Research Article

A study of the parameters that determine the reactivity of sugarcane bagasse ashes (SCBA) for use as a binder in construction

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Abstract

This paper intends to contribute to the use of waste, as binder replacement, to lower the negative environmental impact of construction. Sugarcane bagasse ash (SCBA) is an agricultural waste which has been reported as pozzolanic. However, the literature on the pozzolanic activity of SCBA is inconclusive. This paper attempts to establish the properties that determine reactivity the most, in an effort to standardise the material, to allow a wider intake into mainstream construction. The fineness, specific surface area, microstructure, composition and amorphousness of ashes from four producers in Brazil are studied, and the microstructure and kinetics of reactions in ash/hydrated-lime pastes investigated with SEM/EDS. The results evidence that, despite their high crystallinity and carbon content, the ashes are pozzolanic. Reactivity was proven with physical (activity index) and chemical tests (conductivity), and hydrates were evident at 7 days. Reactivity increased with rising specific surface area, amorphousness and fineness, but the specific surface area of the particles impacts reactivity the most. This was concluded form the following findings: Fineness is not vital for reactivity, as some coarse ashes produce similar strengths and combine comparable amounts of lime than much finer ashes. The presence of a significant amorphous fraction is not vital for reactivity, as the ashes are active despite consisting mostly of crystalline phases (quartz, corundum and hematite). High carbon contents do not eradicate reactivity, as the ashes with the greatest carbon are the most reactive, achieving the highest strengths and combining the most lime. Finally, all the SCB ashes present a marginal content of anions and alkalis capable of forming detrimental gels and salts, a desirable quality for any pozzolanic material.

Keywords Sugarcane bagasse ash · Reactivity · Specific surface area · Amorphousness · Fineness · Carbon content

1 Introduction

Today, traditional binders are often partially replaced by pozzolans and supplementary cementitious materials (SCMs), some of them waste, to produce materials of lower embodied energy and lower CO_2 emissions and reduce the negative environmental impact of construction. This also carries economic benefits as it lowers the cost of raw materials, and the amount of landfill waste and quarrying, hence contributing to the preservation of natural resources [1, 2]. Most of the 27 members of the family of cements in EN 197-1 [3] include either SCMs or pozzolans; CEM I is the only cement consisting nearly entirely of clinker (calcium silicates). The use of SCMs and pozzolans is escalating. In 2011, the European cement industry used 47.8 million tons of alternative raw materials in cement production [4]. In 2006, the amount of CEM I in the CEM-BUREAU countries was 28% of the total cement production, while blended cements corresponded to 72% [5]. It is estimated that the production of blended cements has a global potential for reducing cement-making CO_2 emission of at least 5% and up to 20% [6].

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Sugarcane bagasse ash (SCBA) is a by-product of the sugar/ethanol agroindustry. Brazil leads the world production of sugarcane, harvesting around 633.26 million tons per year [7]. The ashes used in this research originate from four sugarcane industries in São Paulo which in 2019 processed c. 55% of the total sugarcane produced in Brazil [7]. After milling the sugarcane to extract the juice, the bagasse is transported to a boiler where it is burned to produce steam and electricity. The boiling temperature can vary remarkably, from 700 °C reaching up to 1000 °C [8–10]. The burning of the bagasse leaves approximately 0.3% of ash residue [11]. The ash can be rich in reactive silica and has pozzolanic potential.

Pozzolanic reaction has been investigated for decades and is mainly governed by the amorphous silica and alumina contents and the specific surface of the pozzolan. Initially, a high specific surface enhances pozzolanic reaction but, at later ages, it is essentially controlled by the active silica and alumina content [12]. It is generally accepted that the silica and alumina must be in amorphous form to be active; hence amorphous pozzolans are considered active, while pozzolans with a significant crystalline fraction and low glass content are assumed to have weak activity. Walker and Pavia [13} state that amorphousness determines pozzolan reactivity to a greater extent than any other property (testing ceramics, fly ash, GGBS, kaolin, microsilica and others). However, despite the general assumption that the crystalline fraction is non-reactive, some crystalline minerals can bind lime [12, 14–16]; hence, the amount of lime bound by a pozzolan's crystalline fraction can be underestimated.

Most authors agree that SCBAs are pozzolanic [17]. Torres et al. [18] found that the pozzolanic activity index of a SCBA with significant amorphous content exceeded the minimum ASTM C618 requirement. However, De Soares et al. [19] claim that SCBA has low or no reactivity, and that it should be used as an inert replacement. This was already reported, in 1998, by Martirena-Hernandez et al. [20], who remark that SCBA is not reactive, mainly due to the presence of unburned material and carbon. However, they detected CSH at 7 days in lime/SCBA pastes.

Residual carbon in pozzolans absorbs moisture and can lower reactivity and spoil the rheology of the materials, increasing the water demand for a given consistency. SCBA typically contains carbon residues from the original sugar bagasse, and some authors have attempted to rid of this carbon by calcination to enhance pozzolanic activity [21–23]. In contrast, other authors claim that a high carbon content does not adversely affect reactivity. Chusilp et al. [24] demonstrated that even though, at early ages, carbon content (> 10%) lowers early strength, at later ages (28 days), 20% carbon content provides compressive strengths similar to the control mix.

The effect of the alkali content on the reactivity of the ashes has not yet been investigated. Alkalis can speed up the dissolution of the amorphous silica favouring early pozzolanic reaction; however, when present in the pore solution at later ages, they adversely affect durability due to alkali-silica reaction. Shi and Day [25] examined the effect of activators (Na_2SO_4 and $CaCl_2$) on pozzolanic reaction kinetics, concluding that the addition of 4% Na_2SO_4 increased both the early and later strengths while 4% $CaCl_2 \cdot 2H_2O$ lowered early strength but increased late strength.

Research results on the pozzolanic activity of SCBA agree with the general parameters that govern pozzolanic activity presented above. Cordeiro et al. [26] evidenced that the finest SCBA provided the highest pozzolanic activity and density, producing a higher compressive strength, while Cordeiro and Kurtis [11] evidenced that increasing the SCBA's specific surface area enhanced pozzolanic activity and strength, and that early hydration increases with decreasing SCBA particle size. They found a direct linear relationship between strength and specific surface area, with correlation coefficients higher than 94%. They also claim that coarser ashes (D50 c.30 μm) behave as inert mineral additions.

One of the main tests to determine pozzolanic activity is to measure the strength of pozzolan composites compared to a reference material (mechanical index). The pozzolanic reaction is slow; hence, pozzolans may not increase early strength but can significantly increase ultimate strength [12]. This has been confirmed for SCBAs: Arenas-Piedrahita et al. [27] found that 10–20% SCBA decreased compressive strength at early ages but, after 90 days, the strength was similar or surpassed the control sample. Furthermore, strength development in pozzolanic materials not only relates to the evolution of the pozzolanic reaction (amount of combined lime and hydrates formed), but also to the physical filler effect of the pozzolans that increases density and strength and is enhanced by the presence of finer particles. The filler effect of the non-reactive part of the SCBAs is also noted in the literature [11, 19].

Previous authors have investigated the reactivity of SCBA in Portland cement (PC) systems. Some authors report that 20–30% of cement can be replaced with SCBA without prejudice to strength [18, 28, 29] or even a strength increase [24, 30]. Some authors set the optimum replacement of PC with SCBA in concrete at 20% [31] others at 15% [32] and others even lower, at 10% [33, 34] or 5% [35, 36].

As it can be seen from the literature, the results are inconsistent, with lack of agreement on the SCBA reactivity and ample variability on the optimal substituion of binder with SCBA. The disparity of the results suggests that the nature of the ashes significantly determines the

properties of the composites. SCBA is a natural material and, as such, it is subject to variability. Sales and Lima [37] note that some SCBAs have poor reactivity due to the lack of control on the production parameters, and this restricts use. In addition, the presence of contaminants and varying burning conditions result in SCBAs with different chemical composition and reactivity. Hence, it is fundamental that individual sources of SCBA are evaluated. This paper investigates the properties and reactivity of four ashes. It intends to contribute to the existing knowledge of SCBA ashes to enhance their use in construction. It also attempts to determine which of the SCBA properties determine reactivity the most, in an effort to standardise the material and allow a wide intake into mainstream construction.

2 Materials and methods

A hydrated lime (CL90-S) complying with EN 459-1 [38] and a siliceous sand of grading and composition complying with the European standards were used. The ashes were sourced from four Brazilian sugarcane industries. The raw SCBAs were oven-dried at 105 °C for 24 h. Grinding was then performed to enhance fineness using a TEMA T100 Disc Mill (1000 RpM; max sample 150 g). As relatively small quantities are required for analysis and testing, homogenisation was ensured by subsampling, using a Rotary Sample Divider laborette 27, to ensure representativity.

2.1 Grading, specific surface area and microstructure

The particle size distribution was measured by laser diffraction using a Mastersizer 2000, composed of three units, the Mastersizer 2000 unit; the Hydro 2000G wet dispersion unit; and the Autosampler 2000. Laser diffraction measures the angular distribution and intensity of the light by particles in suspension and uses the Mie theory of diffraction to process the results. Lime was included as a control material to which compares the grading of the ashes. The solubility of lime in water is ignored in the calculations, as it is very low at room temperature, and the measurement was carried out instantaneously on dispersion. The specific surface area of the particles was determined with a Quantachrome Nova 4200e and the BET method, a model isotherm based on adsorption of nitrogen gas on a surface. The microstructure of the ashes was analyzed with a scanning electron microscope (SEM), Tescan Mira XMU, revealing the surface topography, morphology and pore system with a backscattered electron (BE) signal detector.

2.2 Chemical composition, mineral composition and amorphousness

The chemical analysis as percentage by oxides was performed by Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP-AES) analysis. Oxide concentration was calculated from the determined elemental concentration. The carbon content was determined by thermal decomposition using the loss on ignition (LOI) method. The carbon was calculated from the difference in weight at a set temperature. The mineralogical composition and amorphous character of the ashes were analysed by X-Ray Diffraction (XRD), using a Phillips PW1720 XRD with a PW1050/80 goniometer and a PW3313/20 Cu k-alpha anode tube at 40 kV and 20 mA. All measurements were taken from 3° to 60° (2θ) at a step size of 0.02°/s.

2.3 Reactivity by electrical conductivity

The conductivity variation of lime-ash suspensions over time was monitored to assess the ability of the ashes to combine lime—$Ca(OH)_2$–and thus their reactivity according to de Luxan et al. [39]. A sample (0.8 g) of ash was added to a saturated hydrated-lime solution (0.2 g of lime in 100 mL of water). The solutions were kept in sealed flasks to avoid evaporation and carbonation. A thermostatic bath was used to maintain the flasks at constant temperature (20 °C). The loss in conductivity was measured at intervals over 168 h using a WTW Conductivity Meter ProfiLine Cond 197i.

The conductivity loss (%) was calculated according to the following equation:

$$C(\%) = \frac{C_i - C_t}{C_i} \times 100$$

where C_i is the initial electrical conductivity of a $Ca(OH)_2$ suspension before adding the ash; C_t is the electrical conductivity of the ash and lime suspension measured at time intervals.

2.4 Reactivity by strength development

The pozzolanic activity of the ashes was measured by monitoring strength development according to EN 450-1 [40]. The mechanical index was calculated as the ratio of the compressive strength of the ash specimens to a standard lime mix. 40×40×160 mm prisms were produced with a ratio by mass of 1:1:3:2 (lime: SCBA:sand: water) for SCBA 1 and SCBA 2; and a slightly lower water content (1:1:3:1.5) for SCBA 3 and SCBA 4. The water content was lower in order to meet the lower water demand of the ash

which was determined according to the research results and equation in Walker and Pavia [41]. The 1:1 (lime:ash) content was chosen to be able to compare with former literature [13, 17, 41]. The specimens were demolded after 3 days and cured for 90 days at 20 °C and 60% relative humidity. The flexural strength was assessed following EN 196-1 [42].

The activity index (mechanical index) is adopted from EN450-1-fly ash (FA) for concrete. In this standard, the activity index is the ratio (in percentage) of the compressive strength of standard mortar bars prepared with 75% test cement and 25% fly ash (by mass) to the compressive strength of the standard bars prepared with 100% cement. The FA standard limit value for the activity index (single results) is 70% at 28 days. To investigate the SCBAs, we have adopted the same method. However, we cannot compare the results nor apply the standard limits as we are not using cement but lime, and furthermore, we are using waste of lower activity.

2.5 Microstructure and kinetics of reaction of sugar bagasse ash/hydrated-lime pastes

Pastes were fabricated with each of the ashes and hydrated lime (CL90-S) at 1:1 ratio by weight. The pastes were studied with a SEM/EDS system. The study focussed on the quality and quantity of pozzolanic hydrates at 7 and 28 days, and the microstructure of the pastes. In the pastes, the reaction between hydrous silica and lime should produce C–S–H. As this reaction has a varying stoichiometry, the calcium/silicate (C/S) ratios of the C–S–H product were measured and compared with results in the literature. The SEM imaging results are qualitative; therefore, the quantification of the mineral phases was carried out by EDS and XRD. The XRD results were quantified using the refractive indices of the minerals, the relative intensity (%) and the area of the peaks in the XRD trace. Also, the chemical composition of mineral phases in the lime paste was determined by SEM/EDS according to their relative proportions and C/S ratio.

3 Results

3.1 Grading, specific surface area and microstructure

According to the particle size distribution (Fig. 1 and Table 1), SCBA 1 and SCBA 2 are the finest ashes. They present similar curves, with 90% of the particle volume under 29.7 μm and 33.03 μm respectively, well under the particle size of the hydrated lime with 90% of particles under 57.98 μm. The SCBA 3 and SCBA 4 ashes are much coarser
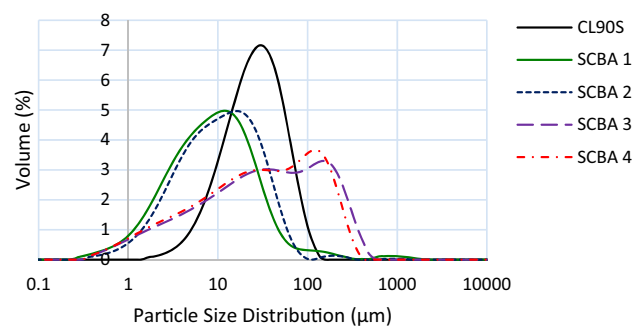

Fig. 1 Grading of the ashes by laser diffraction

and exhibit a broader range of particle sizes. As it can be seen from the laser grading, in all the ashes, over 75% of the particle volume falls into the hydrated-lime particle range (1.44–138.04 μm)—above 92% for SCBA 1 and SCBA 2. According to the D50 parameter (median diameter or medium value of particle size distribution), SCBA 1 is the finest (D50 = 8.14 μm–50% of the particles in the sample are larger than 8.14 μm, and 50% smaller) and SCBA 3 the coarsest (D50 = 31.26 μm).

In cement technology, it is considered that particles larger than 45 μm are difficult to hydrate and those larger than 75 μm seldom hydrate entirely [43]. The cumulative volume shows that less than only 7% of particles are greater than 45 μm in the SCBAs 1 and 2 ashes, as opposed to a much higher percentage (42–45%) in the SCBA 3 and 4.

The specific surface area of the ashes (Table 1) agrees with the particle size distribution, since the smaller the particle size the greater the specific surface area. However, the great disparity in the results (the SCBA 1 and SCBA 2 ashes present much greater specific surface area than SCBA 3 and SCBA 4) suggests a porous character for the SCBA 1 and SCBA 2 ashes. It is likely that the high carbon content of the SCBA1 and 2 ashes has contributed to their high specific surface and has also enhanced their porosity. The SCBAs 1 and 2 ashes show specific surface areas greater than typical pozzolanic materials such as rice husk ash (RHA) and microsilica (13.7 m^2/g and 26 m^2/g) [41, 44]. The D values rate SCBA 1 as the finest; however, the SCBA 2 has a much greater surface area indicating that SCBA2 is the most porous among all the ashes.

The SEM analyses showed that the ashes contain an heterogeneous mixture of irregular, spherical, prismatic and flaky particles (Fig. 2) which agrees with other authors [17, 23, 37]. In the SCBA 1 and SCBA 2 ashes, residual organic matter is evident as fibres and tabular particles, and cavities are filled with small particles which increases surface area (Fig. 2a–d). The SCBA 3 and SCBA 4 ashes contain more abundant coarse quartz than the other ashes (Fig. 2e,

Table 1 Analyses of laser grading results, percentage of particles that determine reactivity and specific surface area of the ashes

	D10 (µm)	D50 (µm)	D90 (µm)	% > 45 µm	% > 75 µm	Specific surface area (m²/g)
SCBA 1	1.82	8.14	29.68	6.19	3.35	25.639
SCBA 2	2.29	9.88	33.03	6.19	1.18	78.337
SCBA 3	2.54	31.26	190.50	44.72	32.96	0.514
SCBA 4	2.39	28.13	154.65	42.50	30.41	2.199
CL90-S	8.15	24.13	57.98	24.69	5.47	16.09

f), and the quartz is often covered with agglomerates of fine spherical, flaky and needle-shaped particles. The SCBA 1 and SCBA 2 particles are finer and more porous, agreeing with the laser grading and specific surface area results.

3.2 Chemical composition, mineral composition and amorphousness

As it can be seen from Table 2, the chemical composition of the SCBA ashes compares well with others previously studied [4–6, 27]. They include abundant silica (SiO_2) agreeing with previous authors. According to Cordeiro and Kurtis [6], silica is the main component of SCBA and arises from the original hydrated silica ($SiO_2 \cdot nH_2O$) skeleton of the plant and soil contamination.

There are large discrepancies in the amount of SiO_2 in the ashes from the four different providers (Table 2): the SCBA 3 and SCBA 4 ashes are the most siliceous ($SiO_2 > 90\%$ by mass) while SCBA 1 and SCBA 2 ashes only contain 39 and 59%, respectively. As it can be seen in Table 3, the silica in SCBA 3 and 4 is crystalline quartz. Therefore, it is attributed to contamination, by sand attached to the sugarcane either during harvesting or later in the soil, as reported by previous authors [6, 12]. The low carbon content of SCBA 3 and 4 agrees with this. Hence, the SCBA 1 and 2 providers probably collected the ash from the incineration chamber while the SCBA3 and 4 providers collected it from dumping sites.

The active alumina content also contributes to pozzolanic activity forming calcium aluminate hydrates (C–A–S–H and C–A–H), also, aluminium can replace silicon in the C–S–H structure. The aluminium content in the ashes varies considerably, with SCBA1 showing the highest content at 13% and SCBA3 and 4 the lowest at 0.74 and 1.69, respectively.

The XRD results were quantified using the refractive indices of the minerals, the relative intensity (%) and the area of the peaks in the XRD trace (Table 3). The results (Fig. 3 and Table 3) indicate that the silica is mostly crystalline quartz, and the aluminium is mainly present as corundum (Al_2O_3). Corundum is stable at high temperatures and, in small quantities, it doesn't significantly influence

long-term pozzolanic activity [45]. It seems from the results that no metallic aluminium (which can cause expansion in an alkaline solution during setting) is present in the ashes [29]. Calcite peaks were only identified in SCBA 1 (Fig. 3a) agreeing with the chemical composition results (highest CaO of all ashes at 3.19%). No calcite appears in the SCBA 2 trace (Fig. 3b) despite its calcium content (CaO = 2.42%), probably due to being under the XRD detection limit (5%). The XRD patterns of ashes SCBA 3 and 4 are nearly identical and highly crystalline, showing only quartz and minor hematite (Figs. 3c and 3d). SCBA1 has the highest iron content (11.40%) (Table 2) which is minor in the other ashes at c.2%. The mineralogical analyses (Table 3) indicate that the iron is present as crystalline hematite (Fe_2O_3). Due to the low solubility and slow dissolution kinetics of hematite, it is likely that this iron does not contribute or contributes faintly to the pozzolanic reaction.

The SCBA 1 and SCBA 2 ashes present high LOI, hence a high carbon content, whereas the SCBA 3 and SCBA 4 values are low (≤ 0.41). The high LOI agrees with former authors [46, 47] but comparable low values are also found in the literature [17, 26]. The high LOI indicates temperatures in the low range and uncontrolled combustion.

The absence of high-temperature phases such as cristobalite and mullite, associated with temperatures above 800 °C, suggests production temperatures in the medium-to-low range agreeing with the high LOI.

The high LOI of ashes SCBA 1 and 2 is over the standard limits prescribed for pozzolans and SCMs such as FA and GGBS (Table 4). However, Chusilp et al. [24] and Ganesan et al. [28] state that a LOI up to 10% in sugar bagasse ashes does not significantly affect the compressive strength of the resultant concrete.

All the ashes comply with the sulphur and chloride limits established by EN and ASTM standards for other pozzolans and SCMs (Table 4). The sulphur and chloride contents are exceedingly low (Table 2), an outstanding quality for any material, as it lowers the risk of expansion by salt attack and delayed ettringite formation. The alkali content is also very low: the highest potassium content is 3.73% in SCBA2, and the sodium content marginal (highest in SCBA2 at 0.16%) (Table 2). Alkalis can speed up the
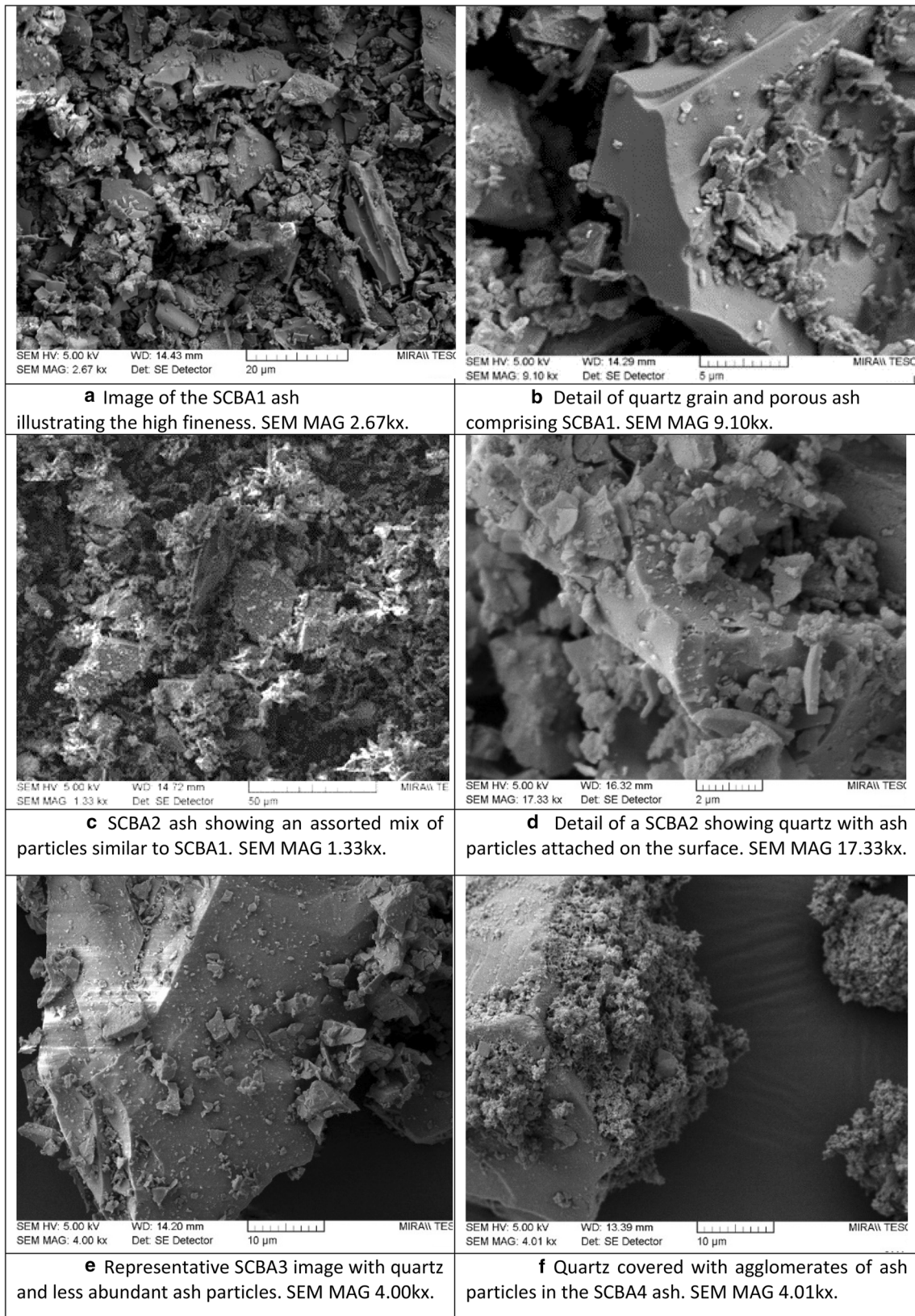
a Image of the SCBA1 ash illustrating the high fineness. SEM MAG 2.67kx.

b Detail of quartz grain and porous ash comprising SCBA1. SEM MAG 9.10kx.

c SCBA2 ash showing an assorted mix of particles similar to SCBA1. SEM MAG 1.33kx.

d Detail of a SCBA2 showing quartz with ash particles attached on the surface. SEM MAG 17.33kx.

e Representative SCBA3 image with quartz and less abundant ash particles. SEM MAG 4.00kx.

f Quartz covered with agglomerates of ash particles in the SCBA4 ash. SEM MAG 4.01kx.

Fig. 2 SEM micrographs showing the microstructure of the SCBA ashes

Table 2 Chemical composition of SCBAs as % oxide by mass

	SCBAs				Sales and Lima [37]			Cordeiro et al. [8]	Cordeiro and Kurtis [11]	Ferreira et al. [46]
	1	2	3	4						
SiO_2	39.00	59.10	96.80	93.40	62.7	93.5	96.2	70.5	80.8	66.80
Al_2O_3	13.65	4.19	0.74	1.69	4.5	1.2	0.2	10.1	5.1	7.70
Fe_2O_3	11.40	2.01	2.06	2.69	8.8	2.6	1.7	7.4	1.6	10.20
CaO	3.19	2.42	0.25	0.36	0.9	0.4	0.1	4.2	3.1	0.94
MgO	1.55	1.65	0.20	0.27	0.6	0.3	<0.1	–	–	0.49
K_2O	1.95	3.73	0.49	0.71	1.8	0.8	0.3	1.8	6.3	1.03
TiO_2	0.90	0.78	0.45	0.97	3.1	0.5	0.2	0.3	0.3	–
P_2O_5	1.16	1.25	0.15	0.16	0.7	0.2	0.1	1.1	0.8	–
SO_3	0.21	0.81	0.02	0.03	0.2	<0.1	0.1	2.3	1.5	–
Na_2O	0.00	0.16	0.00	0.00	0.1	<0.1	–	–	–	<0.10
SrO	0.01	0.02	<0.01	<0.01	–	–	–	–	–	–
BaO	0.02	0.05	<0.01	<0.01	–	–	–	–	–	–
Cl	0.01	0.11	0.01	0.01	–	–	–	–	–	–
LOI	24.10	23.60	0.41	0.31	16.28	0.34	1.04	2.2	0.4	11.47

Table 3 Mineral composition of the sugar bagasse ashes

Material	Main minerals	Minor	Quartz	Hematite	Calcite	Corundum
SCBA 1	Quartz, hematite, calcite	Corundum	c.70%	6%	6–7%	c.5%
SCBA 2	Quartz	Corundum, hematite	>90%	c.5%	–	c.5%
SCBA 3	Quartz	Hematite	>90%	c.5%	–	–
SCBA 4	Quartz	Hematite	>90%	c.5%	–	–

5% is the limit of detection of the technique. ICDD: Quartz (syn) 046-1045; hematite 24–72; corundum 46-1212; calcite 24-27

a XRD trace of SCBA 1.

b SCBA 2

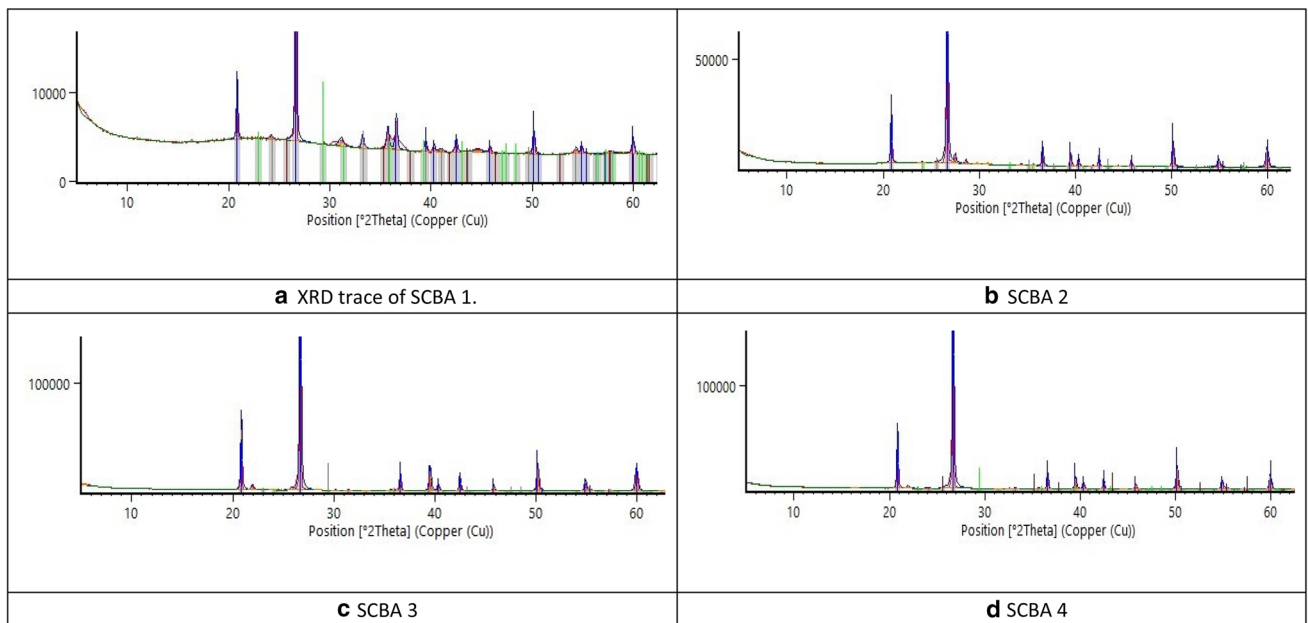**c** SCBA 3

d SCBA 4

Fig. 3 Diffractograms of the ashes

Table 4 Compliance of the sugar bagasse ashes with chemical requirements in standards for pozzolans and SCMs

	% SO$_3$/S^{2-}	MgO (%)	Total alkalis	Cl$^-$	LOI (%)	Σ (Si, Al, Fe) (%)
EN 15167-1 –GGBS requirements for use in concrete, mortar and grout [49]	≤ 2–2.5	≤ 18	–	≤ 0.10	≤ 3	–
ASTM C618—FA requirements for use as a pozzolan [50]	< 5	–	–	–	< 10	≥ 70 class F ≥ 50 class C
EN450-1- FA requirements [40]	≤ 3	≤ 4	≤ 5	≤ 0.10	< 9	≥ 70
SCBA 1	0.21	1.55	1.95	0.01	24.10	64
SCBA 2	0.81	1.65	3.89	0.11	23.60	65
SCBA 3	0.02	0.20	0.49	0.01	0.41	99
SCBA 4	0.03	0.27	0.71	0.01	0.31	98

Table 5 Amorphousness of the SCBAs

Material	Slope base	Slope hump	Amor-phous-ness
SCBA 1	− 37.052	− 63.128	S – I
SCBA 2	− 36.039	− 61.599	S–I
SCBA 3	− 28.228	− 40.974	C
SCBA 4	− 0.002	− 0.002	C

C, crystalline; S, slightly amorphous; I, intermediate

Table 6 Summary of the conductivity variation of the lime-ash solutions

Material	Total conductivity loss (%)	Conductivity drop the first 24 h (%)
SCBA 1	18.37	10.61
SCBA 2	21.02	11.12
SCBA 3	13.47	9.90
SCBA 4	17.65	10.20

dissolution of amorphous silica initially favouring pozzolanic reaction; however, at later ages they adversely affect durability due to alkali-silica reaction forming expansive calcium-alkali-silicate-hydrate gels.

The amorphousness of the ashes was loosely categorised based on the slope of the diffractogram's base line and the background area determined by the lifting of the baseline between 15 and 35 (2Θ) which indicates the presence of amorphous materials. This broad hump cannot be accurately measured; however, the vitreous character of a pozzolanic material is indicated by the size of the hump [48]. According to these results (Table 5), SCBA 1 is the most amorphous (slight to intermediate amorphousness).

3.3 Reactivity by electrical conductivity

It could be argued that there are errors in the conductivity measurements due to two issues: the ashes being soluble in water, and the presence of soluble salts within the ashes. If these were true, these additional cations and anions would affect conductivity inducing errors in the measurement. However, this is not the case in the ashes investigated because the conductivity of ash-water solutions was measured and found negligible (< 1 mS), and the anion and alkali content of the ashes is extremely low (Table 2). In addition, a further error could have been introduced by allowing the process of carbonation fixing the Ca(OH)$_2$

in suspension, rather than the ashes and their pozzolanic reaction. However, the suspensions were kept tightly closed during the test to avoid CO$_2$ inducing carbonation.

The carbon content can impede lime combination by the ash, hence interfering with the results of the conductivity test. However, the results show that the ashes with the highest carbon content (SCBA 1 and 2) combine significant lime showing activity.

In addition, from the differing particle size distributions, it was expected that the finer ashes would combine more lime earlier, leading to more substantial conductivity changes. This is the case, as the finest ashes (SCABA1 and 2) show the greatest conductivity losses.

This method is adopted from de Luxan et al. who tested natural pozzolanic materials including opaline rocks and diatomite earths and volcanic tuffs. These materials are significantly more reactive than our sugar waste ashes. Therefore, we have extended the testing time to provide detail on the early and late reactivity (up to 7 days), and we calculate the conductivity drop in the first 24 h. The total conductivity loss and the loss in the first 24 h are included in Table 6.

Conductivity loss over time is clear in all the lime/ash solutions (Fig. 4), indicating that all the ashes combine lime, hence showing reactivity. SCBA 2 showed a superior conductivity loss suggesting the greatest activity, closely followed by SCBA 1 and 4. The conductivity results agrees with the expected reactivity based on the

Fig. 4 Pozzolanic activity evidenced by the change in the electrical conductivity of ash-lime suspensions over a period of 7 days
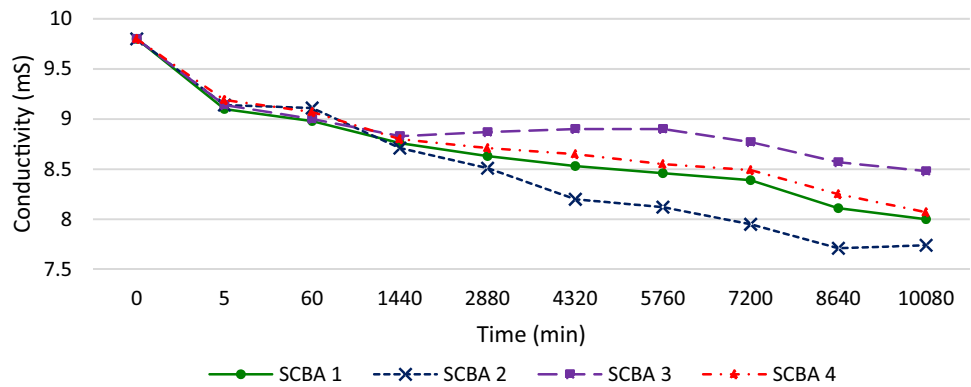
specific surface area, rating the SCBA 2 ash as the most reactive ash followed by SCBA 1, with the SCBA 4 and SCBA 3 ashes being the least reactive (Table 6).

During the first 24 h (up to 1440 min—Fig. 4), the curves show steep slopes whereby all the ashes combine the most lime and the conductivity of the solutions significantly lowers. After 24 h, the lesser steep curves indicate a drop in the reaction rate. SCBA 3 shows a dormant period where the curve is flat tending to inverted. On the contrary, SCBA2 still combines significant lime producing the steepest curve which evidences sustained reactivity. SCBA 1 and 4 also show a sustained, although small, activity.

The conductivity values in Table 6 are in agreement, with previous authors [41] which showed conductivity reduction, in the first 24 h, ranging between 10 and 30% (for 9 pozzolanic materials) and are higher than those found by Tashima et al. [51] for rice husk ash (RHA). Cordeiro and Kurtis [11] claim that coarse ashes (D50 = 30 μm) behave as inert mineral additions, SCBA 3 and 4 are close to this value; however, they combine 9–10% lime in the first 24 h.

3.4 Reactivity by strength development

The SCBA 2 specimens achieved the highest compressive strength, up to 3.62 N/mm² (Table 7), followed by the SCBA 1 specimens at 1.95 N/mm². The SCBA 4 and SCBA 3 specimens reached similar strengths at 1.65 and 1.23 N/mm², respectively. The particle size distribution of the SCBA 1 and 2 ashes, which favours a filler effect, might have contributed to the higher strength results.

The flexural strengths are similar, but the results do not show any specific trend. However, the SCBA 1 and 4 ashes surpassed the flexural strength of the reference (hydrated lime) specimen. According to the strength results, SCBA2 is the most reactive ash followed by SCBA1. Only the SCBA2 ash improved the compressive strength of the hydrated-lime mix. The strength drop is due to the lower hydrated-lime content producing less-carbonated lime binder and resembles the dilution effect reported in cement technology when cement is increasingly replaced with mineral admixtures. The mechanical indices are lower than others previously measured for highly reactive materials such as GGBS (29.5) and microsilica (12.5), but comparable or slightly under those of ceramic dusts (1–3) and some fly ashes (3.4) measured by Walker and Pavia [41]. They also

Table 7 Mechanical properties and mechanical indices for SCBA 1 and SCBA 2 specimens (1:1:3:2) and SCBA 3 and SCBA 4 specimens (1:1:3:1.5) (lime:ash:sand:water)

Material	Compressive strength (MPa)	Flexural strength (MPa)	Mechanical index		Strength variation (%) with respect to the reference	
			Compressive strength	Flexural strength	Compressive strength	Flexural strength
Ref.-hydrated lime only	2.45 (1.12)	0.39 (2.47)	–	–	–	–
SCBA 1	1.95 (4.70)	0.41 (1.80)	0.8	1.0	− 20.66	8.75
SCBA 2	3.62 (2.15)	0.36 (4.30)	1.5	0.9	47.46	− 8.06
SCBA 3	1.35 (3.16)	0.28 (2.66)	0.6	0.7	− 44.83	− 28.05
SCBA 4	1.65 (1.09)	0.46 (5.22)	0.7	1.2	− 32.95	16.83

The italics are the coefficient of variation (%) in brackets

agree with Moropoulou et al. [52] who attained flexural strengths ranging from 0.34 to 0.38 MPa using earth of milos and brick powder.

3.5 Microstructure and kinetics of reaction of sugar bagasse ash/hydrated-lime pastes

The pozzolanic reaction mechanism is slow and has been well documented by previous authors [12]. According to this reaction, when water is added to the ash and lime, the lime -$Ca(OH)_2$—quickly dissociates into Ca^{2+} and OH^- according to the equation below, producing a solution with a high pH.

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2OH^-$$
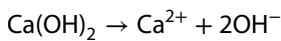
In this alkaline solution, the Ca^{2+} as well as other soluble phases in the pozzolan (alkalis, sulphates and chlorides) dissolve contributing with their ions to the solution. The alkaline medium also causes the pozzolan's silicates to break down (mainly the amorphous phases) and dissolve into the solution according to Shi and Day [25]:

$$\equiv Si - O - Si \equiv +3OH^- \rightarrow [SiO(OH)_3]^-$$
$$\equiv Si - O - Al \equiv +3OH^- \rightarrow [SiO(OH)_3]^- + [Al(OH)_4]^-$$
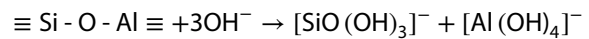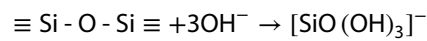
Ca^{2+} ions combine with these dissolved silicate and aluminate anions to form C–S–H and calcium aluminate hydrates. The nature of these hydrates largely depends on the concentration of the ions in solution. Therefore, it was expected that the reaction between the hydrous silica in the ashes and the lime would produce C–S–H. The aluminium content of the ashes is very low except for SCBA1 (Table 2); hence, aluminate hydrates were hardly expected. Furthermore, the mineral analysis evidenced that most of the aluminium in the ashes is present as crystalline corundum (Al_2O_3) which is hardly soluble in any solvent, even in the alkaline solution produced by the hydrolysis of lime.

Despite the crystalline composition of the ashes, including silica as quartz and little reactive aluminium, C–S–H and C–A–S–H were evidenced early (7 days) in the SCBA 1 and SCBA 2 pastes, which also displayed the highest flexural and compressive strengths. Hydrates, showing needle-like morphologies and significant crystallinity were evidenced in the SCBA 1 pastes at 7 days (Fig. 5). These hydrates are similar to products found in lime-RHA
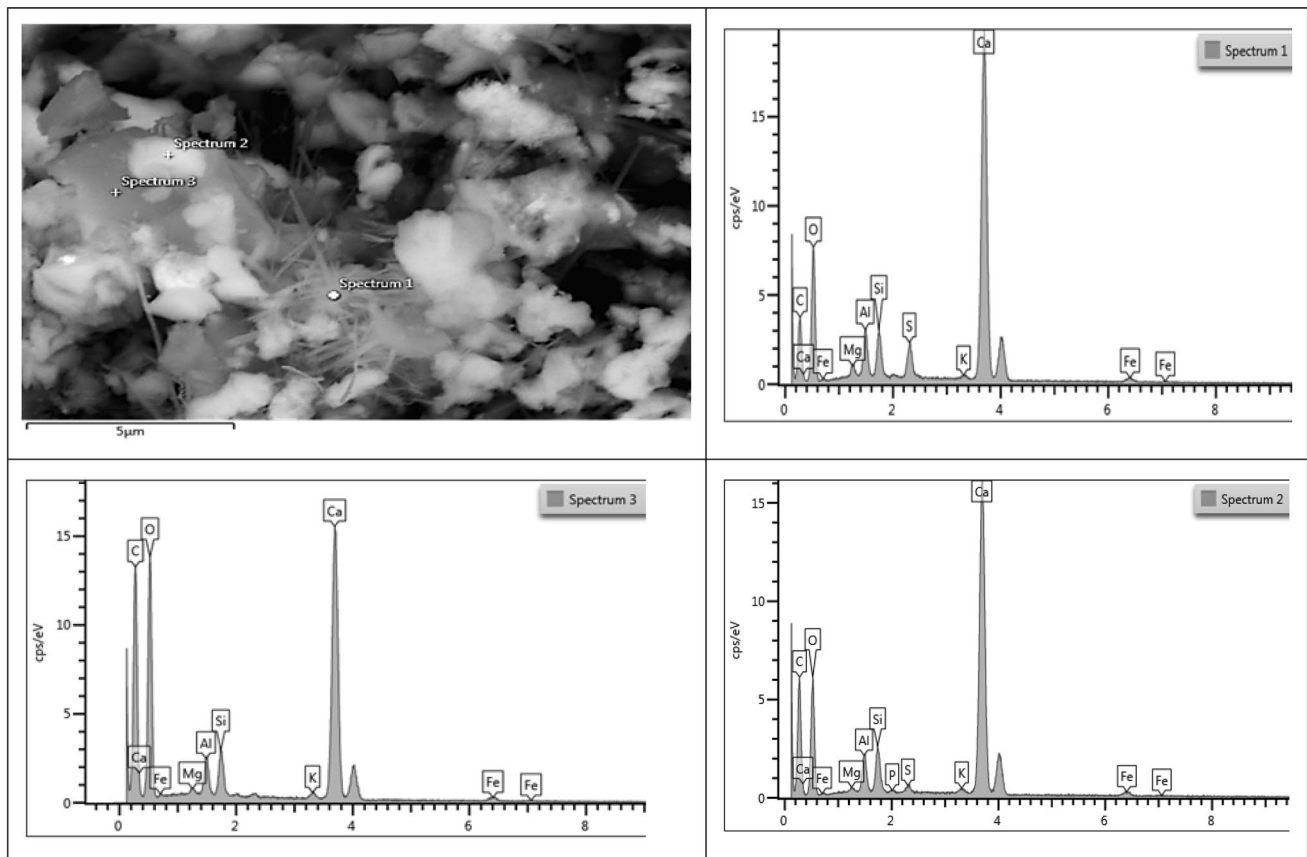

Fig. 5 Lime-SCBA 1 paste at 7 days showing needle-shaped hydrates (EDS spectrum 1), carbonated lime (EDS spectrum 2) and remains of organic matter (EDS spectrum 3)

samples [44]. Alike hydrates have been reported in SCBA pastes by previous authors [20, 21, 53]. At 7 days, the pastes showed the open porous structure typical of lime mortars, with hexagonal portlandite (CH) plates and abundant, irregular carbonated particles (Ca CO$_3$) (Figs. 5, 6). At 28 days, (Fig. 7) denser microstructures were evident comprising abundant carbonated lime and scarce hydrates. At later ages, CH seldom occurs, indicating that the pozzolanic reaction is practically completed, as the consumption of CH over time confirms pozzolanic activity. The qualitative nature of the SEM/EDS analyses did not allow to rate the ashes based on the microscopic study.

As the pozzolanic reaction has a varying stoichiometry, the calcium/silicon (C/S) ratios of the C–S–H hydrates vary, and C/S ratios ranging from 0.6 to 2.0 have been found by previous authors [54, 55]. Pereira et al. [55] identified amorphous matrices in sugarcane straw ash and cement mixes with molar ratios C/S = 0.60, Al/Si = 0.63, Na/Si = 1.34, and K/Si = 0.05. These ratios are similar to those found upon hydration of the lime-ash pastes in this study (Table 8).

4 Discussion

As it can be seen from Table 7, the finest and most amorphous ashes (SCBA 1 and 2), with the greatest surface area (mainly SCBA2), are the most reactive, and, despite their high carbon content, they combined the most lime and reached the greatest strengths. The ash with the greatest surface area combines the most lime, agreeing with former
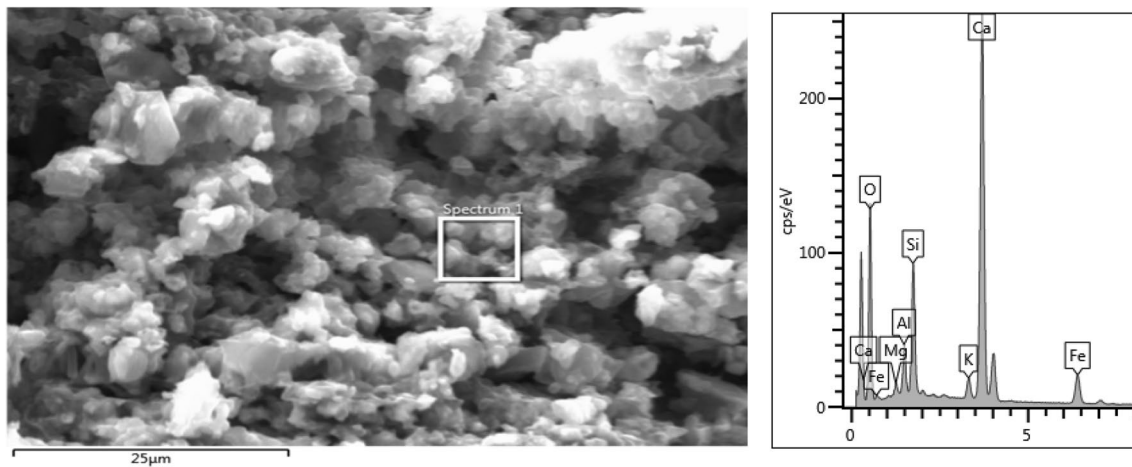

Fig. 6 Lime-SCBA 2 paste showing a porous structure with hexagonal portlandite, carbonated lime and C-A-S-H phases identified by EDS at 7 days
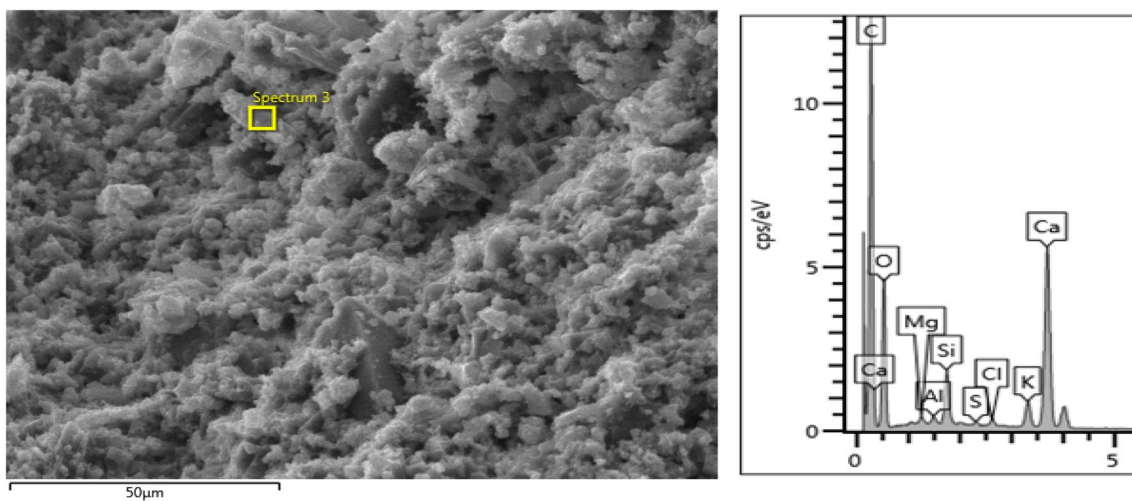

Fig. 7 A representative image of the denser lime-SCBA 2 pastes, at 28 days, showing strong carbonation

Table 8 Chemical composition of mineral phases in the lime paste determined by SEM/EDS

Material	Ca	Si	C/S	Al	Mg	K	Fe	S	Cl	Ref. Figure
SCBA 1–7 d (1)	14.7	11.6	1.3	1.1	0.3	0.1	0.3	1	–	Figure 5
SCBA 1–7 d (2)	12.1	9.1	1.3	0.8	0.1	0.1	0.2	0.2	–	Figure 5
SCBA 1–7 d (3)	11.5	20	0.6	0.9	0.1	0.2	0.3	–	–	Figure 5
SCBA 2–7 d	164.2	91.2	1.8	16	3.9	8.3	29.4	–	–	Figure 6
SCBA 2–28 d	2.5	4.2	0.6	0.1	0.1	0.3	–	–	0.06	Figure 7

Relative proportions and C/S ratio
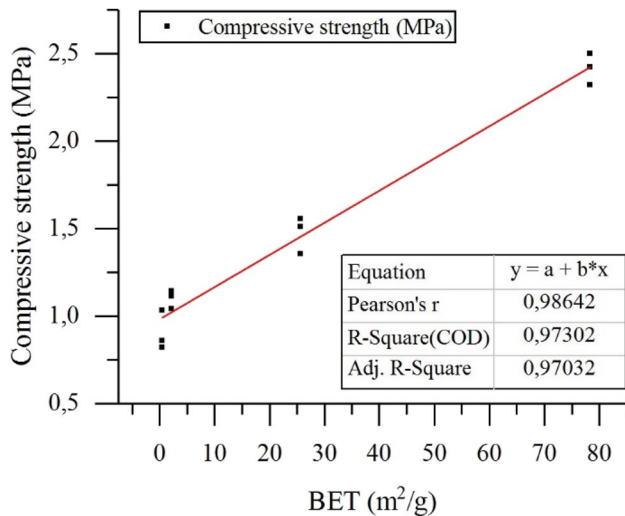
Fig. 8 Relationship between the specific surface area and the compressive strength of the SCBAs
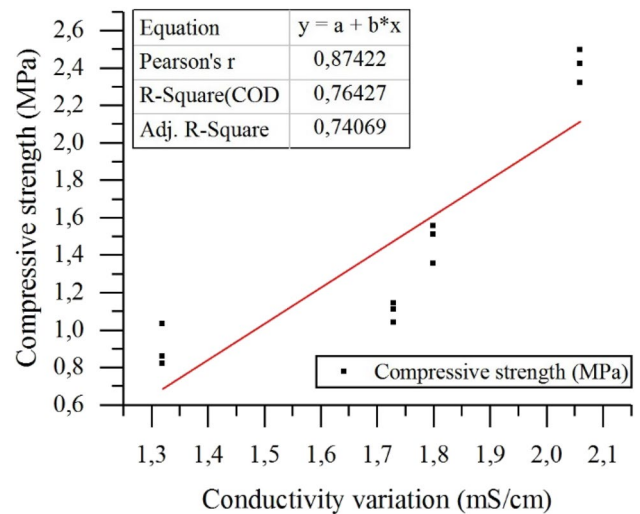

Fig. 9 Relationship between the conductivity variation and the compressive strength of the SCBAs

authors who state that the pozzolanic reaction is initially governed by the specific surface area [12].

A positive correlation (Pearson's $r = 0.986$) exists between the specific surface area of the ashes and their compressive strength (Fig. 8), agreeing with Cordeiro and Kurtis [11]. A positive correlation (Pearson's $r = 0.874$) also exists between the chemical reactivity determined with the conductivity test and the compressive strength (Fig. 9), agreeing with Walker and Pavia [13].

The results evidence that reactivity increases with rising amorphousness, fineness and surface area, and that the carbon content does not eliminate reactivity: the ashes with the greatest carbon content (SCBA 1 and 2) show more significant strength development and early lime combination, whereas SCBA 3 and SCBA 4, with lower carbon (LOI ≤ 0.41), present low reactivity. This agrees with Chusilp et al. [24] who claim that high carbon content in ground bagasse ash does not adversely affect the properties of a mortar.

The lower reactivity of the SCBA ashes 3 and 4 is partly due to strong contamination with soil sand (quartz).

It is widely acknowledged that not only the chemical evolution of the pozzolanic reaction but also the physical filler effect of the pozzolans contribute to the strength of pozzolan composites. However, in the ashes studied, the pozzolanic reaction seems to override the physical effect, as the strength does not seem to change significantly with decreasing particle size.

It seems to form the results that the alumina, iron and alkali content in the ashes are too low to affect pozzolanic activity, as SCBA1 has the highest contents, and yet, SCBA 2 combines more lime in the short term and reaches greater strength. However, this trend is likely to change if the ashes were to contain higher amounts of these elements. Also, the iron and aluminium are present as hematite and corundum which are highly insoluble and unlikely to participate in the pozzolan-lime reactions (Tables 3 and 9).

5 Conclusion

The sugar bagasse ashes investigated show pozzolanic activity. C-S–H was evident at 7 days, and the ashes combined lime and produced strengths similar or superior

Table 9 Summary of results

Ash	SSA	Fineness %		Amor-phous-ness	LOI	Chemical composition					Conductivity drop		Strength	
		>45 μm	>75 μm			SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	24 h	Total	C s MPa	F s MPa
SCBA 1	25.63	6.19	3.35	S–I	24.10	39.00	13.65	11.40	3.19	1.95	10.61	18.37	1.95	0.41
SCBA 2	78.33	6.19	1.18	S–I	23.60	59.10	4.19	2.01	2.42	3.73	11.12	21.02	3.62	0.36
SCBA 3	0.51	44.72	32.96	C	0.41	96.80	0.74	2.06	0.25	0.49	9.90	13.47	1.35	0.28
SCBA 4	2.19	42.50	30.41	C	0.31	93.40	1.69	2.69	0.36	0.71	10.20	17.65	1.65	0.46

to the control mix at 28 days. Ever the coarse ashes with lower specific surface areas combine lime and produce strengths comparable to the control specimen.

Despite the low amorphousness and high carbon contents of some of the ashes, reactivity was evidenced by means of physical and chemical tests, and the results of the mechanical index and conductivity tests are comparable to those by previous authors. The ashes show activity despite the presence of significant crystalline phases such as quartz, corundum and the iron oxide hematite, that are considered non-reactive. In addition, the ashes with the greatest carbon content are the most reactive and their composites reached the highest strength. Fineness triggers the physical packing effect of the pozzolans that increases the strength; however, in the ashes studied, the reactivity seems to override the physical effect, as the strength does not seem to change significantly with decreasing particle size.

A positive correlation exists between the compressive strength and the specific surface area; and between the compressive strength and the chemical activity agreeing with previous authors. It seems from the results that the alumina, iron and alkali content in the ashes do not affect pozzolanic activity, as SCBA1 has the highest contents, and yet, SCBA 2 combines more lime in the short term and reaches a greater 28-day strength. Some of these elements are present as hematite and corundum which are highly insoluble and unlikely to participate in the pozzolan-lime reactions.

The anions and alkalis capable of forming detrimental salts and causing expansion are extremely low or nonexistent in the ashes, well under the standard requirements for fly ash and GGBS.

The reactivity, together with the lack of dangerous and toxic components, place these ashes in a favourable standpoint to be used as binder replacement in construction.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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