determined by the X-ray fluorescence (XRF) technique by using a PANalytical Axios XRF Spectrometer. Table 1 shows the main elements (expressed as oxides) present in bamboo ash. Silica (SiO_2) is the major component in ash, following by CaO, K_2O, Al_2O_3 and SO_3 in concentrations of 5.1, 1.3, 1.2 and 1.1 respectively. Oxides as Fe_2O_3, MgO, P_2O_5 and MnO show contents below 1% and the rest of oxides (Na_2O, ZnO) are present in percentages under 0.1%. Loss on ignition (LOI) was determined by weight loss of dry ash after 1 h at 1050 °C.

The mineralogical composition was studied by XRD (X-ray diffractometer Phillips MPD 1880). The results for this ash are illustrated in Fig. 4. Bamboo ash shows a highly amorphous nature, which corresponds with the broad band localized about 20–30° 2θ. The presence of crystalline minerals was not detected. The band form of this ash is similar to that showed by silica fume, a highly active addition used normally for high performance concrete manufacture [31].

A detailed observation of the ash by SEM (FEI Quanta 600 FEG scanning electron microscope) shows a regular morphology with some smooth surfaces particles on the top of it (Fig. 5). The different EDX microanalyses carried out on the ash sample show similar

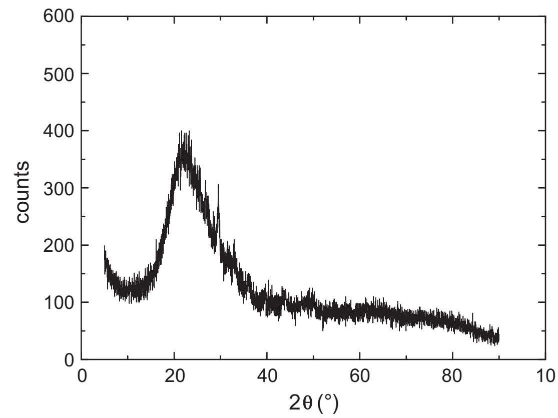

Fig. 4. XRD pattern of the bamboo leaf ash.

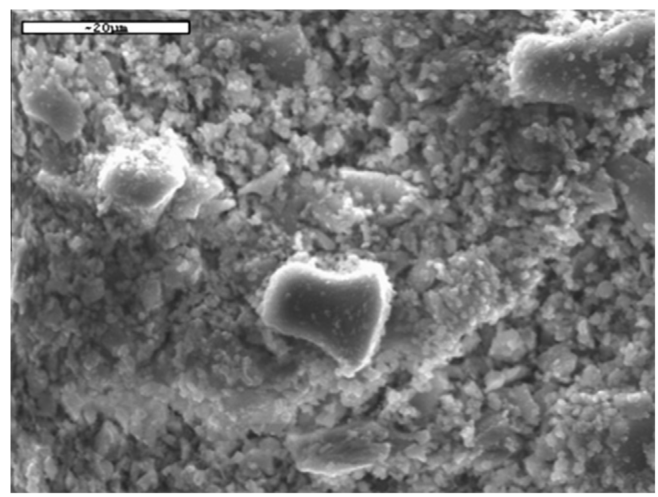

Fig. 5. Morphology of bamboo ash.

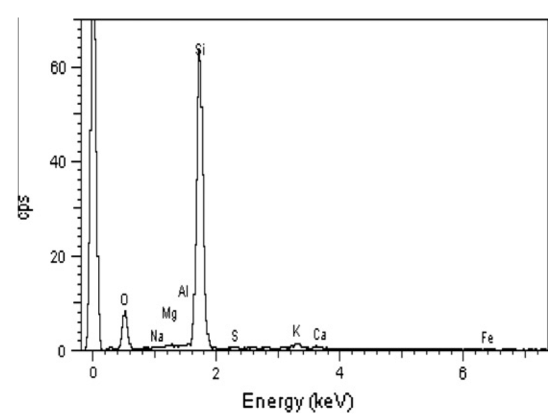

Fig. 6. EDX microanalysis of the ash.

Table 1
Chemical composition of the BLAsh.

SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3	P_2O_5	MnO	ZnO	LOI
Chemical composition (oxides (%))											
80.4	1.22	0.71	0.99	5.06	0.08	1.33	1.07	0.56	0.20	0.07	8.04

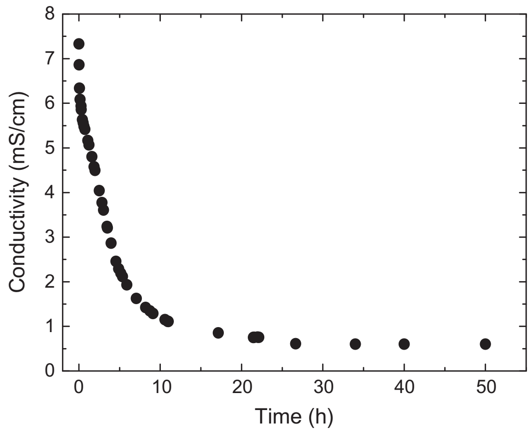

Fig. 7. Curve of conductivity versus reaction times.

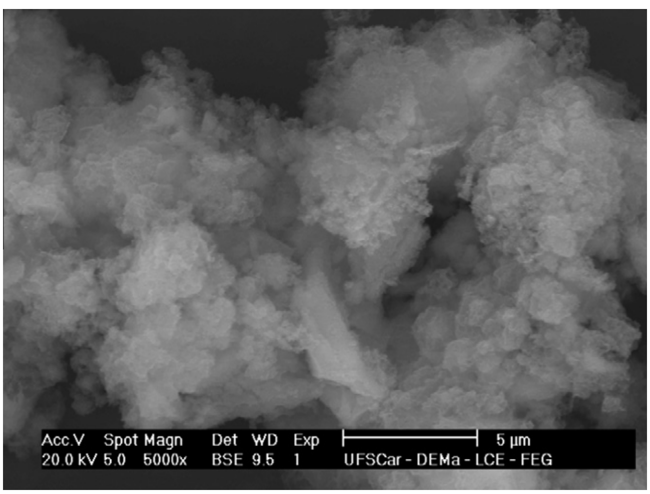

Fig. 8. C–S–H gels morphology by SEM.

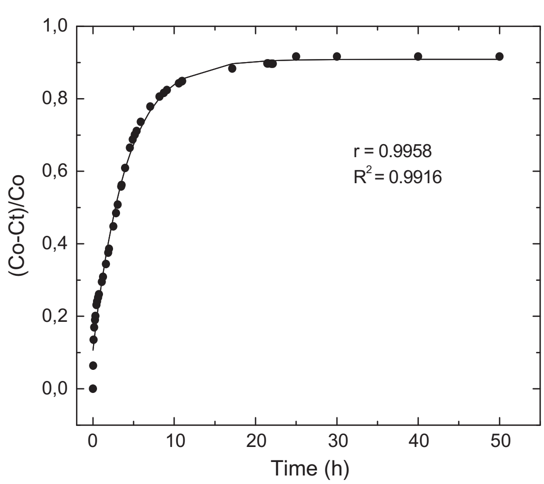

Fig. 9. Relative loss of conductivity versus reaction times. Black circle (experimental), solid line (model).

chemical compositions. The enrichment in Si was present in all of them, as shown in Fig. 6. The microanalysis was taken of the central particle of photograph.

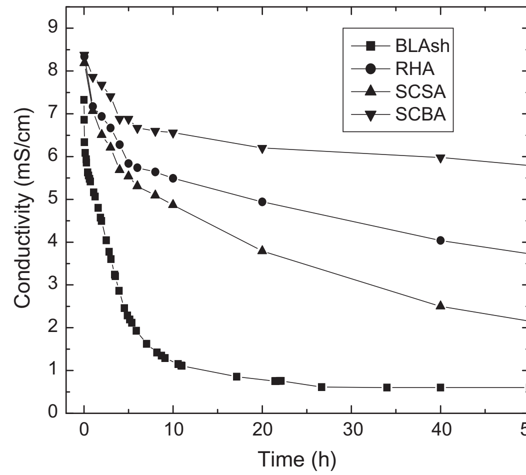

Fig. 10. Variation of conductivity with reaction time for rice husk ash (RHA), sugar cane straw ash (SCSA), sugar cane bagasse ash (SCBA) and bamboo leaf ash (BLAsh).

3.2. Evaluation of pozzolanic activity in the bamboo ash–lime system

The results obtained for pozzolanic activity are shown in Fig. 7 in which the conductivity variations versus reaction times (h) for the pozzolan/calcium hydroxide (CH) suspension are shown. A decrease of the electrical conductivity of the pozzolan/CH system is obtained. This behavior is only attributed to the pozzolanic reaction between amorphous silica and CH to give the formation of C–S–H gels, with the corresponding decrease of the CH concentration in the solution.

As a result of this pozzolanic activity, a considerable variation (loss) of conductivity in early ages is obtained. The stabilization of the curve is reached for long periods of time. This indicates the moment when the reaction has practically finished.

The SEM observation carried out after conductivity testing (Fig. 8) shows the formation of C–S–H gels, rough zones having a sponge-like morphology.

3.3. Application of the mathematical model: determination of the kinetic parameters

Shown in Fig. 9 is the relative loss of conductivity ξ versus time for the BLAsh/CH samples. Solid lines represent the curves of the fitted model. The fitting of the model (Eq. (1)) allowed us to determine the parameters τ, D_e, K and $\Delta G^{\#}$ in each case.

Fitting the relative loss of conductivity versus 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 sum of squares (RSS), residual scatter, residual probability and variance analysis, it can be concluded that: for BLAsh the kinetic control model shows the best correspondence with the experimental data. 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 the high porosity of the reaction product layer in this material, which facilitates a quick diffusion process.

In the current paper, only some statistical parameters (r, R^2, SE, RSS) are shown since the remaining ones (mentioned above) are related to graphic analyses and large tables that would significantly lengthen the paper. The values of the τ parameter, the free energy of activation $\Delta G^{\#}$ and the reaction rate constant K are given

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

Material (ash)	τ (h)	Reaction rate constant (h^{-1})	Free energy of activation $\Delta G^{\#}$ (kJ/mol)	C_{corr}	Correlation coefficient (r)	Coefficient of multiple determination (R^2)	Residual sum of squares
BLAsh	4.1 ± 0.2	(8.41 ± 0.29). 10^{-1}	73.89.1 ± 1.24	0.09 ± 0.008	0.9958	0.9916	0.0293

Table 3
Kinetic parameters for BLAsh, SCBA, SCSA and RHA.

Material (ash)	Reaction rate constant (h^{-1})	Free energy of activation $\Delta G^{\#}$ (kJ/mol)
BLAsh	(8.41 ± 0.29) × 10^{-1}	73.89.1 ± 1.24
SCSA	(4.27 ± 0.35) × 10^{-2}	102.13 ± 0.18
RHA	(1.73 ± 0.12) × 10^{-2}	104.38 ± 0.16
SCBA	(8.04 ± 0.34) × 10^{-3}	106.31 ± 0.10

in Table 2. In Fig. 9, the correlation and multiple determination coefficients r and R^2 are shown.

One of the most important parameters that characterizes the reactivity of the materials is the thermodynamic parameter of activation called free energy of activation. This parameter can be calculated by using the Eyring equation of "The absolute theory of rate processes" [32], which is given by:

$$K = \frac{k_B T}{h} \exp\left(-\frac{\Delta G^{\#}}{RT}\right) \quad (2)$$

where k_B is the Boltzman constant, h is the Planck constant and R is the universal gas constant. T is the temperature, K is the reaction rate constant, and $\Delta G^{\#}$ is the free energy of activation, which characterizes the change of free energy of the system when passing from the initial state to the transition state.

It is well-known that large values of $\Delta G^{\#}$ are correlated with stability kinetics, i.e. low reactivity (small values of K). On the other hand, small values of $\Delta G^{\#}$ are correlated with instability kinetics, i.e. high reactivity (large values of K). The knowledge of $\Delta G^{\#}$ allows us to characterize how fast the reaction occurs; for large $\Delta G^{\#}$ values the reaction will be slower. The literature has reported values of free energy of activation only for some specific systems [33–34]. For the BLAsh–lime system, values of activation parameters of the pozzolanic reaction have not been reported.

In this research, the free energies of activation were calculated by substituting Eq. (2) in the model (Eq. (1)). The resultant equation was fitted to the experimental data (relative loss of conductivity versus reaction time) and the values of $\Delta G^{\#}$ were determined. The values of $\Delta G^{\#}$ so obtained are given in Table 2. The K and $\Delta G^{\#}$ values provide a direct index of the pozzolanic activity of the materials.

Fig. 10 and Table 3 show a comparison (taking into account the variation of conductivity with time and the kinetic parameters respectively) of BLAsh with other pozzolanic agricultural residues reported in the literature [26,30].

According with the values of the kinetic parameters, it is possible to conclude that BLAsh calcined at 600 °C had a higher reactivity (order of 10^{-1}/h and free energy of activation around 75 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 by-products [26,30].

4. Conclusions

From the results obtained in this paper, the following conclusions can be raised:

1. Chemical composition by XRF shows that bamboo ash basically is formed by silica in concentrations of about 80%. The rest of the oxides are present in low concentrations.
2. The controlled calcining of bamboo leaves at 600 °C for 2 h represents suitable conditions to get a totally amorphous material.
3. The pozzolanic activity of ash characterized by means of the electric conductivity method shows a high activity at early ages.
4. The kinetic-diffusive model used in the present paper allows describing the pozzolanic reaction kinetics in the CH/BLAsh system by previously determining the kinetics coefficient (reaction rate constants and free energy of activation). The reaction rate constants jointly with the free energy of activation give a precise index of the reactivity or pozzolanic activity of the materials under analysis.
5. The values of the reaction rate constant and free energy of activation, obtained in the fitting process of the kinetic-diffusive model state that the bamboo leaf ash calcined at 600 °C has high pozzolanic reactivity.
6. Future studies include the analysis of the influence of the calcining temperature on the pozzolanic activity of these agricultural wastes. Also, environmental durability and strength of the resulting mortars remain to be assessed.

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