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Additional Information

Improving the reactivity of a former ground sugarcane bagasse ash produced by autogenous combustion through employment of two different additional grinding procedures

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ABSTRACT

Studies on reactivity of sugarcane bagasse ash (SCBA), obtained by an autogenous combustion process, with low loss on ignition and three different particle sizes were carried out (SCBA-1, SCBA-2 and SCBA-3). The ashes were characterized by their

particle size distribution, chemical composition, X-ray diffraction (XRD), thermogravimetric analysis (TGA) and field emission scanning electron microscopy (FESEM). The ash with lowest particle size was SCBA-3, followed by SCBA-2, which both are finer than SCBA-1. Calcium hydroxide/SCBA blends were assessed by means of loss of electrical conductivity (Lc), TGA and FESEM. Portland cement/SCBA pastes were analyzed through isothermal calorimetry, TGA, FESEM and compressive strength. Results showed that SCBA with lowest particle size (SCBA-3) presented highest reactivity, which resulted in statically significant better compressive strength than that of control paste (with only Portland cement) after 28 days of curing. The improvement on compressive strength by employing SCBA-3 respect to control and SCBA-1 were 17.9 and 14.1%, respectively, after 180 days of curing.

Keywords: pozzolan characterization, calcium hydroxide/pozzolan paste, Portland cement/pozzolan paste, microstructural study, compressive strength test.

Highlights:

- Sugarcane bagasse ash (SCBA) was produced by autogenous combustion.

- SCBA samples with three different particle sizes were assessed by their pozzolanic reactivity.

- The lower the particle size of SCBA, the higher the reactivity.

- The improved reactivity lead to significant higher compressive strengths in pastes.

51 **INTRODUCTION**

52

53 Supplementary cementitious material (SCM) is composed mainly of siliceous or

54 aluminosiliceous compounds in amorphous phase and high specific surface area.

55 Nowadays, different SCMs are being widely used to improve the mechanical properties

56 and durability of Portland cement-based products. This improvement results from

57 chemical, filler and heterogeneous nucleation effects [1–3]. Some phases of SCM react

58 chemically with the portlandite generated from the Portland cement hydration. This

59 reaction leads to forming cementing compounds similar to the hydrates from Portland

60 cement reactions (e.g. C-S-H and C-A-S-H) [4]. The filler and nucleation effects are

61 related to the particle size and specific surface area of SCM. In this case, the filler effect

62 is characterized by particles of SCM that improve the packing density of the hydrated

63 Portland cement matrix, which reduces porosity and increases mechanical properties and

64 durability [5]. Regarding to nucleation effect, it is related to the increase of nucleation

65 sites by the presence of SCM fine particles in the paste [1]. These nucleation sites increase

66 the available surface area for hydration product precipitation. Consequently, the hydration

67 process of the Portland cement accelerates, which forms a denser structure that increases

68 the mechanical behavior at early ages [5–8].

69

70 The SCM performance is mostly influenced by its mineralogy, chemical composition and

71 physical properties. These properties highly influence the chemical, filler and

72 heterogeneous nucleation effects [8-10]. Related to SCM mineralogy, studies showed that

73 most crystalline phases (e.g. quartz) present low solubility in cement pore solution [7]. In

74 contrast, the amorphous phase is more soluble than crystalline phases, which results that

75 the former is generally more reactivity than the latter [8]. Regarding to chemical

76　composition of SCM, its can be mainly composed by SiO_2, Al_2O_3 and CaO. Considering

77　the reactive part of SCM, chemical composition influences on the chemistry and content

78　of reaction products [9]. For example, an increase of a reactive siliceous-based SCM in

79　Portland cement paste results on a decrease of the portlandite content, and it is expected

80　a formation of a C-S-H with lower C/S ratio than those generated in the cement hydration.

81　In the case of an SCM composed by reactive aluminosilicates added to a Portland cement

82　paste, it is also detected a reduction of the portlandite content in the paste, but it is

83　expected some formation of C-A-S-H and a AFm phase. Respect to physical properties

84　of SCMs, it can be highlighted the particle size distribution and specific surface area,

85　which can vary according to nature and source of the SCMs [10]. These parameters

86　influence mostly the filler and heterogeneous nucleation effects.

87

88　Silica fume, fly ash and metakaolin are examples of SCM commonly utilized in cement-

89　based materials [4]. In the case of developing countries, the interest in agroindustry

90　residues to be utilized as SCM materials increased in the last decades due their great

91　availability and potential to improve the Portland cement-based composites properties

92　[11]. In this way, one of these wastes is the sugarcane bagasse ash (SCBA). In fact,

93　sugarcane has experienced an increase in production of over 50% in the last decade

94　especially due to ethanol production [12]. Consequently, the bagasse, the main sugarcane

95　by-product, increased in the same way. The bagasse is usually utilized as biomass to

96　produce energy in boilers, and this process yields the waste called sugarcane bagasse ash.

97

98　The SCBA composition is mainly based on silica in both phases, amorphous and

99　crystalline [7,13]. The presence of crystalline phases may be due the soil contamination

100　by quartz or when the burning process reaches high temperatures to form cristobalite [14].

101 However, these crystalline phases are not interesting due its low reactivity [13,15].

102 Therefore, a solution found by researchers is to grind the SCBA to an appropriate particle

103 size [7,13,14]. The grinding process can increase the reactivity of the SCM and also can

104 favor both filler and nucleation effects. Previous studies concluded that crystalline phases

105 can present some reactivity when the ash is ground [15]. Another problem detected in

106 recent studies is the incomplete calcination from power plants that results in high loss on

107 ignition (LOI). Therefore, a careful calcination or a necessity to reburn the ash is one of

108 the solutions improve SCBA pozzolanic activity [16-17].

109

110 Thus, this study intends to produce a sugarcane bagasse ash (SCBA) by an autogenous

111 burning process, which is a process that demands low energy requirement to produce an

112 ash with low LOI, and to assess three different grinding processes in order to determine

113 the highest reactivity of the SCBA. These SCBA samples were tested in blends with

114 calcium hydroxide, their activity in Portland cement pastes and the influence on

115 mechanical properties of cement-based pastes. The compressive strength results were

116 compared using one-way analysis of variance (ANOVA) followed by Tukey test with a

117 p-value of 0.05. The aim is to determine the best grinding process to use the total potential

118 of the SCBA.

119

120 **EXPERIMENTAL**

121

122 *SCBA production and characterization*

123

124 Sugarcane bagasse was obtained from Suzanápolis (Brazil). This bagasse was burnt by

125 an autogenous burning process for 14 hours with a peak temperature of 700 °C. Then, the

126 ash was sieved (300 μm mesh size) for 10 minutes to remove some unburned particles.

127 After that, the bottom ash was dry-ground using a tumbling mill (cylindrical shape, 100

128 cm length and 80 cm diameter) to generate the SCBA-1, wherein the grinding

129 specifications were an ash input of 5 kg for 50 min, employing 52.5 kg of steel cylinders

130 of 25.4 mm height and 19.1 mm diameter as grinding media. From SCBA-1, two other

131 samples were obtained by distinct grinding procedures. First, an SCBA (named SCBA-

132 2) was produced by using a Fritsch Pulverisette 5 planetary mill for 20 min, with 30 g of

133 feed, 43 10-mm zirconia spheres, and 0.3 wt% of commercially available glycol-based

134 grinding aid (containing 48% of $C_4H_{10}O_3$ and $C_2H_6O_2$ active chemicals) in a 500 mL

135 grinding vessel. The third ash (denominated as SCBA-3) was produced by using a

136 Szegvari attrition mill, model 1-S (Union Process, Inc., U.S.A.) with 50% filling and

137 ethanol P.A. (99.9%) as dispersant (640 kg/t solid/solution mass ratio) in a 1200 mL

138 grinding vessel. The total grinding time was 6 h, and 360 cm^3 of 3-mm diameter zirconia

139 spheres were used as grinding media. After grinding, SCBA-3 was oven-dried at 60 °C

140 for 24 h.

141

142 The particle size distribution of SCBA samples was obtained using a Malvern Instruments

143 Mastersizer 2000. SCBA chemical compositions were determined by energy dispersive

144 X-ray fluorescence spectrometry (Shimadzu EDX 720). BET specific surface area was

145 assessed by N_2 adsorption using a Micromeritics ASAP 2020 device. The samples were

146 treated at 150 °C during 8 h. X-ray diffraction (XRD) of the SCBA specimens was

147 performed using a Bruker D8 Focus diffractometer with Cu-Kα operation at 35 kV and

148 35 mA, angular range from 8 to 60°, 0.03° step size and 1 s per step. Thermogravimetric

149 analysis (TGA) was performed by means of a Mettler-Toledo TGA 850 model for SCBA

150 samples and reactivity studies of calcium hydroxide-SCBA (CH-SCBA) and cement-

151 SCBA (PC-SCBA) pastes. Brazilian type V Portland cement (high-early strength) was

152 used in this study and its chemical composition is shown in Table 1. Field emission

153 scanning electron microscopy (FESEM) was used to investigate SCBA and CH-SCBA

154 specimens by a ZEISS Supra 55. Electrical conductivity of CH-SCBA suspensions was

155 measured by a Crison micro CM2201 instrument. The suspensions were maintained in a

156 controlled temperature using a Julabo SW22 shaking thermal bath.

157

158 Table 1 – Chemical composition of cement and SCBA-1, in wt%

159

160 *Reactivity studies in calcium hydroxide-SCBA (CH-SCBA) blends*

161

162 Reactivity studies on CH-SCBA samples were carried out by means of electrical

163 conductivity of CH-SCBA suspensions, thermogravimetric analysis and FESEM

164 micrographs of CH-SCBA pastes. Calcium hydroxide (purity > 95%) was supplied by

165 Panreac S.A. The electrical conductivity test was performed as proposed by Tashima et

166 al. [18]. This test consists of adding 1 g of solid material (sum of CH and SCBA) in an

167 Erlenmeyer flask with 50 mL of deionized water. The flask is maintained in a shaking

168 thermal bath with constant temperature (40, 50 or 60 °C) during the testing period of 7

169 days. Electrical conductivity values were measured after the following times: 0, 4, 8, 24,

170 48, 72, 96, 120, 144 and 168 hours. The CH:SCBA proportions assessed were 2:8 to 4:6

171 for SCBA-1, 2:8 to 3.5:6.5 for SCBA-2 and 2:8 to 4.5:5.5 for SCBA-3 (by mass). Results

172 were presented by means of the loss of electrical conductivity (Lc) in order to classify the

173 SCM as proposed by Tashima et al. [18]. After the end of the test, the suspensions were

174 filtered, and the solid part that remained was passed by thermogravimetric analysis in the

175 temperature range of 35–600 °C at a heating rate of 10 °C/min in N_2 atmosphere (gas

176   flow of 75 mL/min). Regarding CH-SCBA pastes, thermogravimetric analysis was

177   carried out in the same conditions as the solid part of the electrical conductivity tests. The

178   CH:SCBA proportion analyzed for pastes was only 5:5 (by mass) for the SCBA-1, SCBA-

179   2 and SCBA-3 samples with a constant water/solid ratio of 0.80. The pastes were assessed

180   after 1, 2, 3 and 7 days of curing at 20 °C and relative humidity (RH) higher than 95%.

181   Finally, regarding FESEM studies, only the SCBA-1 blended with calcium hydroxide

182   was assessed. The CH-SCBA-1 ratio was 5:5 at a water/solid proportion of 0.80, cured

183   after 7 days at 20 °C and RH > 95%. The nomenclature for these pastes is CH-SCBA-x-

184   y/z, where x is associated with SCBA type (1, 2 or 3 for SCBA-1, SCBA-2 and SCBA-3,

185   respectively) and y/z is the CH:SCBA proportion.

186

187   *Reactivity studies and compressive strength of cement-SCBA (PC-SCBA) pastes*

188

189   Reactivity studies on PC-SCBA samples were performed by calorimetric and

190   thermogravimetric analyses in cement-based pastes. Brazilian type V Portland cement

191   (high early strength with 512 m^2/kg Blaine fineness) was used in this work. Isothermal

192   calorimetry tests were performed at 25 ± 0.02 °C using a Calmetrix I-CAL 2000

193   calorimeter. Duplicate samples containing approximately 50 g from each mix were

194   evaluated. Pastes were monitored for 3 days. The PC-SCBA proportions assessed were

195   100/0 (control, named PC-CTRL), 90/10 and 80/20 at a constant water/solid ratio of 0.50

196   for the three SCBA samples. The nomenclature for these pastes is defined by PC-SCBA-

197   x-y/z. Again, x is related to the SCBA type, and the y/z value is the cement/SCBA

198   proportion. Each paste was mixed by adding solids to mixing water and superplasticizer

199   in a 100 mL plastic beaker, and stirring by hand with a spatula for 2 min.

200   Thermogravimetric analysis of PC-SCBA pastes was performed in the temperature range

201 35–600 °C with a heating rate of 10 °C/min in N_2 atmosphere (gas flow of 75 mL/min).

202 Using the same mixes of the calorimetry, pastes were evaluated after 1, 2, 3, 7, 28 and

203 365 days of curing at 20 °C and RH > 95%.

204

205 The specimens of all mixes for compressive strength tests were prepared in a 5 L planetary

206 mixer for 3 min. Three cylindrical specimens (2.5 cm diameter and 5 cm high) for each

207 mix were cast and maintained in a 100% relative humidity container for 24 h to prevent

208 moisture evaporation. After, the specimens were demoulded and transferred to a lime-

209 saturated water bath at 25 °C until testing. Compressive strength tests were carried out at

210 3, 7, 28 and 180 days of curing on a Shimadzu UHI-500kNI hydraulic servo-controlled

211 machine, with a displacement rate of 0.5 kN/s. The results were submitted to one-way

212 analysis of variance (ANOVA) with Tukey's multiple-range test statistical analysis

213 (analysis of variance) to assess significant differences between pastes using the software

214 Statistica 10 (StatSoft Inc.).

215

216 **RESULTS AND DISCUSSION**

217

218 *SCBA characterization*

219

220 Fig. 1. Particle distribution of SCBA-1, SCBA-2 and SCBA-3: cumulative passing (left),

221 and volume (right)

222

223 Table 2 – Particle size diameters and BET specific surface area of SCBA-1, SCBA-2 and

224 SCBA-3.

225

226 Particle size distributions of SCBA-1, SCBA-2 and SCBA-3 are depicted in Fig. 1. Table

227 2 shows the particle diameter for cumulative passing of 10% (D_{10}), 50% (D_{50}), 90% (D_{90})

228 and the mean particle size (D_m). Regarding the SCBA-3 sample, it presented a D_m of 3.62

229 μm, and 92% of the particle volume were particles with diameter less than 10 μm, while

230 SCBA-2 had D_m of 15.48 μm, and 53% of the particle volume were particles with

231 diameter less than 10 μm. In the case of SCBA-1, its D_m was 26.58 μm, and 44% of the

232 particle volume were particles with diameter less than 10 μm. It is observed that the

233 grinding process was effective: SCBA-3 presented the lowest particle size, followed by

234 the SCBA-2, where both were finer than the SCBA-1. In addition, it can be observed that

235 the SCBA-3 presented the most homogenization of the particles considering that the

236 diameters of the particles are less than 30 μm, with a high concentration of particles of

237 size in the diameter range of 1–2 μm (Fig. 1b). On the other hand, SCBA-1 and SCBA-2

238 presented more heterogeneous particle sizes. Specifically, regarding the SCBA-1, there

239 are particles around 100 μm in diameter that were not observed for the other two SCBA

240 samples. BET specific surface area values are also presented in Table 2, and they

241 corroborated the particle size results. In fact, the lowest BET value was observed for

242 SCBA-1, which was the ash that suffered the softest grinding among the three evaluated

243 samples. The ultrafine wet-grinding of SCBA-3 was responsible for a significant increase

244 in specific surface area. SCBA-2 showed an intermediate BET value between that of

245 SCBA-1 and SCBA-3, which is in accordance with the different grinding processes

246 adopted in this work.

247

248 Fig. 2. X-ray diffraction patterns of SCBA-1, SCBA-2 and SCBA-3 (key: quartz, Q;

249 calcite, C; orthoclase, O)

250

251   The chemical composition of SCBA-1 is shown in Table 1. As expected for this type of

252   pozzolan [7,11], the major oxide in its composition is SiO_2. Other compounds with

253   significant quantities were K_2O, Al_2O_3, CaO, Fe_2O_3, and SO_3. The content of K_2O may

254   modify the amount of available alkalis in cement, causing changes in the fresh and

255   hardened properties of cement-based material. Thus, the amount of SCBA that could be

256   used as partial replacement of cement should be carefully observed. In this work,

257   considering a maximum replacement level of 20% for SCBA, the alkali equivalent

258   content ($Na_2O_{eq} = Na_2O + 0.658$ x K_2O) changed from 0.7% (low-alkali cement) to 2.2%

259   (high-alkali cement). This increase in Na_2O_{eq} could be a limiting factor for SCBA-cement

260   mixes considering durability aspects [19]. It is important to note the relatively low loss

261   on ignition (LOI) for this type of ash when compared to one obtained in power plants

262   [17], which was due to the uncontrolled autogenous burning of sugarcane bagasse. X-ray

263   diffraction patterns of three SCBA samples are shown in Fig. 2. The main crystalline

264   phases observed were quartz (SiO_2, PDF Card #331161), calcite ($CaCO_3$, PDF Card

265   #050586) and orthoclase ($KAlSi_3O_8$, PDF Card #310966). In a higher magnification of

266   the SCBA-1 diffractogram (Fig. 2), it can be observed that there is a deviation in the

267   baseline in the Bragg angle range of 15–40°, which is characteristic for the amorphous

268   phase of the ashes.

269

270   Fig. 3. DTG curves of SCBA-1, SCBA-2 and SCBA-3

271

272   DTG curves of SCBA-1, SCBA-2 and SCBA-3 are shown in Fig. 3. Peaks in five

273   temperature ranges were detected. In the first temperature range (35–265 °C) and the

274   second temperature range (265–310 °C), the mass loss was common for the three ashes,

275   and it is related to the water loss of the samples [20]. Related to the third temperature

276 interval (310–360 °C), only a band for the SCBA-2 was noticed; this mass loss is related

277 to the decomposition of the grinding aid. The mass loss in this interval for SCBA-2 was

278 0.80%; whereas, for SCBA-1 and SCBA-3, it was 0.30% for both ashes. Regarding the

279 fourth range (360–560 °C), it is related to the decomposition of unburned carbon in the

280 ashes. It is noticed that there are two peaks in the DTG of SCBA-2 and SCBA-3, whereas

281 there is only one peak in the DTG of SCBA-1. As observed in the particle size studies,

282 the particle diameter for SCBA-2 and SCBA-3 is finer and better defined than for the

283 SCBA-1, which makes the peaks on DTG to be well defined. Finally, regarding the fifth

284 temperature interval (560–640 °C), which is related to the decomposition of carbon

285 dioxide from the carbonates, it was observed that SCBA-1 presented the highest mass

286 loss compared to SCBA-2 and SCBA-3 (0.80, 0.20 and 0.15%, respectively). These

287 results are in accordance with those observed for XRD analysis, where it is observed that

288 there are more intense peaks of calcite for the SCBA-1 than for SCBA-2 and SCBA-3.

289

290 Fig. 4. SEM micrographs of SCBA-1 (a, b and c), SCBA-2 (d, e and f) and SCBA-3 (g,

291 h and f)

292

293 SEM micrographs of SCBA-1, SCBA-2 and SCBA-3 are shown in Fig. 4. SCBA-1

294 particles present irregular shape, which can be detected some particles with equiaxial,

295 elongated and plate forms (Fig. 4a). Some thick quartz particles can be detected in SCBA

296 (Fig. 4b). In a higher magnification, it can be observed the irregular form with more detail

297 (Fig. 4c). Regarding to shape of SCBA-2 particles, it presented more regular particles

298 than SCBA-1, which were detected mostly equiaxial (Fig. 4d) and more rarely some

299 elongated particles (Fig. 4e). Fig. 4f shows some equiaxial particles of SCBA-2. Related

300 to morphology of SCBA-3 particles, it was presented the most homogeneous distribution

301 than the other two ashes (Fig. 4g). As the other ashes, the shape of SCBA-3 particles is

302 irregular, which were detected some mostly equiaxial forms (Fig. 4h and Fig 4i).

303

304 *Reactivity studies in CH-SCBA blends*

305

306 Fig. 5. Loss of electrical conductivity (Lc) for SCBA-1 (a, b and c), SCBA-2 (d, e and f)

307 and SCBA-3 (g, h and i) for testing temperatures of 60, 50 and 40 °C

308

309 Results of electrical conductivity studies were represented by means of loss of electrical

310 conductivity (Lc, in percentage). This representation allows to classify the SCM reactivity

311 using a chart proposed by Tashima et al. [18]. Fig. 5 shows Lc values of the SCBA-1,

312 SCBA-2 and SCBA-3 in the testing temperatures of 60, 50 and 40 °C for specific

313 CH:SCBA ratios. It is noticed that, for some blends, the loss of electrical conductivity

314 value is maintained as constant during the testing time, and, for other blends, the Lc values

315 increased with the testing period. The first behavior occurs because the SCM in the blend

316 cannot turn the suspension into an unsaturated state: the concentration of Ca^{2+} and OH^-

317 did not reduce during the testing time. On the other hand, when the Lc values increase, it

318 means that the SCM was able to turn the suspension state into an unsaturated one. In this

319 case, the blends that presented values of Lc higher than 20% after the testing time were

320 considered as unsaturated. For the three SCBA samples, it can be observed that blends

321 reached unsaturation more quickly according to higher temperatures. In addition, there

322 are more blends that reached the unsaturated state for the highest temperature than those

323 in the lowest one. These behaviors were expected, since the SCM reaction is accelerated

324 in a higher temperature. In order to classify the SCM, it will be indicated which blends

325 were in the unsaturated state for each SCBA sample and testing temperature. For the

SCBA-1 sample, the blends that turned into an unsaturated state were CH:SCBA ratios

of 2:8 (for 60, 50 and 40 °C), 2.5:7.5 (for 60, 50 and 40 °C) and 3:7 (for 60 and 50 °C).

In the case of SCBA-2, the blends that achieved unsaturation were 2:8 and 2.5:7.5 (for

60, 50 and 40 °C). Finally, for the SCBA-3 specimen, four blends reached the unsaturated

state: 2:8 (for 60, 50 and 40 °C), 2.5:7.5 (for 60, 50 and 40 °C), 3:7 (for 60 and 50 °C)

and 3.5:6.5 (only for 60 °C). Noting the blends that achieved an unsaturated stated for

each SCM in the chart proposed by Tashima et al. [18], it can be concluded that the

reactivity of SCBA-1 and SCBA-2 is classified as low, and SCBA-3 as medium. SCBA-

1 and SCBA-2 presented similar reactivity. Moreover, the reactivity of SCBA-3 was

higher than that of the other two samples, which can be compared to the densified silica

fume and sugarcane straw ash [18,21]. According to this test, the grinding process utilized

for SCBA-3 increases its reactivity.

Fig. 6. DTG curves of the solid remaining in the flask for CH:SCBA ratios of 2:8 and 3:8

for the SCBA-1, SCBA-2 and SCBA-3

After the end of test, the solids remaining in the flask containing CH:SCBA proportions

of 2:8 and 3:7 from the three types of SCBA samples were assessed by TGA. Fig. 6 shows

the results by means of DTG curves. In this study, three temperature ranges present

interesting mass losses: 1) dehydration of the C-S-H compounds (100–180 °C); 2) mass

loss of the C-A-S-H compounds (200–240 °C); and 3) dehydration of the calcium

hydroxide (510–570 °C). In all DTG curves, it can be observed that there is no peak in

the third range because there is no calcium hydroxide in solid phase. This was expected,

since these blends presented Lc values higher than 20%. This behavior means that the

suspensions turned into an unsaturated state due the consumption of the calcium

hydroxide by SCBA. Consequently, the peaks in the first and second ranges presented some mass loss due the formation of C-S-H and C-A-S-H products from SCBA reaction with calcium hydroxide.

Fig. 7. DTG curves of 50-50 CH-SCBA samples cured after 7 days at 20 °C

Table 3 – TGA mass losses of the CH-SCBA samples after 1, 2, 3 and 7 days of curing at 20 °C (P_T: total mass loss; P_H: mass loss related to C-S-H/C-A-S-H; FL (%): Percentage of fixed lime)

Fig. 8. Linear relationship between P_H (%) and FL (%) parameters

TGA results on CH-SCBA pastes are represented in Fig. 7 by DTG curves for samples CH-SCBA1-50/50, CH-SCBA-2-50/50 and CH-SCBA-3-50/50 cured for 7 days at 20 °C. It can be observed that SCBA reacted with the calcium hydroxide since peaks in the first and second ranges, related to the C-S-H and C-A-S-H, respectively, were detected in TG curves. Despite the SCBA reaction that occurred with CH, all three ashes could not consume all the calcium hydroxide within the curing time analyzed. In fact, the peak related to the dehydration of the calcium hydroxide was observed in the third part of the DTG curves. Mass losses for the CH-SCBA samples cured after 1, 2, 3 and 7 days are listed in Table 3. Regarding to the parameters of this table, P_T is the total mass loss, P_H is the mass loss related to C-S-H/C-A-S-H dehydration and FL is the percentage of fixed lime. Values of P_H and FL for CH-pozzolan pastes were calculated according to Payá et al. [22]. In general terms, the values of P_H and FL increased with the curing time, indicating that all SCBA samples reacted with the calcium hydroxide from the pastes

376   (resulting in an increase of the fixed lime) and formed SCBA reaction products (leading

377   to an increase of the P_H value). Fig. 8 shows that there is a linear relationship between the

378   P_H and FL values. This linear relationship indicates that the calcium hydroxide consumed

379   (increase of the FL value) along the curing time took part in the SCM reaction to form

380   hydrated products (increase of the P_H value). For the three types of SCBA samples, the

381   main part of the SCBA reaction occurred in the first 3 days of curing: their FL values

382   were approximately 60% until this curing time. However, it was observed that there was

383   some SCM activity in the interval of 3–7 days due the increase of the FL values in this

384   curing interval (from 57–63% to 70–84%). This behavior shows that SCBA presents an

385   important reaction in the first days of curing, but there is still some activity in the longer

386   curing time. Comparing the three SCBA samples, CH-SCBA-3-50/50 presented the

387   highest reactivity until 7 days of curing (FL value of 84.23%), and CH-SCBA-1-50/50

388   and CH-SCBA-2-50/50 presented similar activity at the same curing time (FL values of

389   70.27 and 72.67%, respectively). These results are in accordance with the electrical

390   conductivity studies.

391

392   Fig. 9. SEM micrographs of the CH-SCBA-1-50/50 paste

393

394   Fig. 9 shows the micrographs of CH-SCBA-1-50/50 paste after 7 days of curing at 20 °C.

395   In Fig. 9a, a general view of the reaction products and an SCBA particle covered by

396   cementing gels can be observed. In a higher magnification (Fig. 9b), it can be noticed that

397   the cementing gels are the most abundant reaction products from the SCBA reaction.

398   Another product identified in the micrographs was the ettringite (Fig. 9c), which was

399   expected since the SCBA-1 presents some sulphates in its chemical composition. In

400   Figure 9d, a C-A-S-H product (like platelet structure) has been observed. As the lime

401   fixation of the CH-SCBA-1-50/50 was 70.23% after 7 days of curing, which indicates

402   that the SCBA-1 could not have reacted with all the calcium hydroxide, then some

403   unreacted particles of calcium hydroxide are common in the paste.

404

405   *Reactivity studies in PC-SCBA pastes*

406

407   Fig. 10. Specific heat rate (a) and released heat (b) from PC-SCBA pastes

408

409   Specific heat rate curves of all cement-based pastes are shown in Fig. 10a. The initial

410   period of hydration (first peak) was not evaluated in this study because cement wetting

411   occurred outside the calorimeter. In relation to the induction period (seen in detail in the

412   enlarged plots for the first 6 h of hydration in Fig. 10a), where the specific heat rate drops

413   to very low values, all mixes containing SCBA resulted in curves with a shift to the right

414   of the PC-CTRL curve. This could be explained by the presence of carbonaceous

415   compounds in all SCBA samples since loss on ignition of SCBA was 8.2% (Table 1).

416   This behavior was previously observed for an SCBA with high loss on ignition [17].

417   Moreover, the presence of grinding aid in SCBA-2 promoted a considerable extension in

418   the induction period [23] for PC-SCBA-2 mixes in comparison with PC-CTRL, which

419   was more visible for PC-SCBA-2-80/20. During the acceleration period (where the heat

420   rate increases quickly), two peaks were observed in all pastes. The first peak is associated

421   with the C_3S hydration, and the second one (maximum heat rate in all curves) corresponds

422   to the reaction of C_3A to form ettringite. This last peak was clearly increased in SCBA-

423   paste curves, which is related to the content of Al_2O_3 and SO_3 in significant amounts in

424   all SCBA samples. The evolved heat curves of all pastes are shown in Fig. 10b. In this

425   case, two tendencies could be observed for blended pastes in relation to PC-CTRL. In

426   general, the total heat was lower for SCBA pastes than that observed for the control, with

427   a more pronounced decrease in released heat for PC-SCBA-2-80/20 due to the significant

428   retardation effect [22]. This decrease could be related to the dilution effect provided to

429   cement replacement by different SCBA samples [7]. On the other hand, the released heat

430   curve of PC-SCBA-3-90/10 was very different compared to the other SCBA pastes. In

431   this case, the total heat was higher than that of PC-CTRL. The very high specific surface

432   area of SCBA-3 could explain this behavior, promoting heterogeneous nucleation that

433   improved considerably the cement hydration. The pozzolanic effect of this ash also may

434   contribute to increasing the released heat during the first days of hydration.

435

436   Fig. 11. DTG curves of PC-SCBA samples cured after 7 days at 20 °C

437

438   Table 4 – TGA mass loss of the PC-SCBA pastes cured after 1, 2, 3, 7, 28 and 365 days

439   at 20 °C (P_T: total mass loss; P_H: mass loss related to C-S-H/C-A-S-H; FL (%): Percentage

440   of fixed lime)

441

442   In TGA studies, DTG curves of PC-SCBA pastes cured for 7 days at 20 °C are depicted

443   in Fig. 11. The presence of SCBA in the pastes did not alter the reaction product

444   formation, since the pastes with SCBA presented peaks in the first and second

445   temperature intervals (related to C-S-H and C-A-S-H dehydration, respectively), which

446   is similar to the control. Related to the third temperature range (dehydration of

447   portlandite), the increase of SCBA proportion in the mix results in a reduction of the peak

448   depth, which can be justified by two factors: the reduction of the cement proportion in the

449   blend and pozzolanic reactions. Therefore, the higher content of SCBA in PC-SCBA

450   pastes resulted in portlandite consumption to form new reaction products.

451

452   Mass loss from TGA studies on the pastes cured after 1, 2, 3, 7, 28 and 365 days at 20 °C

453   are summarized in Table 4. Values of P_H and FL for cement/pozzolan pastes were

454   calculated according to Payá et al. [24]. For all pastes, the P_T and P_H values increased

455   until 7 days of curing and practically maintained as constant until 365 days. This behavior

456   indicates that a greater part of the reaction occurred during 7 days. Regarding the SCBA

457   influence on these parameters, P_H values from SCBA-containing pastes presented values

458   higher than that of the control paste in the first 3 curing days. This suggests that the SCBA

459   had positive effects on the mix in the early curing times. After this curing time, in the 7–

460   365 curing day period, the values were similar, with a small advantage to the SCBA pastes

461   over the control. Related to last parameter, the fixed lime (FL), it can have positive or

462   negative values. In the first case, the consumption of the portlandite by the SCBA results

463   in a positive value of fixed lime. In the second one, the fine particles from SCBA act as

464   nucleation sites, and more hydrates and portlandite could be observed in the PC-SCBA

465   paste compared to the reference. This means that there are more hydrates and portlandite

466   in a PC-SCBA paste than there should be in a cement paste corrected by the percentage

467   of cement from PC-SCBA paste. Therefore, it yields negative values of fixed lime. In the

468   first curing day, negative and positive values of fixed lime were determined. In both cases,

469   it resulted in the increase of the reaction products (by the accelerated cement hydration

470   or SCBA reaction), and this justifies the similar results of P_H values. Interestingly, PC-

471   SCBA-3-90/10 presented an absolute value higher than that of PC-SCBA-1-90/10 after 1

472   day of curing (−9.96 and −0.14%, respectively). This indicates that the SCBA-3 acted as

473   a nucleation site more effectively than SCBA-1 did. However, when the ash partially

474   replaces the cement by 20%, SCBA-SCBA-3-80/20 presented higher fixed lime value

475   than SCBA-SCBA-1-80/20 did. In this case, the finer particles from SCBA-3 in a higher

476   percentage in the blend could react more with the calcium hydroxide than the coarser ones

477   from SCBA-1 in the first day of curing. The fixed lime value increased mostly until 7

478   days of curing for all SCBA-pastes, and minor changes were observed in the 7–365-day

479   period. These P_H and FL values showed that greater part of the reaction occurred in curing

480   interval of 1–7 days. This is in accordance with TGA studies on CH-SCBA pastes, which

481   showed that important calcium hydroxide content was consumed in the first 7 days of

482   curing. In addition, these behaviors were also observed previously with another SCBA

483   [7].

484

485   An interesting fact observed in the TGA results is related to the pastes that contain SCBA-

486   2. The fixed lime of PC-SCBA-2 pastes presented higher values than those of PC-SCBA-

487   1 and PC-SCBA-3 pastes after the first day of curing. As shown in the CH-SCBA pastes,

488   the reactivity of SCBA-2 is similar to that of SCBA-1 and lower than that of SCBA-3.

489   Therefore, the high values found for lime fixation of the PC-SCBA-2 pastes are not

490   related to the portlandite consumption. Probably, it can be related to the inhibition of the

491   portlandite production from the cement hydration, as shown in calorimetric analyses (Fig.

492   10). Despite this, apparently the reaction products were not affected by the presence of

493   SCBA-2, since the mass loss related to the reaction products (P_H) is similar for other

494   pastes. This behavior was caused by using a grinding aid during the milling process to

495   produce the SCBA-2. In this way, some SCBA-2 sample was passed by three different

496   treatments in order to remove the grinding aid, and the resulting ash was assessed by TGA

497   to compare to the control and an SCBA-2 without treatment. The first procedure was to

498   wash the SCBA-2 sample with ethanol and acetone, solvents of the grinding aid,

499   producing the SCBA-2-EA. Another treatment was calcination of the SCBA-2 until 400

500   °C for 3 hours (temperature that decomposes the grinding aid, as observed in TGA

501 studies), yielding SCBA-2-400C. And the last treatment was to wash the SCBA-2 with

502 water, another solvent for the grinding aid, generating SCBA-2-W. In order the evaluate

503 the treated SCBA-2 samples, cement pastes in the proportion of PC-SCBA-2 equal to

504 80/20 were carried out in TGA studies after 1, 3, 7 and 28 days of curing at 20 °C.

505

506 Fig. 12. DTG curves of PC-SCBA2 samples cured after 7 days at 20 °C

507

508 Table 5 – TGA mass loss of the PC-SCBA-2 pastes cured after 1, 2, 3, 7 and 28 days at

509 20 °C (P_T: total mass loss; P_H: mass loss related to C-S-H/C-A-S-H; FL(%): Percentage

510 of fixed lime)

511

512 Fig. 12 illustrates DTG curves of pastes after 7 days of curing. Related to the peaks on

513 the first and second temperature ranges, significant differences were not observed.

514 Related to the third part of the curve, the treatments on the SCBA-2 were effective since

515 the peaks related to the portlandite for the treated SCBA-2 (PC-SCBA-2-80/20-EA, PC-

516 SCBA-2-80/20-400C and PC-SCBA-2-80/20-W) were deeper than that without any

517 treatment (PC-SCBA-2-80/20).

518

519 Table 5 shows the mass loss from the TGA studies on pastes after 1, 3, 7 and 28 days of

520 curing at 20 °C. In general, the mass loss related to the dehydration of reaction products

521 (P_H) for PC-SCBA-2-80/20 was higher than that of the pastes with the treated SCBA-2

522 until 28 days of curing. This suggests that the grinding aid did not affect the formation of

523 reaction products, as mentioned previously. However, based on the fixed lime values, an

524 important difference among the PC-SCBA-2 pastes is observed. For example, after 28

525 days of curing, the PC-SCBA-2 pastes with treatment presented FL values in the range of

526 39–65%, whereas for PC-SCBA-2-80/20 one it was 81.70%. In this case, the most

527 effective treatment was the calcination at 400 °C that produced the SCBA-2-400C. This

528 is because the PC-SCBA-2-80/20-400C presented fixed lime evolution in the same order

529 of magnitude as PC-SCBA-1-80/20 and PC-SCBA-3-80/20 pastes did during the curing

530 period 1–28 days.

531

532 *Compressive strength of pastes*

533

534 Fig. 13 – Compressive strength of pastes after 3, 7 and 28 days of curing at 20 °C

535

536 Fig. 14. Relationship between the mass loss from dehydration of the reaction products

537 (P_H) and the compressive strength of pastes

538

539 Compressive strength values of PC-CTRL, PC-SCBA-1-80/20, PC-SCBA-2-80/20 and

540 PC-SCBA-3-80/20 pastes cured for 3, 7, 28, and 180 days at 25 °C are illustrated in Fig.

541 13. For all mixes, the compressive strength values significantly ($p \ll 0.05$) increased with

542 the curing time as expected. With respect to the SCBA influence, after 3 days of curing

543 the SCBA-pastes presented no significantly different results (analysis of variance) from

544 each other and from the control, indicating that SCBA influenced positively on the

545 compressive strength since the early curing time. The dilution effect in SCBA-pastes was

546 compensated for the portlandite consumption from the SCBA to form additional hydrates.

547 This behavior was observed in the CH-SCBA and PC-SCBA blends studies in the early

548 curing times. At 7 days of curing, the same behavior was observed with no effect of SCBA

549 type on the paste compressive strength according to ANOVA. However, after 28 days of

550 curing, pastes with SCBA-2 and SCBA-3 presented significantly higher compressive

551   strength than that of mixes PC-SCBA-1-80/20 and PC-CTRL based on the Tukey post-

552   hoc tests. This behavior maintained for samples cured after 180 days, which PC-SCBA-

553   3-80/20 presented the highest compressive strength, followed by PC-SCBA-2-80/20. In

554   the case of PC-SCBA-1-20/80, it presented the lowest compressive strength among the

555   SCBA-containing pastes and no statistically significant difference was found between

556   this paste and PC-CTRL. The significant improvement on compressive strength by

557   employing SCBA-3 (PC-SCBA-3-80/20) respect to control (PC-CTRL) and SCBA-1

558   (PC-SCBA-1-80/20) were 17.9 and 14.1%, respectively. Fig. 14 shows that there was a

559   relationship between the P_H values and the paste compressive strengths. Therefore, these

560   compressive strength results were in accordance with the TGA data from PC-SCBA

561   pastes. In fact, PC-SCBA-2-80/20 and PC-SCBA-3-80/20 presented the highest values of

562   P_H at the same curing time. Compressive strength values showed that the additional

563   grinding processes of the SCBA-1 to produce SCBA-2 and SCBA-3 were slightly

564   beneficial after 28 days of curing on compressive strength of pastes. This effect was

565   previously observed for a high-quartz SCBA [7]. Comparing this present study to a

566   previous work with fly ash partially replacing the Portland cement by 15%, mortars

567   containing fly ash with particle size similar to that of SCBA-3 and SCBA-2 presented 10

568   and 5% higher compressive strength than a sample composed of a fly ash with particle

569   distribution similar to that of SCBA-1 after 28 days of curing [25,26]. It can be observed

570   that the compressive strength improvement among the ground fly ashes was similar to the

571   use observed in this study with SCBA. In the present study, as observed in the TGA

572   studies on CH-SCBA pastes, the SCBA-1 showed potential to react with the calcium

573   hydroxide in the same order of the magnitude as the SCBA-3 did, despite the lower value

574   of fixed lime (70.27 and 84.23%, respectively). This can justify the lowest difference in

575 the compressive strength behavior between the samples with SCBA-1 and SCBA-3 from

576 this study.

577

578 **CONCLUSIONS**

579 Based on the results of this experimental work, the following conclusions can be drawn:

580 • Two distinct additional grinding process performed on SCBA-1, which generated

581 SCBA-2 and SCBA-3, resulted in finest particles sizes. BET and particle size

582 characterization results showed that SCBA-3 presented the finest particle size,

583 followed by SCBA-2 and then SCBA-1.

584 • Electrical conductivity and TGA results of CH/SCBA blends showed that the

585 finest particle size resulted in highest activity. Electrical conductivity results

586 classified SCBA-1 and SCBA-2 as low reactivity, whereas SCBA-3 achieved

587 medium pozzolanic activity. In TGA studies, SCBA-3 presented the highest value

588 of fixed lime (84.23%), followed by SCBA-2 and SCBA-1 (72.67 and 70.27%,

589 respectively) after 7 days of curing at 20 °C.

590 • Isothermal calorimetric and TGA studies of CP/SCBA blends showed,

591 respectively, that there was a lower heat generation for SCBA-samples (except for

592 PC-SCBA-3-90/10), and there was detected some portlandite consumption by

593 SCBA due pozzolanic reaction, as showed the calculated fixed lime values.

594 • In the compressive strength tests, pastes cured after 180 days at 20 ºC showed that

595 PC-SCBA-3-80/20 samples presented the significantly highest compressive

596 strength, followed by PC-SCBA-2-80/20. PC-SCBA-1-80/20 and PC-CTRL

597 showed no statistically significant difference of compressive strength value,

598 which they were significantly lower than values for specimens produced with the

599 finest SCBA samples (SCBA-2 and SCBA-3).

600

601 <mark>Thus, these results allow concluding that the both different additional grinding processes</mark>

602 <mark>to generate ultrafine SCBA had positive effects on the pozzolanic reactivity and</mark>

603 <mark>mechanical behavior of cement-based pastes.</mark>

604

608

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