

23 Geopolymers are considered an eco-friendly construction material [1]. This material exhibits great
24 potential in several applications [2]: as binder agent in the stabilization/solidification of industrial
25 wastes [3-6], as fire resistant material in construction [7-10], as hydraulic road binder [11], as
26 acoustic concrete [12-13], among others [14]. The construction field is where geopolymers can
27 compete with cement products [15] due to a large number of useful/beneficial properties: high
28 compressive strength and structural integrity [16], resistance to acids and salts [16, 17], low
29 permeability [16], and medium-high density [18]. The challenge of the current work is to study and
30 evaluate pore generation in a fly ash based geopolymer in order to manufacture a foam product. A
31 foam product is considered a type of lightweight material that consists of a binder with a high
32 degree of void space [19]. There are two traditional methods of pore generation in a mortar [19]: by
33 endogenous gas generation, which can be achieved by mixing gas-releasing agents such as H_2O_2
34 [20], aluminium [21, 22] or silica [23, 24], or by introducing a very large volume fraction of air
35 bubbles, normally using an organic foaming agent (surfactants) [20, 25, 26]. Silicon, in the form of
36 silica fume, was used to generate pores in this work. In general, a reactive metal powder (Si) reacts
37 with water and hydroxide in an alkaline environment producing bubbles of hydrogen gas [19].
38 These bubbles attempt to escape into the air from the geopolymer paste producing the expansion of
39 the geopolymer mortar [27] and generating the porous structure. Silica fume is defined as an
40 inorganic by-product from the metallurgical industry [28]. Several studies have been carried out in
41 which silica fume is added to a metakaolin based geopolymer [24, 29, 30] in order to generate a
42 foam. Prud'homme et al., [23, 24] studied the effect of silica fume content and temperature in the
43 microstructure and porosity of geopolymer foam. Henon [30] concluded that the synthesis of the
44 product and porosity depend to a great extent on the temperature (range 25-70 °C). In his study,
45 geopolymer foams with macroporosities in the range 0.5-1.6 mm were developed. Thermal
46 conductivities of 0.12-0.33 W/(mK) for porosities of 65-85 % were obtained. Papa et al., [29]

47 obtained macroporous structures with roughly rounded pores and a total porosity of about 80%,
 48 thermal conductivities of around 0.16 W/(mK) and acoustic behaviour.
 49 In the current work, fly ash based geopolymers have been prepared adding silica fume in different
 50 proportions in order to evaluate the physical, mechanical and acoustical properties of the final
 51 geopolymeric foam. Two activating solutions and setting temperatures have also been evaluated. In
 52 summary, a dual environmental design has been reached: to recycle industrial by-products produced
 53 in large amounts and to develop an acoustic absorbing material in order to reduce noise pollution
 54 levels.

55

56 2. Materials and methods

57 2.1 Materials

58 A low calcium fly ash (FA) (ASTM class F [31]) from a coal combustion power plant (Los Barrios,
 59 Spain) was used as the geopolymeric precursor. The silica fume (SF) used in this work was supplied
 60 by the Sika© company. Chemical composition of the fly ash and the silica fume (Table 1) was
 61 determined after chemical attack and dissolution at 750 °C (ASTM D-3682-78) [32] using atomic
 62 absorption spectroscopy. The specific gravity, in accordance with EN 1097-7 [33], was also
 63 determined in the fly ash and silica fume (Table 1).

64 **Table 1. Characteristics of fly ash and silica fume**

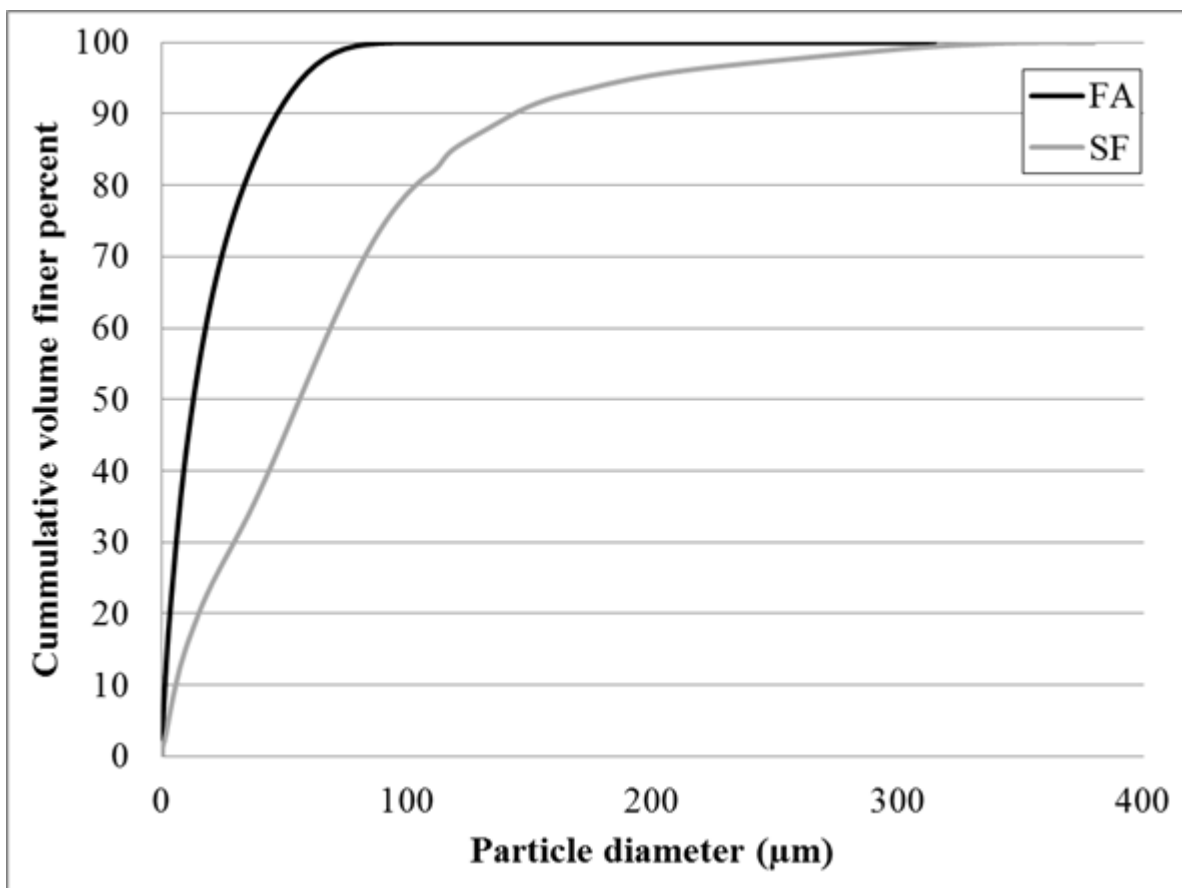
	Moisture (%)	LOI 750°C	Chemical composition (wt %)							Specific gravity (g/cm ³)
			Fe ₂ O ₃	CaO	MgO	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	
FA	0,05	3,32	5,86	3,94	1,84	63,9	21,5	0,68	1,67	1,93
SF	0.03	1,48	0,5	0,51	0,39	96,7	0,43	0,2	0,79	2,04

65

66 High SiO₂ and Al₂O₃ contents were observed in FA. The silica fume was practically SiO₂, and its
 67 specific gravity (2.04 g/cm³) was similar to the fly ash (1.93 g/cm³). Amorphous contents of both

68 materials were determined after the X-ray powder diffraction (XRD) analysis using DIFFRAC.EVA
69 software. Amorphous content of FA and SF were 44.1 % and 67.1 %, respectively.
70 The particle size distribution of both materials was determined using a laser diffraction
71 granulometer provided by Micromeritics, which is equipped with an optical system and data
72 acquisition software. The results are shown in Figure 1.

73 **Figure 1. Particle size distribution of fly ash and silica fume**



74

75 As can be seen in Figure 1, FA presented the finest particle size distribution, with 88 % of particles
76 less than 45 µm. SF showed a coarser distribution than FA, with 80 % of particles lower than 100
77 µm.

78 Two activating solutions were used in this work. The first solution was a mixture of potassium
 79 silicate solution (SiO_2 23 w/w % and K_2O 14.9 w/w %) (Industrias Químicas del Ebro, Spain) and
 80 sufficient potassium hydroxide to increase the $\text{K}_2\text{O}/\text{SiO}_2$ molar ratio up to 1.45 (pH = 13.5). The
 81 second solution was KOH 8M (pH = 14).

82 2.2 Geopolymeric foam preparation

83 Geopolymeric foams were manufactured in a laboratory mixer working at 500 rpm. Fly ash was
 84 mixed with an activating solution until a thixotropic paste was obtained (mixing time was 4
 85 minutes). After that, silica fume was added to the geopolymer paste. A reduction in thixotropy and
 86 workability of the geopolymers was observed as SF was mixed with the geopolymer paste, so extra
 87 water was necessary to improve both characteristics. The pastes obtained were poured into
 88 cylindrical plastic moulds (30 mm-diameter and 40 mm-height). Two setting temperatures were
 89 studied: 40 °C and 70 °C (in the first 24 hours). After setting, samples were demoulded and cured at
 90 room temperature (average temperature: 20 °C; average relative humidity: 45 %) for a further 27
 91 days. Compositions of geopolymeric foams are detailed in Table 2. The samples were cut after 28
 92 days in order to obtain the same sample thickness (40 mm) in the different tests.

93 **Table 2. Composition of geopolymeric foam**

Geopolymer foam	Solid phase (wt%)		Activating solution (g)/Fly ash (g)	Water (g)/Solid phase (g)	Setting temperature (°C)
	FA	SF			
FA100SF0-KSi1-40°C	100	0	0,74	0	40
FA80SF20-KSi1-40°C	80	20	0,74	0,1	40
FA60SF40-KSi1-40°C	60	40	0,74	0,225	40
FA80SF20-KSi1-70°C	80	20	0,74	10	70
FA80SF20-KOH-40°C	80	20	0,71	0	40

95 As can be seen in Table 2, three parameters have been studied: FA-SF proportion (100-0, 80-20, 60-
96 40), activating solution (potassium silicate (KSil) vs potassium hydroxide (KOH)), and setting
97 temperature (40 vs 70 °C). As the silica fume content increased in the mixture, the water/solid ratio
98 also increased. This phenomenon suggests that SF may demonstrate inert material behaviour in the
99 geopolymerization reaction. As the addition of SF produced a foam material, final products
100 obtained from samples FA80SF20 and FA60SF40 were also named geopolymeric foams. However,
101 the final product of the sample FA100SF0 was a geopolymeric paste.

102 **2.3. Methods**

103 **2.3.1. Volume expansion and setting time**

104 The geopolymerization reaction kinetic was studied analysing the setting time. The setting time was
105 determined using a Vicat needle (EN 196-3) [34]. The initial setting time is the time elapsed
106 between the initial instant and the time in which the distance from the needle to the bottom base of
107 the cylinder is 6 ± 3 mm. The final setting time is the time between the initial instant and the time in
108 which the needle shows no visible signal on the upper base of the cylinder. The pore generation
109 reaction kinetic was determined by measuring the expansion volume (EV) of material during setting
110 (mm). EV is the ratio between the foam volume at instant t , $V(t)$, and the initial volume of
111 introduced paste, $V(0)$. For this test, geopolymer samples were moulded in 35 mm-diameter and 80
112 mm-high cylindrical plastic molds no more than half-full. Three specimens of each geopolymer
113 mixtures were used to determine the volume expansion and setting time.

114 **2.3.2. XRD analysis**

115 Geopolymer samples were studied by means of X-ray powder diffraction (XRD) to evaluate their
116 mineralogical composition. A D8 Advance A25 (BRUKER) (40 kV and 30 mA) instrument was
117 used and phase identification was carried out using DIFFRAC.EVA software (BRUKER).

118 **2.3.3. Physical and mechanical properties**

119 Density (ρ) of the geopolymers was measured in accordance with the EN 1936 standard method
120 [35]. Open void ratio (open porosity) was also determined because it is strongly related to the
121 acoustic behaviour of the products. The method of vacuum water saturation was followed in the
122 determination of open porosity (OP %) [35].

123 Compressive strength of geopolymer samples was determined in accordance with EN 196-1 [36],
124 using a Tinius Olsen-TO317EDG machine. Four specimens of each geopolymer (paste or foam)
125 were used to determine the compressive strength, density and open porosity.

126 **2.3.4. Acoustic properties**

127 In order to evaluate the acoustic properties of the geopolymeric samples, sound absorption was
128 determined using a Kundt tube, according to EN ISO 10534-2 [37]. An ACUPRO system,
129 implemented by “Spectronics Inc.”, with two microphones and a SAMSON signal amplifier, was
130 used. Three specimens of each geopolymer have been tested. The thickness of all the tested
131 specimens was 40 mm. The Noise Reduction Coefficient (NRC) was determined by calculating the
132 arithmetic mean of the absorption coefficients at 250 Hz, 500 Hz, 1000 Hz and 2000 Hz.

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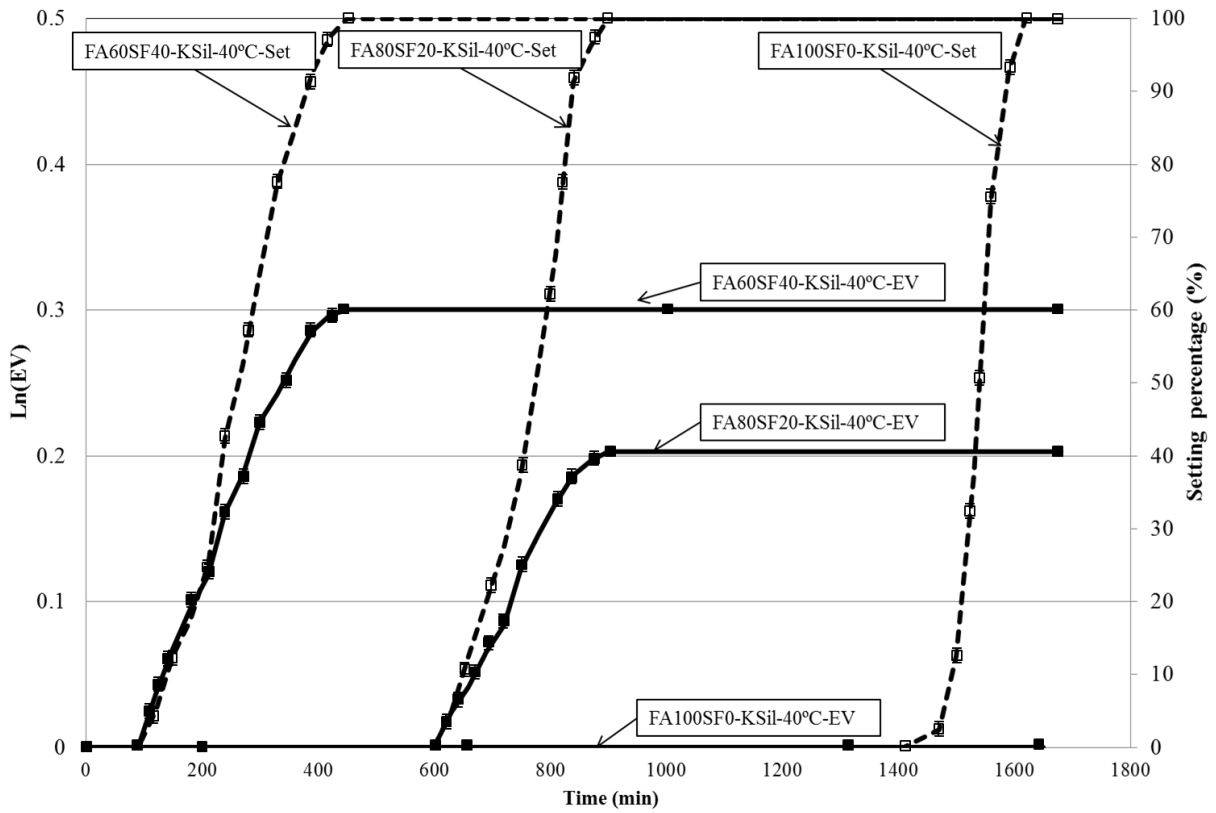
134 **3. RESULTS**

135 **3.1. Expansion volume and setting evolution with time**

136 Figures 2, 3 and 4 show the setting curves (penetration of Vicat needle (mm) in the cylinder versus
137 time) (Set) and the expansion volume curves (expansion volume data (natural logarithm) (EV)
138 versus time), taking into account the three parameters studied: FA-SF proportion (100-0, 80-20, 60-
139 40) in Figure 2, activating solution (potassium silicate vs potassium hydroxide) in Figure 3 and
140 setting temperature (40 vs 70 °C) in Figure 4. Standard deviations are also represented as error bars.

141 **Figure 2. Expansion volume and setting curves for different FA-SF proportions**

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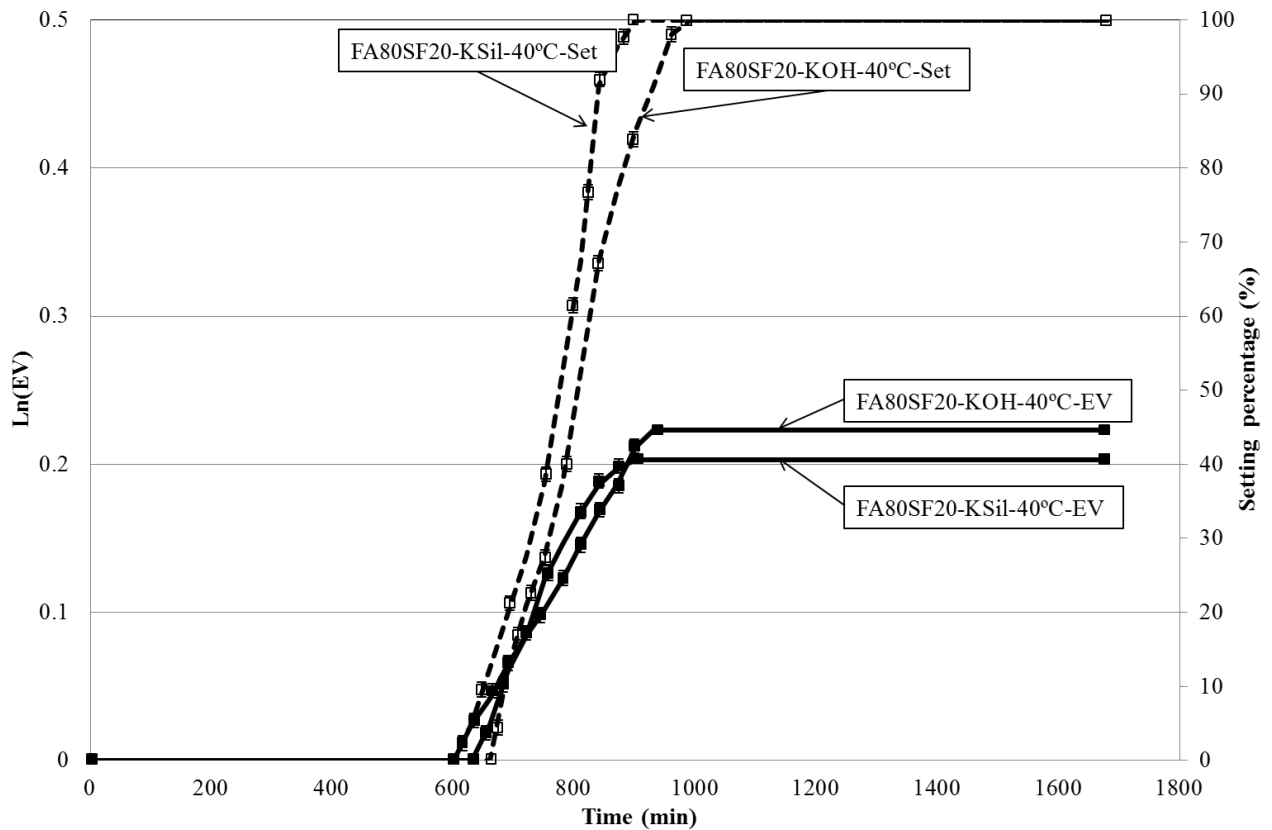


143

144

145 Figure 2 shows that the substitution of FA with SF produced a reduction in the initial setting time
 146 (from 1410 min for geopolymer paste without silica fume (FA100SF0) to 120 min for geopolymer
 147 foam with 40 %wt of silica fume (FA60SF40)) and final setting time (from 1620 min without silica
 148 fume to 450 min with 40 % wt of silica fume). Figure 2 also shows an increment in material
 149 expansion volume as silica fume was added to the mixture due to generation of gas produced by the
 150 use of SF (as SF content was null, EV was null).

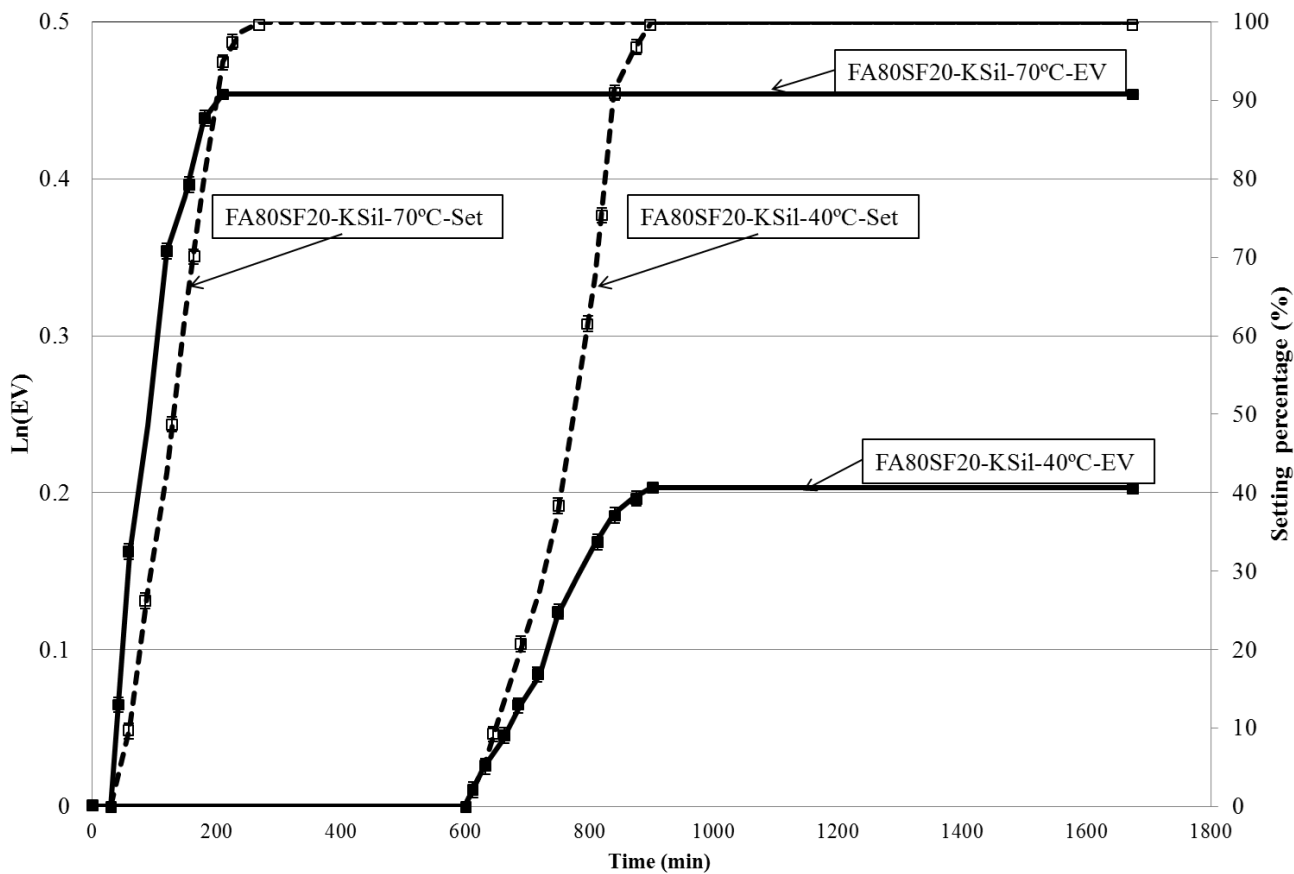
151 **Figure 3. Volume expansion and setting curves for different activating solutions (potassium**
 152 **silicate vs potassium hydroxide)**



153

154 As can be seen in Figure 3, the change of activating solution did not bring about important
 155 variations in setting time and material expansion volume results. A slight reduction in the setting
 156 time in geopolymer foam activated with potassium silicate in comparison with the activation with
 157 potassium hydroxide was observed. A slight increment in expansion volume as geopolymer foam
 158 was activated with potassium hydroxide can also be observed.

159 **Figure 4. Volume expansion and setting curves for different setting temperatures (40 vs 70 °C)**



160

161 Figure 4 shows a reduction in the setting time as the geopolymer was set at 70 °C (final setting time
 162 was 240 min) instead of 40 °C (final setting time was 870 min). An increase in material expansion
 163 with the temperature (more than 100 %) was also observed.

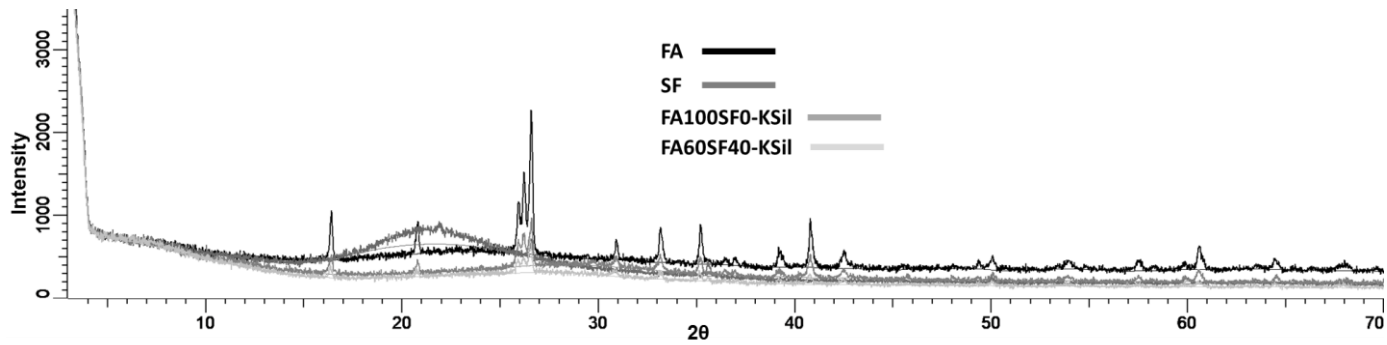
164 **3.2. XRD analysis**

165 XRD patterns of FA, SF, geopolymer FA100SF0-KSiI and geopolymer foam FA60SF40-KSiI set at
 166 40 °C are detailed in Figure 5.

167 **Figure 5. XRD patterns of FA, SF, geopolymer FA100SF0-KSiI and geopolymer foam**
 168 **FA60SF40-KSiI set at 40 °C**

169

170



171

172 SF pattern was characteristic of an amorphous material, with a broad peak in the 2θ range 15-30.

173 FA showed a slight broad peak between 15-38 values of 2θ , so FA could be considered a type of

174 amorphous degree material. Some crystalline peaks were also detected in FA, mainly quartz and

175 mullite. The geopolymer FA100SF0-KSiI and the geopolymer foam FA60SF40-KSiI exhibited very

176 similar patterns analogous to the the FA pattern. They showed a slight shift of the broad reflection

177 regarding the FA pattern, with lower intensity peaks of quartz and mullite than the FA.

178

179 3.3. Physical and mechanical properties

180 3.3.1. Open porosity and apparent density

181 Open porosity and density of geopolymer samples at 28 days of curing are detailed in Table 3.

182 Standard deviations are also specified for open porosities and densities.

183 **Table 3. Open porosity and density**

Geopolymer foam	Open porosity (%)	Density (g/cm ³)
FA100SF0-KSiI-40°C	11.5±1.2	1.4±0.1
FA80SF20-KSiI-40°C	18.1±1.8	1.17±0.1
FA60SF40-KSiI-40°C	28.9±2.5	1.07±0.1
FA80SF20-KSiI-70°C	36.2±3.2	0.97±0.1
FA80SF20-KOH-40°C	20.1±2.1	1.12±0.1

184

185 Open porosity increased and density decreased as silica fume proportion in the mixture was

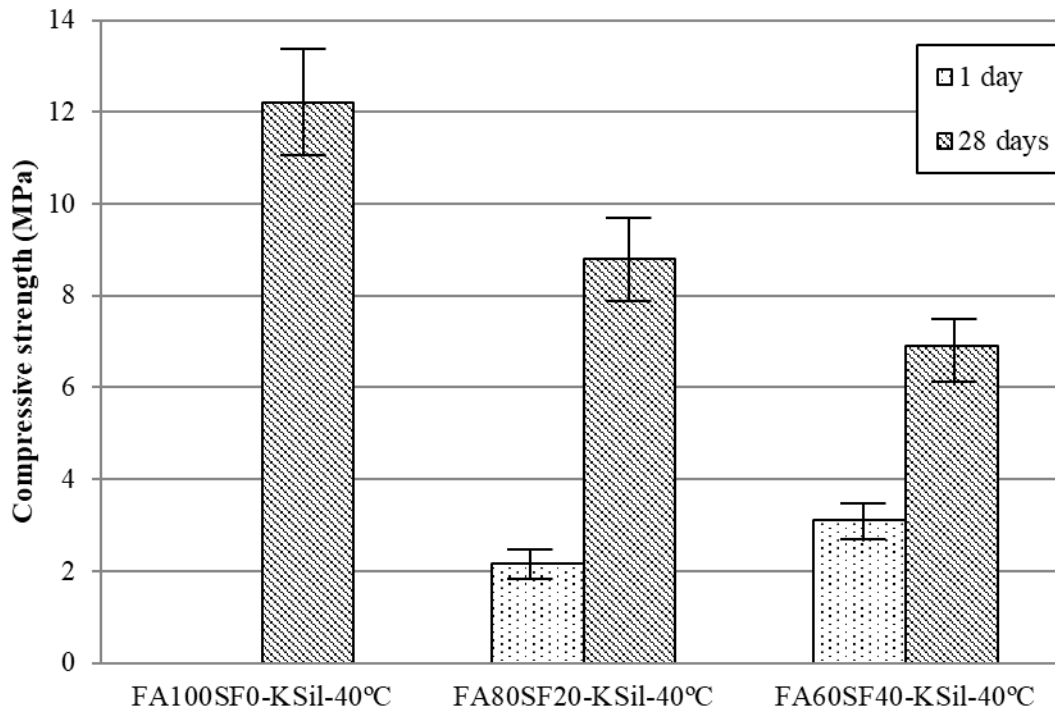
186 increased and as setting temperature was changed from 40 °C (open porosity of 18.1%) to 70 °C

187 (open porosity of 36.2%). Open porosities displayed a slight variation as the activating solution was
188 modified. The geopolymer foam activated with potassium silicate showed a porosity value of
189 18.1%. The value was 20.1% as the geopolymer foam was prepared with potassium hydroxide.

190 3.3.2. Compressive strength

191 Figures 6 shows the effect of FA-SF proportion on the compressive strength values determined at 1
192 day and 28 days of curing. Standard deviations are represented as error bars.

193 **Figure 6. Compressive strength. FA-SF proportion**

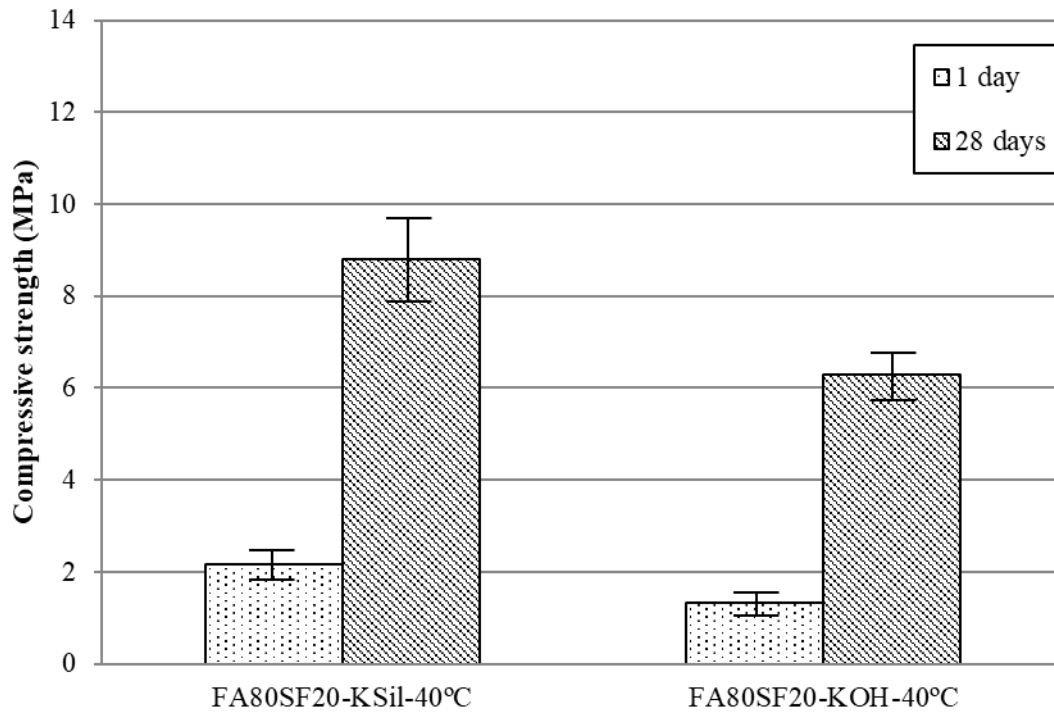


194

195 Two different behaviors depending on the curing time have been observed: a positive effect of silica
196 fume in compressive strength at early stages (1 day) of curing and a negative effect in the final
197 stages (28 days). As can be seen, there was no compressive strength data of the geopolymer paste
198 (FA100SF0-KSil) because this material did not set at 1 day.

199 Figure 7 represents the compressive strength results at 1 day and 28 days of curing for geopolymer
200 foams activated with silicate and hydroxide. Standard deviations are detailed as error bars.

201 **Figure 7. Compressive strength. Activating solution (potassium silicate vs potassium**
202 **hydroxide)**



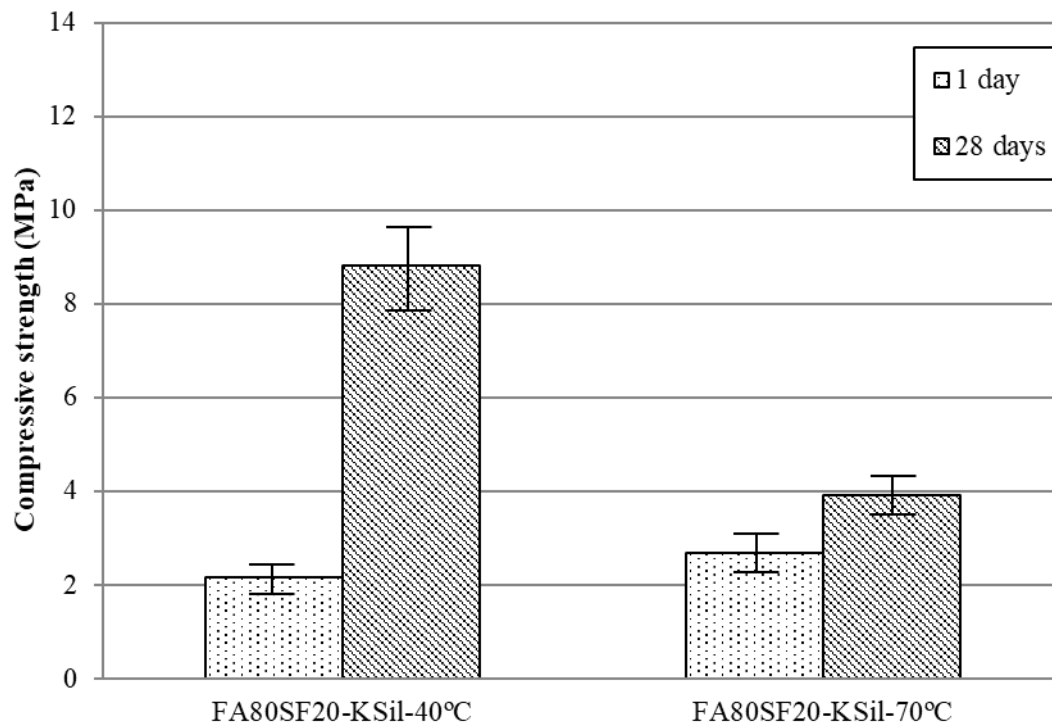
203

204

205 The setting time was slightly higher in geopolymer activated with potassium hydroxide than with
206 potassium silicate, so the compressive strength at 1 day of hydroxide activated geopolymer foam
207 was slightly lower than silicate activated geopolymer foam. The same tendency was observed at 28
208 days of curing; geopolymer foam activated with potassium silicate was stronger than that activated
209 with potassium hydroxide.

210 The effect of setting temperature on compressive strength of the geopolymer foams is displayed in
211 Figure 8. Standard deviations are specified as error bars.

212 **Figure 8. Compressive strength. Setting temperature (40 vs 70 °C)**



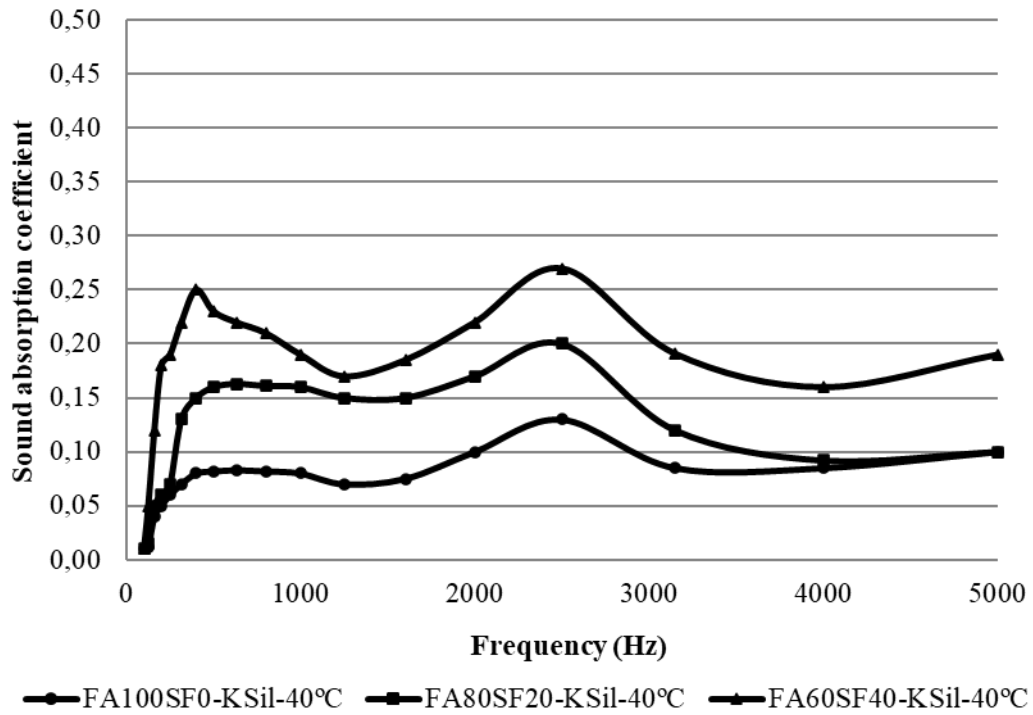
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214 As can be seen, the setting at 70 °C had a positive effect on the compressive strength at early stages
 215 but the effect is negative at 28 days of curing.

216 3.4. Acoustic properties

217 Figures 9, 10 and 11 show the sound absorption coefficient curves, taking into account the three
 218 parameters studied. The sound absorption coefficient is represented in 1/3 octave band of the
 219 frequencies between 100-5000 Hz. Standard deviations are represented as error bars.

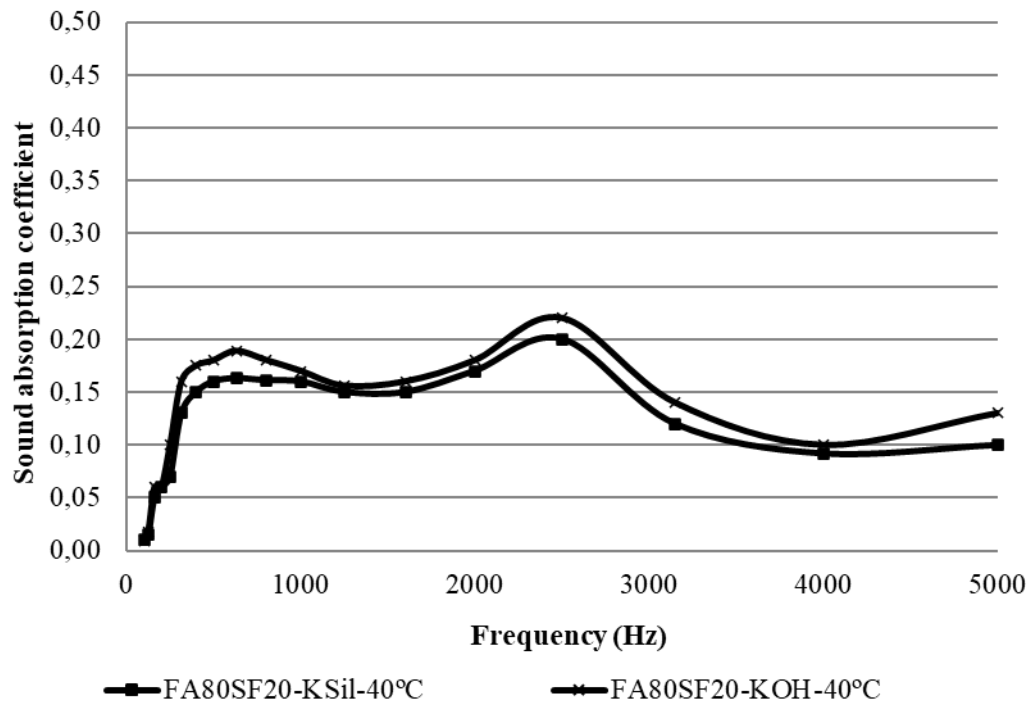
220 **Figure 9. Sound absorption coefficient. FA-SF proportion**



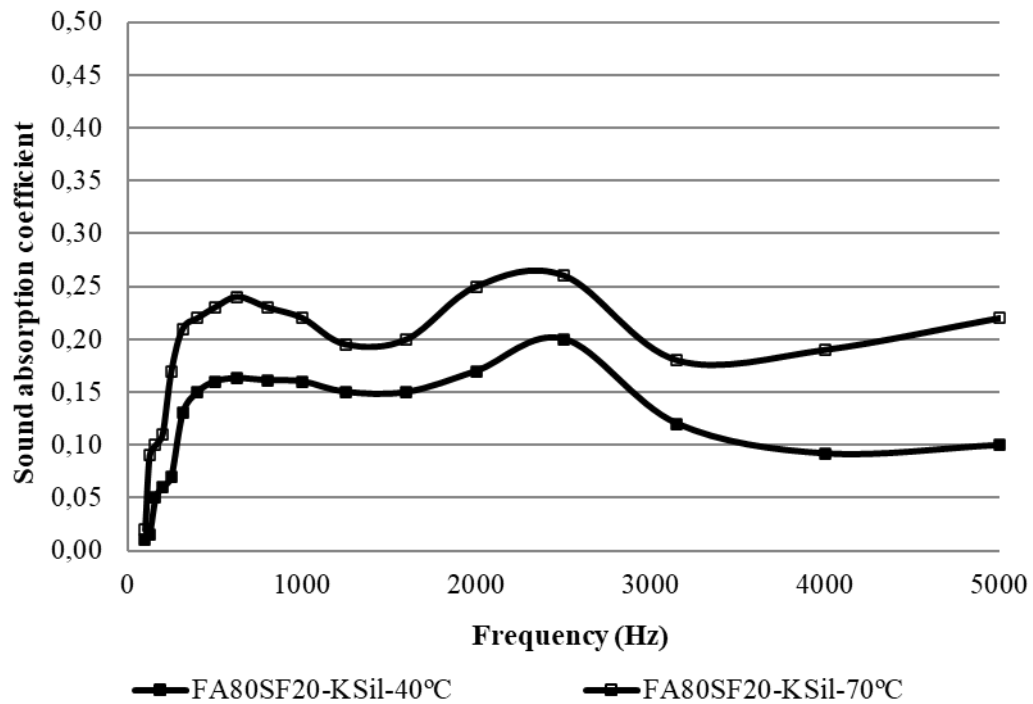
221

222 Figure 9 shows that the three curves had similar shapes (with two peaks at 400 and 2500 Hz,
 223 respectively). As can be seen, the greater the silica fume proportions in the mixture, the higher the
 224 breadth curve, which could be correlated with the highest open porosity.

225 **Figure 10. Sound absorption coefficient. Activating solution (potassium silicate vs potassium**
 226 **hydroxide)**



227 As can
 228 be seen in Figure 10, both curves were extremely similar (the curve of geopolymer foam activated
 229 with hydroxide was marginally above the curve of geopolymer foam activated with silicate). Both
 230 geopolymer foams had a similar open porosity (slightly higher open porosity was observed in the
 231 geopolymer foam activated with hydroxide), which may explain the similarity between the curves.
 232 **Figure 11. Sound absorption coefficient. Setting temperature (40 vs 70 °C)**



233

234 Figure 11 shows that both curves had a similar shape. Two peaks at 400 and 2500 Hz were also
 235 observed. The highest values of the sound absorption coefficient were observed in the geopolymer
 236 foam set at 70 °C since this material presented the greatest open porosity.

237 Noise Reduction Coefficient (NRC) for all geopolymers is detailed in Table 4. This coefficient is a
 238 numerical descriptor of the sound absorption, which is very useful to make comparisons. Standard
 239 deviations are also detailed. Analysing Table 4 and Table 3 we can see that the NRC increased as
 240 porosity increased.

241 **Table 4. NRC of geopolymer foams**

Geopolymer foam	NRC
FA100SF0-KSiI-40°C	0.08±0.01
FA80SF20-KSiI-40°C	0.14±0.01
FA60SF40-KSiI-40°C	0.21±0.02
FA80SF20-KSiI-70°C	0.23±0.02
FA80SF20-KOH-40°C	0.16±0.01

242

243 **4. DISCUSSION**

244 4.1. Volume expansion and setting time

245 Two reactions take place during the geopolymer foam manufacture: the geopolymerization reaction
246 and the pore generation reaction. The geopolymerization reaction occurs when the fly ash reacts
247 with the activating solution, producing an alkali aluminosilicate hydrated gel. The gel obtained in
248 the current work was mainly based on potassium (potassium aluminate silicate hydrated gel) since
249 the activating solution contains potassium as alkali cation [38]. The use of different types of anions
250 (silicate or hydroxides) also affects the final chemical composition of the gel (Si/Al ratios are higher
251 in gels after activation with silicate than with hydroxide) [38, 39]. The pore generation is due to the
252 H₂(g) released during the water reduction and silicon (from silica fume) oxidation, producing
253 Si(OH)₄. The reaction is promoted in basic medium [24] according to:



255 As can be seen in Figures 2, 3 and 4, setting curves exhibited three clearly differentiated stages. The
256 first stage corresponded to the plastic period of the geopolymer, which goes from the time in which
257 needle penetrates completely to the start of setting. The second stage matched with the setting
258 period of the geopolymer, which is between initial and final setting time (needle shows resistance to
259 penetration). The final stage constituted the hardening period.

260 Expansion volume curves also showed three stages. The first stage began with a period of latency
261 during which the expansion volume was null, named latency time [30]. The second stage of the
262 curve exhibited an increase of expansion volume (EV) with the time caused by the generation of
263 gas, which follows a linear regime [30]. The last showed a final expansion volume constant through
264 time.

265 The replacement of fly ash with silica fume produced a reduction in the initial and final setting time
266 of the geopolymer, that is, the geopolymer paste FA100SF0 set more slowly than the geopolymer
267 foam FA80SF20 and the geopolymer foam FA80SF20 more slowly than FA60SF40. This behavior

268 is similar in silica fume blended cement pastes [40] or in silica fume blended metakaolin
269 geopolymers [24] where an increment in silica fume accelerated the setting and hardening process.
270 An increment in material expansion volume as silica fume was added to the mixture was also
271 observed. This is due to the generation of gas produced by the use of SF (according to equation 1).
272 In addition, the latency period matched the plastic period and the setting time coincided with the
273 period in which gas was generating. As gas is generated, the hydrogen bubbles try to escape from
274 the material. When the geopolymer is still fluid in the plastic period (setting has not yet taken
275 place), the gas bubbles generated can leave the geopolymer. However, while the geopolymer is
276 setting and the material is hardening, the gas becomes trapped in the structure, expanding the
277 geopolymeric matrix and increasing the expansion volume.

278 The type of activating solution did not significantly affect the setting time or the expansion volume.
279 A slight reduction in the setting time of geopolymer foam activated with potassium silicate
280 compared with that activated with potassium hydroxide was observed. Some authors [39] noted that
281 geopolymers activated with soluble silicate showed higher reaction rates than those activated with
282 hydroxide. Furthermore, previous work of the authors [41], where the activation with hydroxide
283 versus silicate was studied, concluded that systems activated with hydroxide (KOH 8M) presented
284 (fewer) lower degrees of reaction, at the same time, than systems activated with silicates (potassium
285 silicate), which could provide evidence of the high rate of reaction with silicates. Furthermore, pH
286 of the activating solution must be also considered. Some authors [42] revealed that high
287 concentration solutions presented a coagulation of silica, reducing the dissolution of Si^{4+} and Al^{3+}
288 from the raw material. In the current work, pH of potassium hydroxide solution was >14 and pH of
289 potassium silicate solution was 13.5. Therefore, the increase in setting time using KOH 8M could
290 be the result of some silica coagulation, which may result in some setting delay.

291 Material expansion volume was slightly higher in geopolymer activated with potassium hydroxide,
292 possibly due to the different solution pH (pH >14 in the potassium hydroxide solution and pH = 13.5

293 in the potassium silicate solution). As previously mentioned, the pore generation reaction occurs in
294 basic medium, so the addition of a more basic solution could have a positive effect on this reaction.
295 The effect of temperature is also displayed, showing a reduction in setting time and an increment in
296 material expansion as geopolymer foam samples set at 70 °C instead of 40 °C. On one hand, it is
297 well known that temperature is a geopolymerization reaction accelerator [39]. On the other hand,
298 the reaction of equation 1 is enhanced by the temperature [43]. Henon et al. [30] have proposed that
299 the final expansion of metakaolin based geopolymer with silica fume followed an Arrhenius law
300 with the temperature, obtaining a value of activation energy of 11.9 kJ/mol. The activating energy
301 of geopolymer foam FA80SF20-KSil in the current work was also calculated according to the
302 methodology of Henon's work and the value was 27.9 kJ/mol. This value was similar to that
303 obtained in a study carried out by Provis [44] (33 kJ/mol) where metakaolin was used as
304 geopolymeric raw material.

305 4.2. XRD analysis

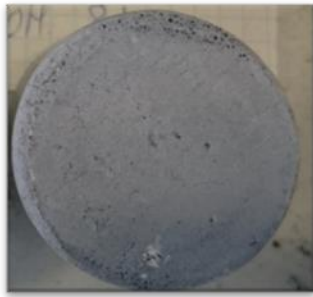
306 XRD results show the changes which took place in the SF and the FA after the geopolymerization
307 reaction. As previously mentioned, FA displayed a slight broad reflection in the 2θ range 15-38
308 with crystalline peaks of quartz and mullite. The geopolymer paste FA100SF0-KSil showed a shift
309 of the broad peak in the 2θ range 22-38 regarding the FA pattern, which provides evidence of the
310 dissolution of SiO_4 and AlO_4 species during the geopolymerization reaction [24]. In addition, the
311 crystalline peaks in the geopolymer (quartz and mullite) were in the same 2θ position as the FA
312 pattern but with lower intensity, which was indicative of alteration of the crystal structure [23]. SF
313 pattern was characteristic of an amorphous material, with a broad peak, which was not found in the
314 geopolymer foam FA60SF40-KSil pattern. The geopolymer foam pattern was rather similar to the
315 geopolymer paste and the FA patterns, with a slight shift of the broad peak in the 2θ position and
316 with the crystalline peaks (quartz and mullite) in the same place but with lower intensity.
317 Comparing the XRD patterns of the geopolymer paste and the geopolymer foam we can see that the

318 silica fume addition did not produce significant differences in amorphous and crystalline phases,
319 except a slight difference in the intensity of the peaks. Amorphous content of both materials were
320 calculated using DIFRACT.EVA software to support these results. FA100SF0 and FA60SF40
321 showed values of 51.6 % and 53.8 %, respectively.

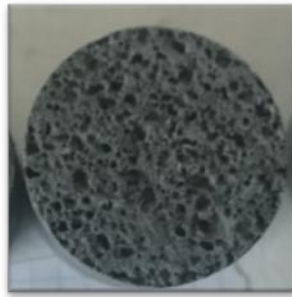
322 4.3. Physical and mechanical properties

323 Table 3 and Figures 2, 3 and 4 provide evidence of the direct relationship between open porosity
324 and expansion volume of the material. The material expansion test (Figure 2) shows higher foam
325 development of the geopolymer as the proportion of silica fume increased, which allows us to view
326 the positive effect of silica fume proportion in pore generation, and consequently, in the open
327 porosity. The same behavior was displayed as the activating solution effect was analyzed.
328 Geopolymer foam activated with hydroxide (Figure 3) showed a slightly higher expansion volume
329 than that activated with potassium silicate, so the amount of generated pore was slightly greater, as
330 was the open porosity. The values of open porosity depend greatly on the setting temperature,
331 producing a high increment in open porosity as the temperature increased from 40 to 70 °C due to
332 the increment in the expansion volume (Figure 4). Photographs of different geopolymer foams are
333 detailed in Figure 12. Analyzing the pore size in the right photograph, it can be observed that pore
334 sizes were in the range 1-3 mm, which are the most appropriate pore sizes for noise absorbing
335 materials [45].

336 **Figure 12. View of the some of the geopolymer foam (A. FA100SF0-40°C; B. FA60SF40-40°C;**
337 **C. FA80SF20-40°C; D. FA80SF20-70°C) after 28 days of curing**



A. FA100SF0-40 °C



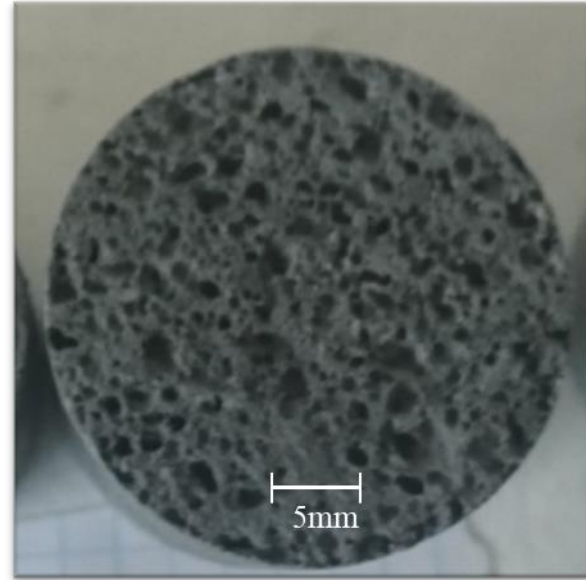
B. FA60SF40-40 °C



C. FA80SF20-40 °C



D. FA80SF20-70 °C



B. FA60SF40-40 °C

338

339 The effect of silica fume content, activating solution and setting temperature on compressive
340 strength depend on the curing time in which the test was carried out: early stages (1 day) or final
341 stages (28 days). It seems that compressive strength at early stages was controlled by the setting
342 rate. Therefore, variables that produce quick setting times such as using a high content of silica
343 fume (up to 40%), to set at 70 °C and to activate with potassium silicate, can improve the
344 compressive strength at 1 day of curing. However, the compressive strength at final stages was
345 controlled by the open porosity values. The parameters that reduced the open porosity (no use of
346 silica fume, to set at 40 °C and to activate with potassium silicate) also produced an enhancement of
347 the compressive strength at 28 days of curing. It is well known that an increase in voids or pores in
348 any material (while the rest of the parameters remain unchanged) always causes a reduction in the
349 compressive strength since the volume/resistant mass ratio is lower [46], so the poorest mechanical
350 properties were observed in the most porous geopolymer foams.

351 4.4. Acoustic properties

352 Taking into account that sound absorption in a porous material is related to the energy loss through
353 friction with the wall of the holes, higher porous structures showed higher sound absorbing
354 properties [12]. Therefore, to increase the silica fume proportion and to increase the setting
355 temperature from 40 to 70 °C produced a positive effect on sound absorption. In addition, these
356 geopolymer foams exhibited sound absorption coefficients similar to or better than commercial
357 porous concrete [46] and other sound absorption materials containing wastes, such as bottom ashes
358 [46] or ceramic wastes [47].

359

360 **5. CONCLUSIONS**

361 The main aim of this experimental study was to develop geopolymeric foams using fly ash and
362 silica fume. The foam products presented porous material characteristics with relatively good noise
363 absorption properties. Three parameters have been studied: silica fume content, activating solution
364 and setting temperature. Five properties have been evaluated: setting time, expansion volume, open
365 porosity, compressive strength (at early and final stages) and sound absorption, all of which are
366 closely linked.

367 The substitution of fly ash with silica fume and the set at 70 °C reduced the initial and final setting
368 time and increased material expansion. The first observed effect was an increment in the open
369 porosity of the geopolymer foam. Taking into account that the sound absorption in a porous
370 material was related to the energy loss through friction with the walls of the holes, less porous
371 structures showed lower sound absorbing properties. Therefore, geopolymer foam prepared with the
372 highest amount of silica fume and set at 70 °C showed the greatest noise reduction coefficient. The
373 effect of the three parameters on compressive strength depends on the curing time. The behaviour of
374 the materials at early and final stages of curing is not the same since the compressive strength
375 values at early stages (1 day) were controlled by the setting rate. However, the compressive strength
376 values at 28 days were directly related to the open porosity values.

377 It seems that to replace one activating solution for another did not produce significant differences. A
378 slight reduction in setting time using silicate, and a slight increment in open porosity (expansion
379 volume) using hydroxide was observed, possibly due to the variations in the pH values of solutions
380 and the different degrees of reaction reached by the different material products obtained.

381

382 **Compliance with Ethical Standards:**

383 Authors declare that they have no conflicts of interest.

384

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388

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