



New use of sugar cane straw ash in alkali-activated materials: A silica source for the preparation of the alkaline activator

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HIGHLIGHTS

- Sugarcane straw ash (SCSA) was used as activator for alkali activated material (AAM).
- The new activator was prepared by reaction of SCSA with NaOH.
- The SCSA-based activator was used for the alkali activation of blastfurnace slag.
- Similar behaviour for SCSA and rice husk ash (RHA) was observed.
- SCSA was a good silica source for replacing sodium silicate in AAM.

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ABSTRACT

Alkali silicates, expensive and highly pollutant chemical reagents, are required to produce the alkaline activator for high-performance alkali-activated materials. This study presents a new silica source for producing the alkaline activator, sugar cane straw ash (SCSA). An activating suspension was prepared with SCSA and NaOH by means of a thermal bottle. The ash reacting time inside the thermal bottle (τ) was assessed from 0 to 48 h, and the SCSA amount in suspension, represented by the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio (ε), was analysed from 0 to 1.82. Compressive strengths were obtained from blast-furnace slag-based mortars that were cured for three days at 65 °C, with the optimal mortars produced when $\tau = 24$ h and $\varepsilon = 1.46$. Comparison of these new SCSA systems with two common silica sources, sodium silicate chemical reagent and rice husk ash, revealed that SCSA yielded lower results than the former and similar results to the latter silica source.

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1. Introduction

Alkali-activated materials (AAMs) are alternative binders to Portland cement. Their advantages over Portland cement are lower energy consumption and reduced CO₂ emissions during the manufacturing process [1,2]. AAMs are obtained by mixing an amorphous aluminosilicate (solid precursor) with an alkaline activator (also called by activating solution or alkaline solution), where the product from this chemical reaction hardens and forms a material with interesting mechanical properties [1,2]. Examples of common solid precursors are blast-furnace slag (BFS), metakaolin and fly

ash. The most common reagents in alkaline solutions are alkali hydroxides and silicates [1,2].

The use of waste materials in AAM production has become a recent trend [3–5]. This study describes the utilisation of sugar cane straw ash (SCSA), an ash rich in amorphous silica (SiO₂), in AAM production. The management of this waste is a particular interest due the recent increase of sugar cane production in Brazil. In 2014, this country produced 736.11 million tonnes of sugar cane were produced, which accounts for ~40% of the worldwide production [6]. The potential of SCSA in AAM comes from prior studies that assessed its reactivity as pozzolan in calcium hydroxide blends [7–9]. Authors observed that the ash presented high reactivity [7,8], where the SCSA activity was also compared to a densified silica fume [9]. The justification for such SCSA reactivity is due the presence of the amorphous phase (mainly silica) observed in

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X-ray diffraction. In the tests carried out, SCSA consumed large amount of calcium hydroxide to form C-S-H (calcium silicate hydrate) gels. This reaction product was observed in the following tests: X-ray diffraction [7], thermogravimetric analysis [7,9], Fourier transform infrared spectroscopy and electrical conductivity measurement [9]. As consequence for the high consumption of calcium hydroxide observed in these studies, SCSA was utilised as partial replacement of the Portland cement. Authors concluded that the replacement percentage range of 20–30% presented similar mechanical behaviour than the control [10–12]. The pozzolanic characteristic of the SCSA was the reason for such good results: the consumption of portlandite was observed by thermogravimetric analysis [10] and X-ray diffraction [11]. Related to the use of SCSA in AAM, the ash was previous studied as solid precursor in a binary system with blast furnace slag [13]. The samples of blast furnace slag and SCSA presented higher compressive strength than the control since 3 days of curing at 25 °C. Authors observed that the amorphous silica from SCSA improved the blast furnace slag matrix, explaining the great results. Now, this study presents another use of SCSA in AAM production, with SCSA being utilised as a source of silica in the production an alkaline suspension (alkaline activator).

The SiO_4^{4-} anion is a key component in high-performance AAMs, because it favours the formation of a denser and stronger structure [1,2]. Sodium silicate (SS) currently provides an important reactive part of silica that is widely used in combination with sodium hydroxide to produce the activating solution [1,2]. However, the main disadvantages of SS are mainly due that it is the most expensive raw material to produce AAM and the highest emitter of greenhouse gases among the basic materials [14–17]. The cost of the sodium silicate can be around of 20% to produce a AAM concrete, depending on the dosage. About the greenhouse gases, authors found that emissions from the production of the sodium silicate represents a 50–70% of the total in an AAM concrete design. This high value is due the melting of the raw materials (silica and sodium carbonate) until 1400 °C to produce the sodium silicate [17]. One of the key research targets in the near future will thus be the assessment of new sources of silica to produce the activating solution due the sodium silicate disadvantages [18,19]. One of the key research targets in the near future will thus be the assessment of new sources of silica to produce the activating solution [20–25]. Results from these studies about new silica source to the alkaline activator will be discussed in the Results and Discussions section, where they will be compared to the use of SCSA to the same purpose.

The main objective of this study is to assess the potential of employing SCSA as the silica source to prepare the alkaline suspension (alkaline activator). SCSA was utilised in combination with sodium hydroxide to produce an aqueous NaOH/SCSA suspension by means of a thermal bottle. The resulted blend of water, NaOH and SCSA were not filtered, resulting in a suspension and not in a solution. The study is divided into three parts: 1) deriving the optimum time for dissolving the ash to prepare the activating reagent; 2) understanding the influence of SCSA on the activating reagent; and 3) comparing SCSA to commercial SS chemical reagents and alternative silica sources. After this study, it is expected to produce an alkaline reagent with best SCSA content and the finest dissolution time in a thermal bottle.

2. Experimental

2.1. Materials and equipment

BFS was supplied by Cementval S.A., Puerto de Sagunto, Spain, in the form of large grains, which was then milled in a ball mill for 30 min to obtain a fine material that could be used as the solid precursor in AAM production. The chemical composition of BFS is shown in Table 1. BFS is primarily composed of CaO, SiO_2 , Al_2O_3 and MgO. Its mean particle diameter (D_{mean}) was 25.6 μm . Sodium hydroxide (NaOH, or NH in the context of solution composition and specimens nomenclature) is the chemical reagent utilised to prepare the alkaline solutions and suspensions. This material was used in form of pellets, and was supplied by Panreac S.A., with a purity of 98%. Natural sand from Caolines Lapidra (Lliria, Valencia, Spain), with a fineness modulus of 4.30, was utilised to prepare the mortars.

SCSA was utilised as the silica source to produce the activating suspension. The ash preparation process consisted of the following steps. The straw was first collected from sugar cane plantations near Ilha Solteira city (São Paulo, Brazil), and then transformed to ash by means of an autocombustion process. This procedure took 6 h, and a maximum temperature of 700 °C was observed during the combustion process. The ash was then sieved (MESH #50; 300 μm) to remove any unburned particles. To complete the process, the sieved ash was milled in a ball mill for 50 min. This milling process was carried out to reduce liquid absorption capability of the ash and, consequently, enhancing rheological properties of the fresh AAM. Table 1 shows the chemical composition of SCSA, with SiO_2 (58.6 wt%), Al_2O_3 (9.0 wt%) and Fe_2O_3 (8.4 wt%) being the most abundant compounds. $D_{mean} = 18.1 \mu\text{m}$ after the milling process. The amount of insoluble residue was also determined for SCSA (34 wt%).

RHA was another silica source utilised to produce the activating suspension. This ash served as a comparison material to SCSA. RHA was supplied by DACSA S.A., Tabernes Blagues, Spain, and it was utilised as received ($D_{mean} = 62.3 \mu\text{m}$). Its chemical composition is summarized in Table 1, with the main oxide being SiO_2 (85.6 wt%). The RHA was not milled prior to use, because a previous study concluded that the particle diameter of this ash did not influence the mechanical AAM properties [20]. Unlike RHA, the use of unground SCSA presented rheological problems during the AAM preparation due to considerable water adsorption, thus requiring SCSA to be milled to achieve the appropriate workability of the samples.

Commercial SS was also utilised as a silica source. This chemical reagent was acquired from Merck, and its chemical composition was 8 wt% Na_2O , 28 wt% SiO_2 and 64 wt% H_2O .

2.2. Preparation of alkali-activated materials

Three parameters were held constant during the AAM analysis, with the water/BFS ratio, sand/BFS ratio (for mortars) and Na^+ molality fixed at 0.45, 3.0 and 4 $\text{mol}\cdot\text{kg}^{-1}$, respectively. The preparation of the alkaline suspension and solution played an important role in this study, with two different suspensions and two different solutions analysed: NH/SCSA, NH/RHA (both are suspensions), NH

Table 1
Chemical composition of BFS, SCSA and RHA, in wt%.

Raw materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	SO_3	Cl	Others	LOI
BFS	29.9	10.6	1.3	40.2	7.4	0.6	1.9	–	2.6	5.5
SCSA	58.6	9.0	8.4	4.6	1.6	5.4	1.9	0.7	3.3	6.5
RHA	85.6	0.3	0.2	1.8	0.5	3.4	0.3	0.3	0.6	7.0

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