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

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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].

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

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.

$$M = \frac{m - m_{105}}{m} \times 100 \tag{1}$$

$$VM = \frac{m_{105} - m_V}{m} \times 100 \tag{2}$$

$$A = \frac{m_0}{m} \times 100 \tag{3}$$

$$FC = 100 - (M + VM + A) \tag{4}$$

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 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