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Influence of calcining temperature on the pozzolanic characteristics of elephant grass ash

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ABSTRACT

The influence of calcining temperature on the pozzolanic properties of elephant grass ash (EGA) for application as a supplementary cementitious material is reported. Five different calcining temperatures were used (ranging from 500 to 900 °C for 3 h at 100 °C increments) after a first calcining step at 350 °C for 3 h, a 10 °C/min heating rate, and a 0.04 constant volumetric ratio between the sample and the internal furnace chamber. After calcining and high energy grinding, all ashes were characterized based on particle size distribution, oxide composition, loss on ignition, B.E.T. specific surface area, X-ray diffraction, and scanning electron microscopy. The pozzolanic behavior was investigated based on pozzolanic activity index test and compressive strength of concretes up to 180 days of curing. An expressive decrease in loss on ignition values and, consequently, increase in silica content of EGA produced at higher temperatures were observed. Overall, the results demonstrated that 600 °C was the most suitable temperature for producing EGA. Additionally, the replacement of 20% (in volume) of cement by 600 °C-calcining EGA did not change significantly the 28-day compressive strength of concrete, and increased the strength after 180 days of curing in relation to a reference concrete.

1. Introduction

Worldwide, the constant demand for concrete, as well as the environmental impact and large amounts of energy spent in its production, have stimulated the adoption of sustainable practices by the cement industry. Overall, the main practices considered to mitigate environmental damage associated with the Portland cement manufacture include increasing the energy efficiency of processes, promoting the use of renewable energy sources, and partially replacing cement by supplementary cementitious materials.

As regards the partial cement replacement by supplementary cementitious materials, silica-rich industrial and agricultural by-products have been extensively employed for decades, i.e., fly ash, silica fume, and rice husk ash (RHA). Recently, some studies have shown the feasibility of producing pozzolan from burning of sugar cane bagasse (SCBA) [1–3], sugar cane straw [4,5] and some types of grass used as biomass. For example, Nimityongskul et al. [6] studied a pozzolanic ash produced from vetiver grass (*Chrysopogon zizanioides*) for using in rural areas of tropical countries. In this case, chemical and physical properties of cementitious products with ashes produced from two distinct vetiver grass genotypes were investigated. Furthermore, it was observed that pozzolanic reactions formed hydrated compounds that increased the compressive strength and reduced the water absorption of mortars.

Wang et al. [7] investigated the calcining of switchgrass (*Panicum vigratum* L.), a low-carbon biomass fuel (carbon neutral in its life cycle), obtaining a pozzolanic ash and optimized energy output with a burning at 550 °C for 4 h. Cordeiro and Sales [8] examined the performance of elephant grass (*Pennisetum purpureum*) ash (EGA) after distinct grass pretreatments. The results indicated that the pozzolanic activity of ashes significantly increased when the grass was pretreated with acid leaching and washing in hot water to remove impurities. A study conducted by Nakanishi et al. [9] showed a similar behavior of EGA and silica fume in terms of fixed lime values, pointing to the pozzolanic activity of this material. EGA may be an interesting alternative for several countries, i.e. Brazil, where all fly ash and blast-furnace slag sources are already incorporated by the cement industry.

Elephant grass is a fast-growing biomass source [10], enabling up to two harvests per year. Seye et al. [11] highlighted the

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similarities between the sugar cane and the elephant grass since both species share similar morphological structures and chemical composition. For this reason, the ashes from these materials should likewise be expected to have similar properties, which might be an interesting aspect in the development of proper disposal strategies for EGA. In addition, the similarities with the sugar cane bagasse suggest the existence of optimal calcining conditions for production of pozzolan from elephant grass. In this context, the present work evaluated the influence of the calcining temperature of elephant grass on the pozzolanic characteristics of EGA, considering percentage of oxides, loss on ignition, X-ray diffraction, B.E.T. specific surface area, and pozzolanic activity index with cement Portland tests. Furthermore, the performance of a selected EGA was compared with a pozzolanic SCBA in a structural concrete by compressive strength tests up to 180 days of curing.

2. Materials

The elephant grass used was collected in a ceramic plant located in the city of Campos dos Goytacazes (Rio de Janeiro, Brazil), where it is used as biomass in continuous furnaces for production of bricks and tiles. Ordinary Portland cement, Brazilian standard sand [12], and deionized water were used to determine the pozzolanic activity index. High early strength Portland cement, carboxylic ether polymer superplasticizer (32.6% oven-dried residue), water, and locally available fine (river sand of 4.8 mm maximum size and 1.93 fineness modulus) and coarse (crushed charnockite of 9.5 mm maximum size and 5.75 fineness modulus) aggregates were used for concrete production. A pozzolanic SCBA produced under controlled calcining conditions [13] was used to compare the effect of EGA on the concrete compressive strength. Table 1 summarizes the percentage of oxides, loss on ignition and some physical characteristics of SCBA and high early strength cement.

3. Methods

3.1. Production of EGAs

Elephant grass was mowed and homogenized using a knife mill (Thomas). Then, the air-dry material was divided into five parts, each burned in an aired laboratory muffle furnace according to the two-step procedure. The first temperature was stipulated at 350 °C, while the second one ranged between 500 and 900 °C, at 100 °C increments. All calcining operations were conducted at 10 °C/min heating rate and 3 h residence time. Samples were burned in alumina containers and a 0.04 volumetric ratio was kept constant between the EGA sample and the internal furnace chamber. This calcining approach was previously used to produce optimized high amorphous silica content from SCBA [14].

The calcining procedures were confirmed considering thermogravimetric analysis (TGA) of elephant grass, by using a proximate analysis two-gas flow test. Duplicate analyses were conducted in a Hitachi STA7300 analyzer with approximately 15 mg sample into a platinum crucible. The sample was heated from 30 to 105 °C under 75 mL/min N_2 flow at 20 °C/min heating rate, and 5 min hold up time. Then, another heating step was performed from 105 to 950 °C at 25 °C/min, and 16 min hold up time, while the last 10 min was performed under oxidizing conditions (75 mL/min synthetic air flow). From the two-gas TGA, proximate analysis of EGA was quantified considering the percent values of moisture (M), volatile matter (VM) and ash content (A) according to Eqns. (1)–(3), respectively. In addition, fixed carbon (FC) was obtained by difference (Eqn. (4)). Table 2 shows the proximate analysis values of elephant grass.

$$M = \frac{m - m_{105}}{m} \times 100 \quad (1)$$

$$VM = \frac{m_{105} - m_V}{m} \times 100 \quad (2)$$

$$A = \frac{m_0}{m} \times 100 \quad (3)$$

$$FC = 100 - (M + VM + A) \quad (4)$$

Considering m the sample mass, m_{105} the sample mass after drying at 105 °C, m_V the sample mass after the complete volatilization (at about 38 min), and m_0 the sample mass after heating at 950 °C under an oxidizing atmosphere (at about 58 min). The values of m_V and m_0 were taken from TGA curve considering the peaks on differential thermogravimetric analysis (DTG) curve (Fig. 1).

In accordance with the TGA of elephant grass, 500 °C was the lower calcining temperature, and the maximum temperature of 900 °C was chosen based on literature data that indicated evidence of the initial crystallization of the silica present in samples of SCBA [14] around this temperature.

Calcined samples were ground in a planetary mill (Pulverisette 5, Fritsch) for 15 min using 40 g of EGA in a 500 cm^3 tempered tool-steel bowl filled with 82 alumina balls (12 mm diameter). This procedure was carried out in order to ensure similar particle size distributions to the EGA samples since pozzolanic activity is strongly dependent on the particle size [15]. After calcining and grinding, each EGA was denominated EGA***X***, where ***X*** indicates the temperature in which the second calcining step (500, 600, …, 900 °C) was performed.

3.2. Characterization of EGAs

A laser diffraction-type particle size analyzer (Mastersizer 2000, Malvern) was used to measure the particle size distribution. The percentage of oxides was determined by X-ray fluorescence spectroscopy (EDX-720, Shimadzu). Loss on ignition was obtained

Table 1
Percentage of oxides (in mass), loss on ignition, and some physical characteristics of high early strength cement and SCBA.

Characteristic	Cement	SCBA
SiO_2 (%)	15.5	69.6
Al_2O_3 (%)	4.5	15.7
Fe_2O_3 (%)	2.3	5.7
CaO (%)	71.1	1.3
K_2O (%)	0.6	2.2
SO_3 (%)	3.0	1.6
P_2O_5 (%)	–	0.9
TiO_2 (%)	0.4	0.9
Na_2O	0.4	0.1
MnO (%)	0.1	0.1
Loss on ignition (%)	2.1	2.1
Density (kg/m^3)	2960	2335
B.E.T. specific surface area (m^2/g)	–	25.0
D_{50} (μm)	17.0	7.4

Table 2
Proximate analysis of elephant grass.

	M (%)	VM (%)	FC (%)	A (%)
Elephant grass	3.1	67.4	19.8	9.7
Elephant grass (dry basis)	–	69.5	20.5	10.0

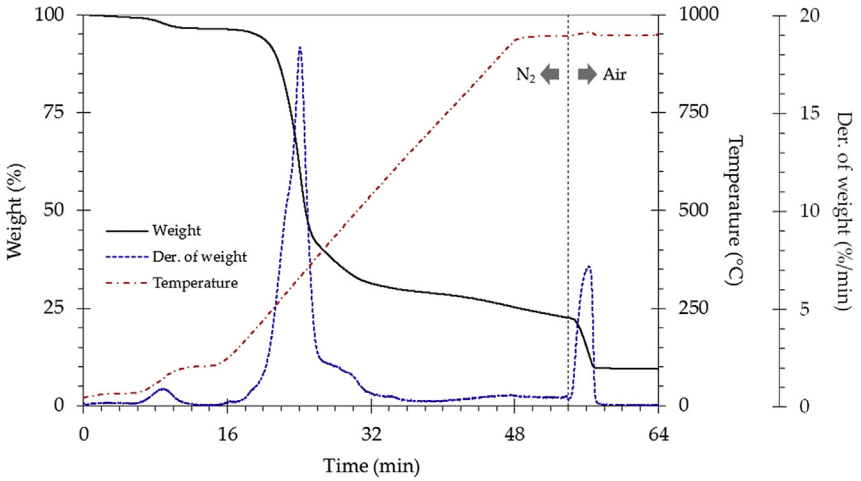

Fig. 1. TGA and DTG curves for elephant grass.

according to ASTM C114-15 [16]. The density and the B.E.T. specific surface area (by N_2 adsorption isotherm) values were determined using helium pycnometer (AccuPyc, Micromeritics) and physisorption analyzer (ASAP 2020, Micromeritics), respectively. The morphology of the EGA particles was observed in a scanning electron microscope (2000 FX, Jeol) operated at an accelerating voltage of 15 kV. Samples were sputtered with gold (approximately 5 nm).

The phase composition of the EGAs was examined by X-ray diffraction analysis (D8 Focus, Bruker). The generator settings were 35 kV and 40 mA (Cu–$K_{\alpha 1}$). Angular step of 0.02° and time step of 1 s from 10 to 60° were considered. A quantitative analysis of EGA600 was performed by X-ray diffraction (X'Pert MPD, Panalytical) with Cu–$K_{\alpha 1}$ radiation. In this case, the experimental conditions were: 45 kV and 40 mA; 2θ scanning range from 10 to 60°; angular step of 0.02°; time step of 20 s. The mineral present in the ash was identified referring to the ICCD powder diffraction database. The Highscore Plus Panalytical software, based on the Rietveld method, was used and the parameters refined included background, weight fractions, lattice parameters, specimen displacement, and the Lorentzian Scherrel broading component. Structural data for quartz and andesine were obtained from the ICDD data files. The peak profile was modeled using a pseudo-Voigt function. The GOF (goodness of fitting) parameter was below 3.0. The amorphous content was quantified by the difference in a 1:5 mix containing EGA and rutile (99.5% purity TiO_2) as an internal standard.

Pozzolanic activity was assessed by determining the pozzolanic activity index with Portland cement according to the Brazilian standard ABNT NBR 5752 [17]. Thus, mortars were produced with sand/cement and water/cementitious materials (w/cm) ratios of 3.0 and 0.52, respectively. Cement, standard sand, and water were used in a reference mortar. The other mixes were made with 35% (by volume) partial replacement of cement by each EGA***X***, besides the materials used in the reference. Then, the pozzolanic activity index was calculated using the ratio between the compressive strength of blended and reference mortars after 28 days of curing at 40 °C.

3.3. Application in concrete

A reference concrete was mix-designed using a software (BetonLab Pro2) in accordance with the Compressible Packing Model [18] for a 35 MPa compressive strength on a 28-day basis, and a fixed slump [19] ranging from 120 to 160 mm. Both CCE600 and SCBA were used in 20% (by volume) partial replacement of Portland cement. Consequently, both blended mixes had higher w/cm ratio than the reference concrete. The compositions of the concretes for a constant volume of 1 m^3 are reported in Table 3.

The concretes were mixed for 8 min in a 154-L vertical-axis mixer. Cylindrical molds (15 cm height and 7.5 cm diameter) were used for compressive strength tests. The specimens were cast in two layers and compacted using a vibrating table. The fresh specimens were covered with plastic sheets to prevent evaporation. After 24 h, the samples were demolded and placed in a lime-saturated water curing at room temperature until the test ages (7, 28, and 180 days). Before the strength tests, the bases of the cylinders were surface-ground with a wet diamond wheel. Then, four specimens for each mix were tested by compression using a servohydraulic machine (UH-F500kNI, Shimadzu) with 0.5 mm/min axial displacement rate. Compressive strength data were analyzed by means of Analysis of Variance (Anova) and Test of Tukey at a significance level of 5%.

4. Results and discussion

The laser particle size distribution curves of different EGA samples are shown in Fig. 2. It was observed that the high energy grinding in the planetary mill promoted a similar size distribution of the different samples, resulting in D_{50} (50% passing) values around 10 μm (from 7.6 to 13.2 μm). The equalization of particle size distribution was essential to compare the different ashes since particle size strongly influences the pozzolanic activity of the material. By comparison with SCBA, a D_{50} of 10 μm was desirable to confer high reactivity to this biomass ash [20].

Table 4 shows the percentage of oxides and the loss on ignition

Table 3
Mix proportioning (kg/m^3) of concretes.

Material	Mixture		
	C-REF	C-SCBA	C-EGA600
Portland cement	387.4	309.9	309.9
SCBA	–	57.1	–
EGA600	–	–	60.1
Fine aggregate	779.2	779.2	779.2
Coarse aggregate	893.7	893.7	893.7
Water	229.3	229.3	229.3
Superplasticizer	0.3	0.3	0.3

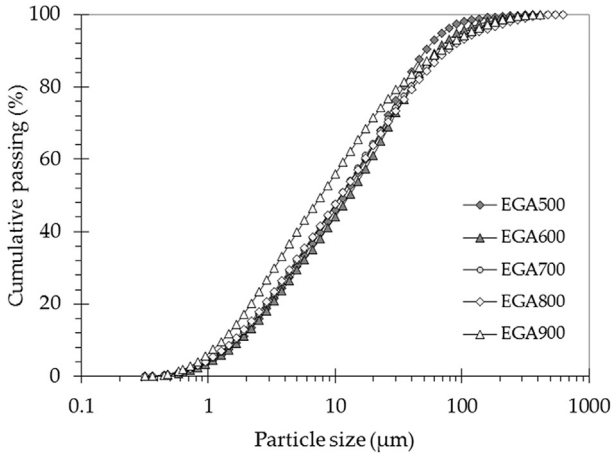

Fig. 2. Cumulative particle size curves for EGAs produced on different calcining temperatures.

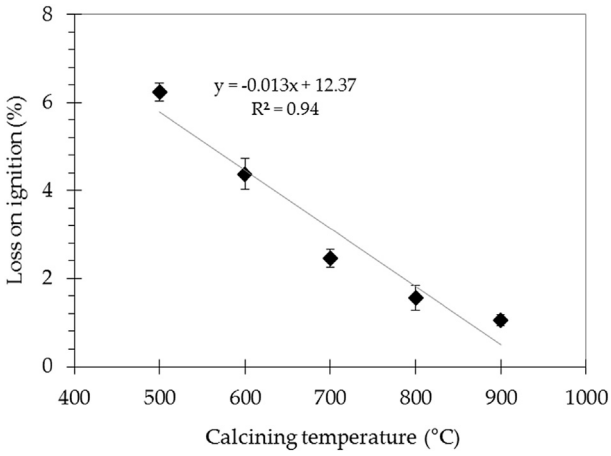

Fig. 3. Linear relationship between loss on ignition values and calcining temperature used to produce each EGA.

Table 4
Percentage of oxides and loss on ignition of EGA samples produced on different calcining temperatures.

Compound	EGA500	EGA600	EGA700	EGA800	EGA900
SiO_2	51.1	52.2	55.1	56.4	55.1
Al_2O_3	22.4	22.7	23.2	22.8	22.1
Fe_2O_3	7.8	7.6	7.6	7.0	9.2
K_2O	4.8	5.4	4.5	4.9	5.0
SO_3	2.2	2.0	2.1	2.2	1.8
TiO_2	1.1	1.0	1.0	0.8	1.2
MnO	0.2	0.2	0.2	0.2	0.2
CaO	2.5	2.8	2.2	2.4	2.5
P_2O_5	1.6	1.7	1.6	1.8	1.8
Loss on ignition	6.3	4.4	2.7	1.6	1.1

values of the different EGA samples. Silica was the main constituent detected in all ashes, with contents above 50% in mass. Due to the elimination of organic compounds at higher temperatures, which was shown in Fig. 1, the increase in calcining temperature resulted in slight increases in silica content. Obviously, this behavior was expected and similar silica contents were observed in studies with SCBA [14,21] and switchgrass [7]. Considerable percentages of Al_2O_3, Fe_2O_3 and K_2O were also observed. It should be noted that the levels of major constituents SiO_2, Al_2O_3 and Fe_2O_3 were over 80% in all EGAs. Another interesting point it was that the presence of iron oxide gave EGA samples a light-pink color. It should be noted that the content of K_2O may be a limitation to the amount of EGA that may be used in replacement of cement since this oxide may negatively affect the durability of concrete. As an example, a maximum content of alkalis (equivalent Na_2O) of 0.6% is recommended [22] for use in concretes containing alkali-reactive aggregates. Neville [23] highlighted that greater the amount of alkalis in the binder, the lower the gain in concrete strength. On the other hand, K_2O could speed up the pozzolanic reactions [24] of EGA due to the increase in pH of pore solution. The presence of K_2O in EGA samples was also detected by Cordeiro and Sales [8] and Nakanishi et al. [9]. In both studies, K_2O levels were prevented from rising by pretreatment of elephant grass prior to calcining.

From the very high contents of volatile matter and fixed carbon of elephant grass (Table 2), the loss on ignition values of all ashes were decreased considerably. This confirmed the adopted calcining procedures, which allowed efficient reductions in loss on ignition (ranging from 1.1 to 6.1%). Similar reduction was previously reported for SCBA production [14]. As can be seen in Fig. 3, a good linear fit ($R^2 = 0.94$) represented the inversely proportional relationship between the results of loss on ignition and calcining temperature. The same behavior was previously described for RHA [25] and SCBA [14]. Except for EGA500, all samples were under 6% limitation, as specified by ASTM C 618-15 [26] and Brazilian standard ABNT NBR 12653 [27].

The X-ray diffractograms (XRD) of EGA samples (Fig. 4) indicated the presence of quartz and andesine as crystalline phases. The occurrence of crystalline phases was probably due to the contamination of elephant grass by soil. This type of contamination was also found in previous studies with RHA [28,29] and SCBA [2,30–32]. Recently, Nakanishi et al. [9] observed EGA samples rich in quartz. There were no expressive changes in XRD patterns due to the increase in calcining temperature, especially with regard to crystallization or decomposition of crystalline phases. On the other hand, the comparison of the diffractograms of EGA samples showed clearly that the intensity of the main quartz peak increased when calcining temperature increased.

Examination of the quantitative phase analysis using the Rietveld method for the EGA600 indicated 14.6% quartz and 75%

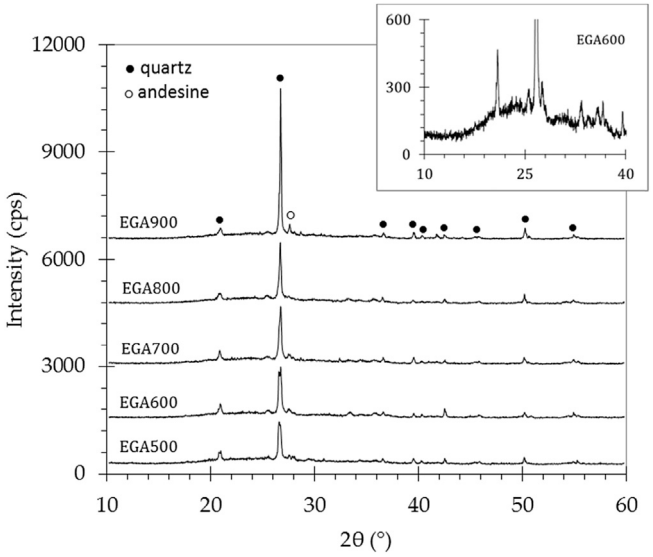

Fig. 4. X-ray diffraction patterns of EGA samples produced on different calcining temperatures. In detail, diffractogram of EGA600 with 2θ between 10° and 40°.

Table 5
Values of density, B.E.T. specific surface area, and pozzolanic activity index for EGA samples.

Ash	Density (kg/m^3)	B.E.T. specific surface area (m^2/kg)	Pozzolanic activity index (%)
EGA500	2461	45.6	93
EGA600	2460	42.1	108
EGA700	2482	40.3	98
EGA800	2497	36.4	94
EGA900	2553	20.3	95

amorphous phase in the sample. In fact, the wide scattering band (amorphous hump) was mainly associated with silica and alumina (2θ ranging between 16° and 30°), and it was evident in EGA600, as shown in the detail of Fig. 4. This was in strong agreement with similar materials like, for instance, RHA [33,34] and SCBA [14]. Cordeiro and Sales [8] described a defined amorphous hump for EGA produced after a preliminary acid leaching treatment of elephant grass.

The density values of the ashes are shown in Table 5. A significant increase in density was observed due to the gradual carbon elimination. As expected, the highest density value was observed for EGA900. Table 5 also shows that the B.E.T. specific surface area of the EGA samples gradually decreased with increasing in calcining temperature. This reduction could be attributed to the removal of carbon in the ash since carbon had a very high specific surface area [35]. Similar values of B.E.T. specific surface area were observed for SCBA samples [1,8,36].

All ashes exhibited a 28-day pozzolanic activity index greater than 75% (Table 5), which is the minimum value as established by ABNT NBR 12653 [27]. Based on this test, all calcining conditions are suitable for the production of pozzolanic EGA. However, the results indicated that the EGA600 had pozzolanic activity index greater than 100%, suggesting that 35% cement replacement by this ash provided a significant increase in compressive strength of the mortar in relation to the other EGA-based mortars. Cordeiro and Sales [8] reported that the pozzolanic reactions of EGA formed CSH and aluminate phases, based on studies in EGA-CH systems. To allow for direct comparisons, the particle size distributions of different ashes were equalized since the pozzolanic activity index, in general terms, consider both physical and chemical effects of pozzolan on the mortar compressive strength. Therefore, in this study, it was assumed a similar physical effect for ashes, and the differences in pozzolanic index values were explained due the variability in chemical reactivity among the EGA samples. Thus, the results suggested that the highest pozzolanic activity values occurred in EGA produced in the range between 600 and 700 °C.

Although there were little differences among the pozzolanic activity values for distinct calcining temperatures, the results of the pozzolanic index considered with other characteristics studied (especially B.E.T. specific surface area and loss on ignition) indicated that EGA600 was the most suitable pozzolan, among the five samples evaluated in this work. The high amorphous content indicated in the quantitative X-ray diffraction analysis for the EGA600 sample confirmed the pozzolanic character of the ash. Thus, the pozzolanic activity of EGA was associated with over 70% amorphous compounds (silica and alumina), ultrafine particle size, and high specific surface area.

The EGA600 had particles of varied morphology with fibrous and cellular particles, as shown in the scanning electron micrographs (Fig. 5). The same spongy morphology (irregular and porous) was also observed for EGA samples in other calcining temperatures. Fig. 5a shows a fibrous particle EGA with the typical cellular structure of agricultural ashes. In fact, the high specific surface area of EGA (Table 5) was associated with the porous structure of the particles (Fig. 5b). The observation of ground EGA

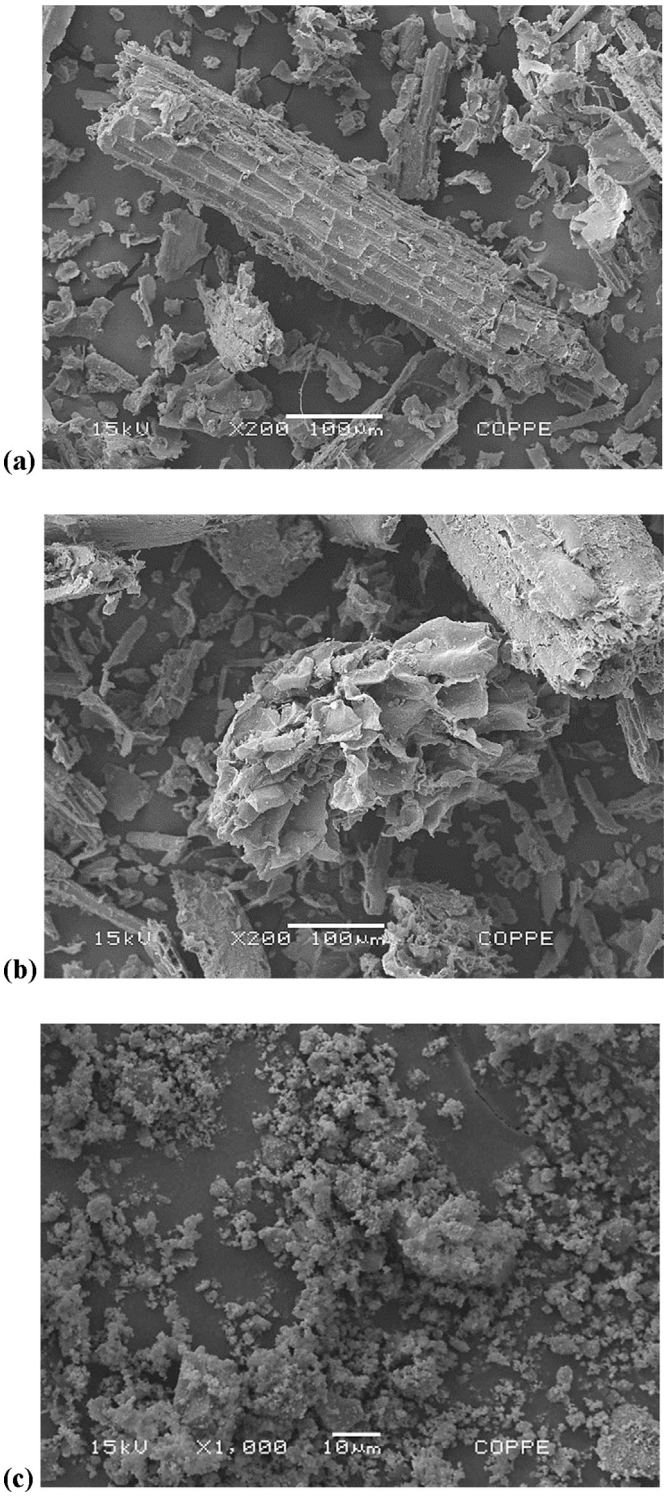

Fig. 5. Scanning electron micrographs of EGA600 before – (a) and (b) – and after (c) ultrafine grinding.

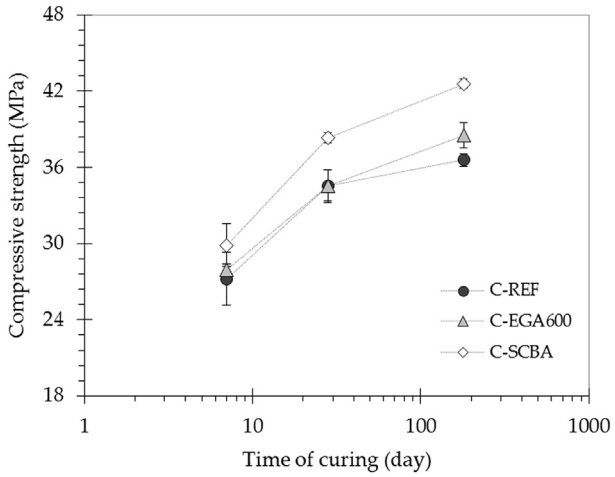

Fig. 6. Compressive strength results of concretes after 7, 28, and 180 days of curing (time in logarithmic scale).

(Fig. 5c) confirmed the particle size distribution results (Fig. 2), which showed that most of the particles were less than 10 μm in size. In this case, EGA coarse cellular-based particles seems to have been totally broken down by the ultrafine grinding.

The behavior of compressive strength of the concretes studied within a 180-day period into curing is illustrated in Fig. 6. After 7 days, no significant difference was observed between the reference and EGA600 mixes, although the cementitious content was lower in C-EGA600 than in C-REF. At this age, the concrete containing SCBA exhibited a strength 10% higher than C-REF. On day 28, the same behavior was observed, with EGA600 compensating for the lower cementitious consumption (and consequently the higher w/cm ratio), and SCBA promoting a significant increase in compressive strength. In the evaluation after 180 days of curing, C-SCBA also showed highest compressive strength among the evaluated concretes, with about 43 MPa (16% higher than the C-REF). At this age, all concretes significantly differed in compressive strength, and C-EGA600 presented an intermediate behavior between C-REF and C-SCBA. The difference between the C-EGA600 and C-REF pointed to the pozzolanic activity of EGA600. On the other hand, the comparison between C-EGA600 and C-SCBA showed that the compressive strength of the concrete containing the pozzolanic SCBA was about 10% greater than that of concrete prepared with EGA600. The highest compressive strengths of SCBA was due to its higher reactivity (associated with its higher silica content).

5. Conclusions

The effect of the calcining temperature of elephant grass on the pozzolanic properties of EGA was investigated in this study. Based on the results, the following conclusions were drawn:

- The two-step calcining procedures afforded the production of pozzolanic EGA at the temperature range from 500 to 900 °C. Except for the ash produced at 500 °C, all other ashes showed loss on ignition values under 5%. The increase in the final calcining temperature resulted in higher density and smaller B.E.T. specific surface area.
- Based on the analytical characterization and pozzolanic activity values, it was concluded that the most suitable EGA was produced at 600 °C under laboratory conditions. Nevertheless, the pozzolanic activity index was not sufficiently changed by calcining temperature.
- Concerning the 35 MPa concrete application, as 20% cement volume was replaced by EGA produced at 600 °C, compressive strength remained essentially constant at early ages, increasing on day 180 of curing in relation to the reference concrete. This occurred with a higher water/cementitious materials ratio, in comparison with the reference, thus indicating the positive effect of the EGA on the compressive strength.
- Finally, the effect of EGA on the compressive strength of concrete was comparable to a known pozzolanic SCBA. It is noteworthy that additional studies need to be carried out with other EGA contents and concrete durability tests.

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