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1 **Aggregate size effect on the development of cementitious compounds in a**
2 **lime-treated soil during curing**

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25 ***Abstract***

26 This work aims to investigate the aggregate size effect on changes in mineralogical
27 composition and microstructure of lime-treated compacted soils. Three soil powders with
28 different maximum aggregate sizes ($D_{max} = 5, 1$ and 0.4 mm) were prepared prior to the
29 treatment with 2% of lime. X-ray diffraction (XRD), environmental scanning electron
30 microscope (Env. SEM) coupled with chemical analysis using energy dispersive X-ray
31 spectrometry (EDX) and mercury intrusion porosimetry (MIP) were used to analyse untreated
32 and treated samples at various curing times. Crystallized C-S-H on tobermorite form was
33 identified in the lime-treated soil prepared with large aggregates after one year curing, and an
34 evident increase in nanopores less than $0.1 \mu\text{m}$ C-S-H was also observed due to C-S-H
35 creation. By contrast, in the case of smaller aggregates, no obvious C-S-H peaks were
36 observed by XRD technique after the same curing time, even though some evidence of such
37 phases are provided by Env. SEM coupled to EDX analysis. But a large amount of
38 undetectable nanopores less than 6 nm (considering the MIP technical limitation) was
39 supposed to be formed and could be attributed to the creation of nanocrystallized C-S-H or
40 poorly-crystallized C-S-H (that may fill the pores larger than $2 \mu\text{m}$). Such type of C-S-H
41 phases occurred when lime was coated in thin layer on the large surface associated to
42 lime-treated soil prepared with small aggregates.

43

44 *Keywords:* lime-treated soil; aggregate size; curing time; mineral composition; microstructure

45

46 **1. Introduction**

47 Lime treatment is an effective soil improvement technique widely applied in the field of
48 construction. It largely modifies the soil geotechnical properties through the physico-chemical
49 reactions within the lime-soil-water system (Boardman et al., 2001; Russo, 2005; Al-Mukhtar
50 et al., 2010; Tang et al., 2011; Tran et al., 2014). When quicklime (CaO), soil and water are
51 mixed together, hydration and ionization of quicklime immediately take place. Then, the Ca²⁺
52 ions in the pore water released by calcium hydroxide (Ca(OH)₂) is adsorbed by ion-exchange
53 at clay minerals surface. The diffuse hydrous double layer surrounding the clay particles can
54 be modified by the Ca²⁺ ion-exchange process, resulting in the flocculation-agglomeration of
55 clay particles (Bell, 1996). These modifications of clay particles induced by lime addition will
56 largely improve the workability of soil by reducing the plasticity, the swelling and shrinkage
57 (Bell, 1989; Russo, 2005). In the long-term, the main reactions between lime and clay
58 minerals are of pozzolanic nature which contributes significantly to the improvement of soil
59 mechanical behaviour in terms of shear strength, shear modulus, compression strength and
60 compressibility (Rajasekaran and Narasimha Rao, 2002; Khattab et al., 2007; Consoli et al.,
61 2009; Tang et al., 2011; Dong, 2013). The mechanical improvement is to be attributed to the
62 creation of cementitious compounds generated by the pozzolanic reaction, coating the soil
63 particles and bonding them together (Bell, 1996; Onitsuka et al., 2001; Nalbantoglu, 2006).

64 Mineralogical studies of cementitious compounds have been undertaken in recent years. The
65 cementitious compounds can be of various forms due to the different mineralogical
66 composition of soils containing mainly clay minerals such as kaolinite, montmorillonite or
67 illite, and other minerals like quartz and feldspars. Generally, the main cementitious

68 compounds are calcium silicate hydrate (C-S-H), calcium aluminate hydrate (C-A-H) and
69 calcium alumino-silicate hydrate (C-A-S-H) (Khattab, 2002; Rios et al., 2009; Maubec, 2010;
70 Al-Mukhtar et al., 2010). For lime-kaolinite mixture, the production of C-S-H, C-A-H and
71 C-A-S-H was reported by many researchers (Goldberg and Klein, 1952; Eades and Grim,
72 1960; Glenn and Handy, 1963; Willoughby et al., 1968; Bell 1996). C-S-H and C-A-H were
73 also detected in the lime-treated montmorillonite (Bell 1996; Hilt and Davidson, 1960). Arabi
74 and Wild (1989) noted that C-S-H hydrates were present in the lime-treated marls containing
75 illite, quartz and feldspar. Eades et al. (1962) also identified the production of C-S-H in the
76 lime-treated quartz.

77 Even though many studies were performed on lime-treated soils, most of them focused on the
78 lime-treated soil samples prepared in the laboratory. However, often lower performance of
79 lime-treated soils and poor durability of lime treatment are observed in the field conditions.
80 Puppala et al. (2006) reported about 40% lower for stiffness and 20 to 30% lower for strength
81 in the case of treatment in field. Similar results were reported by other researchers
82 (Horpibulsuk et al., 2006; Kavak and Akyarh, 2007). Additionally, higher hydraulic
83 conductivity and swelling potential of lime-treated soils in the field conditions were observed
84 (Bozbey and Guler, 2006; Cuisinier and Deneele, 2008). There are several factors that can
85 contribute to this difference between field and laboratory conditions. In addition to the
86 climatic factors, especially the wetting/drying cycles and freezing/thawing cycles (Pardini et
87 al., 1996; Guney et al., 2007; Tang et al., 2011; Stoltz et al., 2012), the aggregate size may
88 play an essential role in the hydro-mechanical behaviour of lime-treated soils (Tang et al.,
89 2011, Wang et al. 2015). The bender elements testing performed on the lime-treated samples

90 prepared with different maximum aggregates sizes ($D_{max} = 5, 2, 1$ and 0.4 mm) revealed that
91 lime-treated soils prepared with larger maximum aggregate size presented a relatively lower
92 stiffness (Tang et al., 2011). Dong (2013) also indicated that the lime-treated soil prepared
93 with larger aggregates was more sensitive to wetting/drying cycles. Note that aggregates are
94 assemblages of adjacent soil particles in which the cohesive forces are larger than the
95 disrupting force (Kemper and Chepil, 1965). In the field construction, the
96 scarifying/pulverizing process is performed before the lime treatment to control the soil
97 aggregate size. However, the size of soil aggregates in the field can still reach several
98 centimetres, which is much larger than that of soil aggregates prepared in the laboratory
99 before sample reconstruction. In the laboratory, natural tested soils are usually air-dried,
100 ground and sieved into few millimetres. For example, Du et al. (2014) who studied the
101 engineering properties and microstructure of the cement-stabilized contaminated soil,
102 prepared the samples with kaolin clay which had a maximum aggregate size lower than 2 mm;
103 while Cai et al. (2015) used reactive magnesia to treat in the laboratory a silty soil with the
104 same maximum aggregate size. Jiang et al. (2016) also reported that the used soil was first
105 passed through the sieve with 0.5 mm size prior to treatment.

106 As the improvement in the mechanical behaviour of soils by lime treatment is proven to be
107 primarily controlled by the cementitious compounds from the pozzolanic reactions, it is
108 expected that the different behaviours of treated soils with various aggregate sizes can be also
109 interpreted from mineralogical analysis. However, no studies have been conducted on this
110 aspect. This constitutes the main objective of the present work. In this study, three different
111 maximum aggregates sizes ($D_{max} = 5, 1$ and 0.4 mm) of soil powders were prepared before

112 lime treatment. The creation of cementitious compounds was investigated by X-ray diffraction
113 (XRD). In addition, environmental scanning electron microscope coupled with energy
114 dispersive X-ray spectrometry (Env. SEM-EDX) and mercury intrusion porosimetry (MIP)
115 were applied to investigate the microstructure and chemical composition of the treated soils.

116 **2. Materials and methods**

117 The tested soil was taken in Héricourt (France). It is a plastic silt with a clay-size fraction of
118 27 %. The basic geotechnical properties of this silt given by Wang et al. (2016) are listed in
119 Table 1. This soil corresponds to a silt of high plasticity (MH) following the Unified Soil
120 Classification System (USCS). To prepare soil powders with different maximum aggregate
121 sizes, natural soil was first air-dried, gently ground to crush the block of soil and passed
122 through three target sieves (their maximum diameters, D_{max} , are 5, 1 and 0.4 mm,
123 respectively). The large aggregates which could not pass through the sieve were ground
124 manually until all particles passed through, ensuring no changes in mineralogical
125 compositions during sieving (Tang et al., 2011). Afterwards, soil powders S5, S1 and S0.4 are
126 obtained, with D_{max} equal to 5 mm for S5, 1 mm for S1, and 0.4 mm for S0.4. Figure 1 shows
127 the aggregate size distributions of the three soil powders, determined by dry sieving.
128 Quicklime was used in this study and it has a high purity with a CaO content as high as
129 97.3 %. Particle size analysis shows that 82.7 %, 95.2 % and 100 % of this lime could pass
130 through 80 μm , 200 μm and 2 mm sieves, respectively (Dong, 2013). A lime content of 2 %
131 by mass was selected as binder dosage.

132 The dry soils were firstly mixed thoroughly with 2 % quicklime. Then, distilled water was
133 added by spray into the dry soil-lime mixture to obtain the target water content ($w = 17$ %, dry

134 side of the optimum according to the proctor curve). Static compaction was performed after a
135 mellowing period of 1 hour, to prepare soil samples at the target density ($\rho_d = 1.65 \text{ Mg/m}^3$).
136 The samples were carefully wrapped by plastic membrane and scotch tape immediately after
137 compaction. The well-covered sample was stocked in a hermetic box for curing in a chamber
138 at a temperature of $20 \pm 2 \text{ }^\circ\text{C}$. Prior to mineralogical and microstructural analyses, the samples
139 were freeze-dried following the procedure proposed by Delage and Pellerin (1984).

140 X-ray diffraction (XRD) analysis was performed on both untreated and treated samples. To
141 prepare soil powder for this analysis, freeze-dried and oven-dried samples were crushed and
142 ground to pass through a $32 \text{ }\mu\text{m}$ sieve. After sieving, soil powders were mixed well manually
143 in agate mortar and sprinkled gently in XRD sample holder using a $65 \text{ }\mu\text{m}$ sieve. The top
144 layer was removed carefully by cutting the surface with a thin razor blade leading to a smooth
145 surface without compaction (such preparation allows to decrease the preferential orientation
146 of clay particles). XRD patterns were obtained using a D8 Advance diffractometer from
147 Bruker (θ - θ configuration, Cobalt anode, $E = 35 \text{ kV}$, $I = 40 \text{ mA}$, no monochromator, LynxEye
148 detector). A continuous scan mode, between 3 and $80^\circ 2 \text{ theta}$, at a rate of $1 \text{ s per } 0.01^\circ 2 \text{ theta}$
149 was selected. Diffractograms were exploited with EVA program coupled with the ICPdf2
150 mineralogical database.

151 Environmental scanning electron microscope (Env. SEM, Quanta 400 from FEI company)
152 coupled with energy dispersive X-ray spectrometry (EDX from EDAX company) was
153 employed to observe the microstructure and to study the chemical composition of treated
154 samples. The EDX probe which provides an order of magnitude of the chemical content of
155 elements such as Ca, Si and Al was calibrated before the observations. Images were collected

156 on fresh fractured surface after freeze-drying. Secondary or back scattered electron mode was
157 selected in low vacuum mode (no metal coating was applied before observation).

158 The mercury intrusion porosimetry (MIP) test was carried out on freeze-dried samples. The
159 applied pressure ranged from 3.4 kPa to 230 MPa. The corresponding mercury intrusion
160 diameter varied from 0.006 μm to 355 μm . In the analysis, the surface tension of mercury was
161 taken equal to 0.485 N/m and the contact angle of mercury was taken equal to 130°.

162 Table 2 presents the test program. All the 2 % lime-treated samples were freeze-dried before
163 test except a piece of treated sample S1 after 1-year curing that was oven-dried at 105 °C
164 prior to testing. Untreated soil sample was also tested by XRD to obtain data as reference.

165 **3. Experimental results**

166 *3.1. Mineralogical analysis*

167 Figure 2 shows the XRD patterns of untreated soil and lime-treated soil S1 after 1-year curing.
168 The results illustrate that the main minerals in untreated soil are quartz and clays with the
169 presence of calcite and feldspars. The identification of clay minerals confirms the presence of
170 kaolinite ($d \sim 7 \text{ \AA}$), illite/muscovite ($d \sim 10 \text{ \AA}$), and montmorillonite ($d \sim 12\text{-}14 \text{ \AA}$). This clay
171 composition is consistent with previous analyses made by Deneele and Lemaire (2012), who
172 studied the same soil as that used in this study. The reflections associated with these clay
173 minerals (with quite similar intensity or position) are also present in the lime-treated soil. For
174 the lime-treated soil S1 ($t = 1 \text{ year}$) after freeze-drying, one new reflection at 2 theta equal to
175 34.2° ($d \sim 3.04 \text{ \AA}$) is identified, suggesting creation of the C-S-H phase. This C-S-H phase
176 would correspond to the main peak of synthetic tobermorite 9 \AA with the formula $5\text{Ca}_{0.65}\text{SiO}_2$,

177 2.5H₂O (Pdf file 010-0374) or to the calcium silicate hydrate Ca_{1.55}SiO_{3.5} xH₂O (Pdf file
178 033-0306). In order to remove the doubt of interpretation related to possible confusion among
179 the phases of unreacted CaO, C-S-H (tobermorite and other C-S-H forms) and magnesian
180 calcite, the XRD pattern of treated soil S1 after oven-drying at 105°C was also collected.

181 The new peak previously observed disappeared which tends to validate the identified C-S-H
182 phase. Indeed, oven-drying at 105 °C may dehydrate the C-S-H as suggested by Taylor (1997).
183 Gallé (2001) mentioned also that C-S-H decomposition starts at low temperatures and C-S-H
184 can be partially dehydrated at 105 °C, and then its crystallized structure disappears. Rio et al.
185 (2009) also observed that different stages of C-S-H dehydration took place at a temperature in
186 the range from 100 to 250 °C. If oven-drying at 105 °C can cause C-S-H degradation, it is not
187 the case for the magnesium calcite. Indeed, this latter remains stable under temperature effect
188 because decarbonation of calcite occurs at high temperatures (over 700 °C) according to
189 Collier et al. (2008), Rios et al. (2009) and Al-Mukhtar et al. (2014). If the new peak observed
190 on XRD pattern was calcite, this peak would remain present whatever the sample preparation.
191 It was obviously not the case. Moreover, the absence of new peak at $d \sim 3.04 \text{ \AA}$ at short curing
192 time (after just 1-day curing) allows excluding the presence of unreacted CaO after 1-year
193 curing.

194 Except the C-S-H phase, other minerals in the lime-treated soil seem similar to those of the
195 untreated sample. Note that the crystallized silicate source used to form C-S-H phase could
196 not be clearly identified by XRD because no clear peak decrease was detected, which would
197 signify its dissolution.

198 The effect of curing time and aggregates size can be specifically observed in a smaller range

199 from 31° to 39° 2θ where the C-S-H phase involved on the XRD patterns. The results of
200 treated samples S5 and S0.4 are illustrated in Figure 3a and 3b. Similarly to S1, S5 (Figure 3a)
201 also showed an XRD peak associated with the creation of C-S-H in the case of 1-year curing.
202 However, no sign of C-S-H was detected on the sample after a curing time of $t = 60$ days.
203 Surprisingly, no C-S-H reflection was detected on the XRD pattern of treated soil S0.4 after 1
204 year curing (Figure 3b). This point will be further discussed.

205 *3.2. Microstructure observation and chemical composition analysis*

206 Figures 4a-c present the distribution maps of silicon, aluminium and calcium derived from
207 EDX coupled with Env. SEM observations on treated soil S1 at a curing time of $t = 1$ day. The
208 two first elements belong to aluminosilicates phases such as feldspar and clays.

209 Areas with high concentration of Si in Figure 4a indicate the presence of quartz grains. The
210 calcium distribution map (Figure 4c) is established to localize hydrated lime after short curing
211 time (in this case, Ca^{2+} has not enough time to diffuse homogeneously into soil aggregates)
212 and the product of lime reaction such as C-S-H (at long time of curing). Even though the
213 protocol to mix the soil with lime was optimized to cover all the soil aggregates, calcium
214 seems to be distributed heterogeneously or rather, some aggregates of hydrated lime appears
215 clearly after 1 day of curing in isolated clusters of several micrometers (Figure 4d). It is
216 however worth noting that isolated small hydrated lime particles below the micron scale
217 cannot be distinguished by Env. SEM. The observations do not allow the detection of the
218 initial soil aggregate border formed by lime coating, whereas slight and continuous borders
219 composed by lime are expected on EDX mapping.

220 On lime-treated soil S1 after 1-year curing (Figure 5), the calcium distribution map (Figure 5c)

221 helps to localize the new pozzolanic products as shown in Figure 5d. New phases generally
222 present a typical water sensitive morphology of C-S-H particles characterised by a soft,
223 porous and rounded texture. Such C-S-H phases can be visually detected by operator on Env.
224 SEM images, only when their sizes are large enough to be distinguished, due to the limited
225 resolution of the Env. SEM apparatus applied in this study. If C-S-H phases are present in
226 particle form (but in a size smaller than 1 μm) or in form of thin gel layer, these C-S-H phases
227 are invisible for the operator. However, the C-S-H phases may be present by means of either
228 coating the soil aggregates, or filling the inter-aggregate pores, or binding the adjacent
229 aggregates together.

230 Finally, even though the XRD did not detect crystallized C-S-H in the lime-treated soil S0.4
231 after 1-year curing time, the Env. SEM observation revealed local presence of C-S-H particles
232 as shown in Figure 6. The low quantities of such large C-S-H particles may explain the result.

233 Quantitative EDX analyses complete the Env. SEM observations. Spot analysis at $E = 20 \text{ kV}$
234 were performed on areas situated on the EDX mapping in Figures 4 and 5 considering their
235 high calcium content. Table 3 gives the atomic percentages of the main elements present in
236 such areas compared to the atomic percentage measured on natural soil before lime addition
237 and in the areas far from the selected calcium spot. The calculated atomic ratio Ca/Si is equal
238 to 2.49 in areas rich in Ca^{2+} in soil S1 at $t = 1$ day while far from such area the Ca/Si ratio is
239 equal to 0.06 (with Si/Al = 2.16). Far from the calcium rich areas, the Ca/Si ratio remains
240 stable (or increases slightly to 0.09) even after 1 year of curing. In soil S1 at $t = 1$ day, the area
241 rich in calcium is associated with local hydrated lime particles $\text{Ca}(\text{OH})_2$. These particles
242 surround soil aggregates and calcium can be expected to diffuse into these aggregates. Note

243 that usual stoichiometries allow the identification of Si/Al ratios close to 1, 2 and 2.35 for
244 kaolinite, illite and montmorillonite, respectively (and the Ca/Si ratio is normally close to 0.07
245 in montmorillonite).

246 After a long time curing (1 year) and for any aggregate size, examination of the chemical
247 composition given by EDX on the new formed phase corresponding to C-S-H shows a
248 decrease and an increase of the calcium and silicon contents, respectively, as compared to the
249 initial spot analysis on hydrated lime particles. The Ca/Si ratio measured on C-S-H particles is
250 about 0.65-0.63 (while Si/Al is close to 2.16 or 2.32) and this ratio is in accordance with the
251 composition of synthetic tobermorite 9 Å ($\text{Ca}_{0.65}\text{SiO}_2, 2.5\text{H}_2\text{O}$) found by XRD. This is also in
252 agreement with the observations by Brunauer (1962) and El-Hemaly et al. (1977) suggesting a
253 minimum value of 0.8-0.9 for C-S-H gel produced in the mixture of lime-silica-water. Studies
254 of crystal structures of "tobermorites" (Merlino et al. 1999, 2000, 2001) showed that
255 tobermorite is a series between two endmembers, $\text{Ca}_4\text{Si}_6\text{O}_{15}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$ and $\text{Ca}_5\text{Si}_6\text{O}_{17} \cdot 5\text{H}_2\text{O}$,
256 the Ca/Si ratio varying between 0.66 and 0.83. Wild et al. (1986) also stated that hydration of
257 cement at room temperature created a poorly crystallized C-S-H gel with a high Ca/Si ratio of
258 about 1.5 but the similar C-S-H gel from the reaction in the lime-silica-water mixture had a
259 lower Ca/Si ratio.

260 *3.3. Pore size distribution*

261 Results obtained from MIP tests on the lime-treated soils (S5, S1 and S0.4) during a curing
262 time from 7 to 90 days are presented in Figure 7. Data obtained on soils S5 and S0.4 have
263 been presented previously in Wang et al. (2015). The pore size distributions of all lime-treated
264 samples compacted on the dry side of optimum present a bi-modal behaviour with a

265 population of macro-pores (inter-aggregate pores) and a population of micro-pores
266 (intra-aggregate pores). Note that the delimitation of the pore entrance diameter between the
267 macro-pores and micro-pores is defined as $d = 2 \mu\text{m}$ in this study (Romero et al., 2011).
268 Generally, macro-pores (larger than $2 \mu\text{m}$) are greatly impacted during compaction by the
269 aggregate size: the larger the aggregate size is, the larger the modal entrance size of the
270 macro-pores appears while aggregate size has negligible effect on the micro-pores (smaller
271 than $2 \mu\text{m}$) (Wang et al., 2015). As an illustration, the modal size of macro-pores for the
272 untreated sample S5 is around $73 \mu\text{m}$ and in comparison, lower value is obtained for the
273 untreated S0.4 soil with a main pore family around $14 \mu\text{m}$. Nevertheless, these untreated
274 samples with different D_{max} values share a similar modal size of micro-pores. The mean value
275 of the pore entrance diameter of the micro-pore family remains around $0.3\text{-}0.4 \mu\text{m}$, as seen in
276 Figure 7.

277 Lime treatment progressively impacts the microstructure of soil. The quantity of each pore
278 population was given as the ratio of intruded void ratio to the total void ratio equal to $e = 0.68$
279 (Wang et al., 2016), as illustrated in Table 4. For treated soil S5, an increase in the frequency
280 of the entrance pore diameters between 0.006 and $0.1 \mu\text{m}$ (defined as “nano-pores” in the
281 following text for clarity) can be observed in Figure 7a at a curing time of $t = 90$ days,
282 compared with that after 7-day curing. Particularly, the nano-porosity of sample S5 after
283 90-day curing is 27 % larger than that of the sample after 7-day curing. Less change took
284 place for macro-pores, except a slight decrease in the modal size of macro-pores. The total
285 intrusion value measured on S5 in Figure 7b seems quite similar for the two curing periods.
286 As illustrated in Figure 7c, similar results are observed on treated soil S1: after 90-day curing,

287 a small increase (around 14 %) in the frequency of nano-pores ranging from 0.006 to 0.1 μm
288 is detected and the cumulative intrusion curve of S1 at 90 days is similar to that at 7 days. In
289 the case of sample S0.4, a slight increase of 16 % in the quantity of the nano-pores is also
290 detected for the sample after 90-day curing, in comparison with the sample at 7-day curing. In
291 addition, both modal sizes of macro-pores and micro-pores shift slightly to lower values, by
292 keeping similar shape during curing and decreasing both the quantities of macro-pores and
293 micro-pores, as seen in Figure 7e. It is worth noting that the total intrusion value of S0.4 at 90
294 days decreases significantly compared with that at 7 days, induced by a reduction of both the
295 modal pore entrance size and the quantity of the large macro-pores (in Figure 7f).

296 **4. Discussion**

297 Pozzolanic reactions occur with 2 % lime added to the soil from Héricourt that contains
298 quartz, feldspar and clays, and these reactions are strongly time-dependent. New crystallized
299 phase appeared after a long time of curing (at least 1 year) and the main reflection
300 characterizing this new phase is positioned at 34.2° (2 theta $\text{CoK}\alpha$) (corresponding to $d =$
301 3.04 \AA), close to the main peak of calcite that is present in untreated soil. Comparison of XRD
302 patterns of treated soil S1 after 1-year curing and after oven-drying (at 105°C) or after
303 freeze-drying demonstrated that the new peak at $d = 3.04 \text{ \AA}$ corresponds to the crystallized
304 C-S-H phase (probably at tobermorite 9 \AA) (see Figure 2). The chemical analysis on the
305 C-S-H phase by means of Env. SEM-EDX, gave a Ca/Si ratio equal to 0.63-0.65 for both S1
306 and S0.4, in agreement with the tobermorite 9 \AA composition. No significant reflection
307 corresponding to C-S-H was detected at a curing time of $t = 60$ days. This can be explained by
308 the absence of well crystallized C-S-H or by the low detection limit of XRD technique.

309 Indeed, it is difficult to detect any crystalline phase if it is poorly crystallized, amorphous, or
310 when its quantity is too small, generally below 0.5-1 % (Carter et al., 1987; Mitchell, 1993;
311 Moon et al., 2004). In other words, very low quantity of well-crystallized C-S-H phase or
312 amorphous C-S-H gel may be formed at 60 days, whatever the aggregate size.

313 However, at a long curing time ($t = 1$ year), the aggregate size seems to impact significantly
314 on the pozzolanic reactions among the lime-soil-water system. C-S-H phase can be detected
315 on the treated soil prepared with large aggregates, such as S5 and S1 while crystallised C-S-H
316 are difficult to be observed by XRD technique on soil with small aggregates (as seen in S0.4)
317 even though Env. SEM pictures revealed the presence of isolated C-S-H particles in big
318 clusters as for S0.4. Note that the low frequency of such clusters, or the low degree
319 crystallisation of C-S-H phase in S0.4, explains why they were not detected by XRD.

320 Furthermore, when lime powder was mixed with small-sized soil aggregates such as S0.4, a
321 thin layer of lime-aggregate (named transition zone by Ping et al. (1991)) was expected due to
322 the large contact surface of small aggregates. On the contrary, lime powders can be locally
323 concentrated in thick layer on the surface of large aggregates (such as those in S1 and S5)
324 which induce small contact surface area. This is consistent with the experimental results of
325 Ping et al. (1991), who studied the aggregate size effect on the transition zone between
326 granular aggregate and cement paste. The authors reported that the thickness of transition
327 zone decreases with the decrease of the aggregate size. As curing time increased, this area
328 where lime is concentrated is in favour of the formation of larger well-crystallized C-S-H
329 particles. Conversely, a more homogeneous lime distribution in S0.4 should induce either the
330 creation of a poorly-crystallized or amorphous C-S-H phase or gel, or a nanometric

331 well-crystallized C-S-H particle which is hardly detected by XRD. Even though in S0.4, these
332 C-S-H phases cannot be directly detected by XRD and only localised C-S-H phases were
333 observed by Env. SEM. The appearance of these C-S-H phases was indirectly confirmed by
334 Tang et al. (2011) who conducted bender element tests on lime-treated soils with different
335 aggregate sizes They reported a higher stiffness for the lime-treated soil with $D_{max} = 0.4$ mm
336 at a long curing, compared to that with $D_{max} = 5$ mm. Comparison with the untreated sample
337 showed that the treated samples had a higher stiffness, whatever the aggregate size.
338 Cementitious compounds bond the soil particles together, inducing the increase of the soil
339 stiffness.

340 Furthermore, the aggregate size effect on the microstructure of treated soil is also consistent
341 with previous mineralogical analysis. A relatively larger increase in frequency was observed
342 for the nano-pores ranging from 0.006 to 0.1 μm on treated soil with large aggregates,
343 especially for S5 after long curing time. These nano-pores can be attributed to the formation
344 of well-developed C-S-H phase (a hydrated lamellar structure). This formation was favoured
345 by the thick coating of lime around soil aggregates or by the local lime concentration in
346 clusters as observed by Env. SEM. Similar observations were made by Russo and Modoni
347 (2013) who reported that a pore population of 0.007-0.2 μm was developed in a
348 lime-stabilised soil at long term. Alvarez et al. (2013) found a small amount of pores below
349 0.02 μm , ascribed to C-S-H gel pores. In this study, the formation of C-S-H lead to the
350 creation of nano-pores ranging from 0.006 to 0.1 μm , as observed in the mixture of lime and
351 soils with large aggregates (S5). Such new nano-porous compounds filled the micro-pores
352 (0.1 to 2 μm), decreasing the frequency of these micro-pores. However, less quantity of

353 nano-pores was found in treated soil S0.4 at the same curing period. While a large reduction
354 in the total intrusion value of S0.4 at 90 days was identified, indicating that more undetectable
355 nano-pores were created (their sizes are lower than 0.006 μm that corresponds to the detection
356 limit of MIP measurement in this study), since the initial void ratio of the sample remains
357 identical (Wang et al., 2016).. These undetectable nano-pores are mainly associated with
358 C-S-H phases, which were generated from the pozzolanic reactions in S0.4 sample. This also
359 agrees with the hypothesis of the formation of nano-crystalline C-S-H or small-sized
360 amorphous C-S-H gels, which is invisible by XRD technique and whose nano-pores are
361 undetectable by MIP measurement. Furthermore, these C-S-H phases in S0.4 soil gradually
362 cover the surface of small soil particles to a large extent, bond the adjacent soil particles
363 together, and also gradually block some entrances of both macro-pores and micro-pores. This
364 may explain the small shift of the modal size of both macro-pore and micro-pore populations.
365 As the new phase filled the pores and blocked the pore entrances, initially un-constricted
366 pores can become constricted pores, leading to a decrease in the total intrusion value (Wang et
367 al., 2015). This is in agreement with the observation by Russo and Modoni (2013).

368

369 **5. Conclusion**

370 The changes in mineralogical composition and microstructure of a lime-treated compacted
371 soil during curing were investigated, with emphasis on the effect of aggregate size. On the
372 basis of the experimental results, some conclusions can be drawn, as follows:

373 1) XRD peaks of calcium silicate hydrates (C-S-H) on crystallized tobermorite form were

374 observed on patterns of freeze-dried lime-treated soil sample with large aggregates (S5
375 and S1) after 1-year curing, while no such C-S-H was observed at shorter curing times.
376 Oven-drying at 105°C allowed confirming the mineralogical nature of the new formed
377 phase as C-S-H.

378 2) When a given quantity of lime is mixed with large aggregates (S1 and S5),, lime particles
379 during mixing can be concentrated locally within the mixture in order to form lime
380 aggregates or thick coating on soil aggregates. Such calcium-rich areas contribute to the
381 formation of large and detectable C-S-H crystallites as observed by XRD. By contrast, a
382 diffused distribution of the lime powder was expected in the small soil aggregate-lime
383 mixture (S0.4). The low rate of lime aggregation benefits to the development of small
384 (nano) crystallized C-S-H particles, or poor crystallized C-S-H phases, or amorphous
385 C-S-H gels..

386 3) The decrease of population of micro-pores and the increase of nano-pores in frequency
387 observed in large aggregates lime mixtures (S5 and S1) can be attributed to the formation
388 of visible crystallized C-S-H pores: the new nano-porous compound filled the micro-pores.
389 By contrast, the soil with small-sized aggregates S0.4 mixed with lime behaved differently:
390 very small or nano-sized C-S-H phase homogeneously spread on large soil aggregate
391 surface was expected around soil aggregates (because of the initial thin coating with lime).
392 As a result, the nano-sized pores expected to be less than 0.006 μm in the nano-sized
393 C-S-H particles were too small to be detected by MIP.

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536

537

538 **Table 1 ESEM-EDAX element analysis of S1 at a curing time of $t = 1$ day, both on the spots where lime**
 539 **and clay particle located**

Element	Wt %		At %		K-Ratio	
	Lime	Clay	Lime	Clay	Lime	Clay
C	6.37	4.28	10.85	7.23	0.0171	0.0068
O	50.54	49.47	64.67	62.74	0.0959	0.1652
Na	0.14	0.21	0.12	0.19	0.0004	0.0007
Mg	0.77	0.73	0.65	0.61	0.0035	0.0036
Al	3.65	10.18	2.77	7.65	0.021	0.0624
Si	7.71	22.88	5.62	16.53	0.0529	0.1496
K	0.39	1.76	0.2	0.91	0.0037	0.015
Ca	27.38	2.06	13.99	1.04	0.2574	0.0185
Ti	0.28	0.59	0.12	0.25	0.0022	0.0051
Fe	2.78	7.83	1.02	2.85	0.0236	0.0683

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543 **Table 2 ESEM-EDAX element analysis of S1 at a curing time of $t = 1$ year, on the spot where CSH located**

Element	Wt %	At %	K-Ratio
C	3.33	5.61	0.0064
O	52.49	66.42	0.1295
Na	0	0	0
Mg	2.32	1.93	0.0113
Al	6.83	5.12	0.0407
Si	16.54	11.92	0.1112
K	0.41	0.21	0.0037
Ca	15.55	7.85	0.1421
Ti	0.17	0.07	0.0014
Fe	2.37	0.86	0.0203

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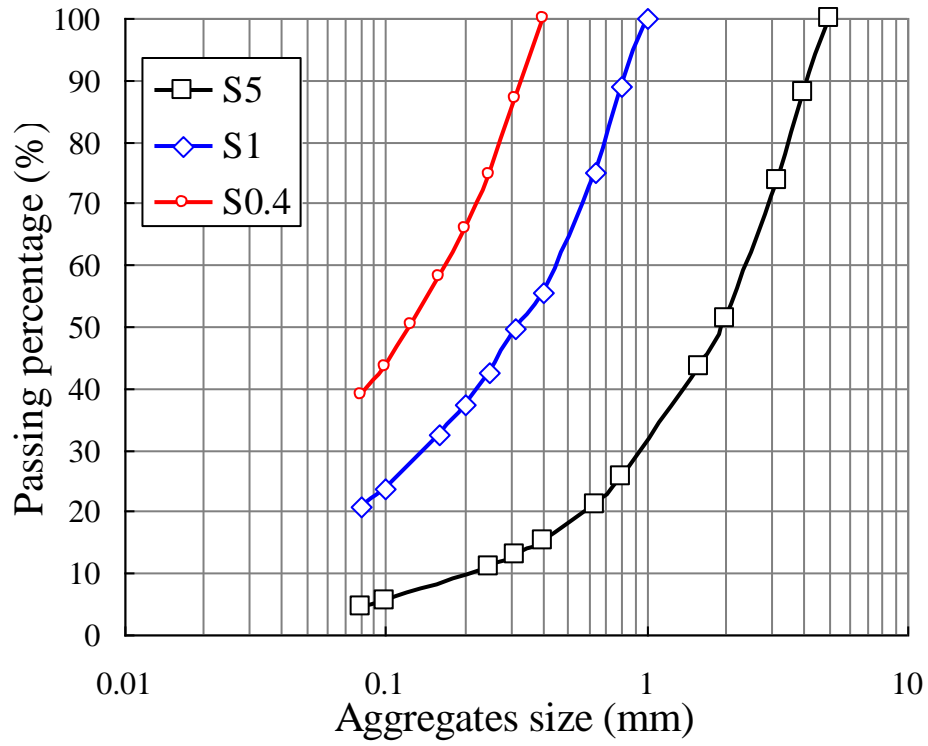
547 **Table 3 ESEM-EDAX element analysis of S0.4 at a curing time of $t = 1$ year, on the spot where CSH**
548 **located**

Element	Wt %	At %	K-Ratio
C	7.1	11.32	0.0147
O	55.15	66.01	0.1435
Mg	1.89	1.49	0.009
Al	6.3	4.47	0.0372
Si	14.2	9.68	0.0953
K	0.25	0.12	0.0022
Ca	12.78	6.11	0.1171
Fe	2.34	0.8	0.0201

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551 **Figure 1 Aggregate size distribution of soil S5, S1 and S0.4**

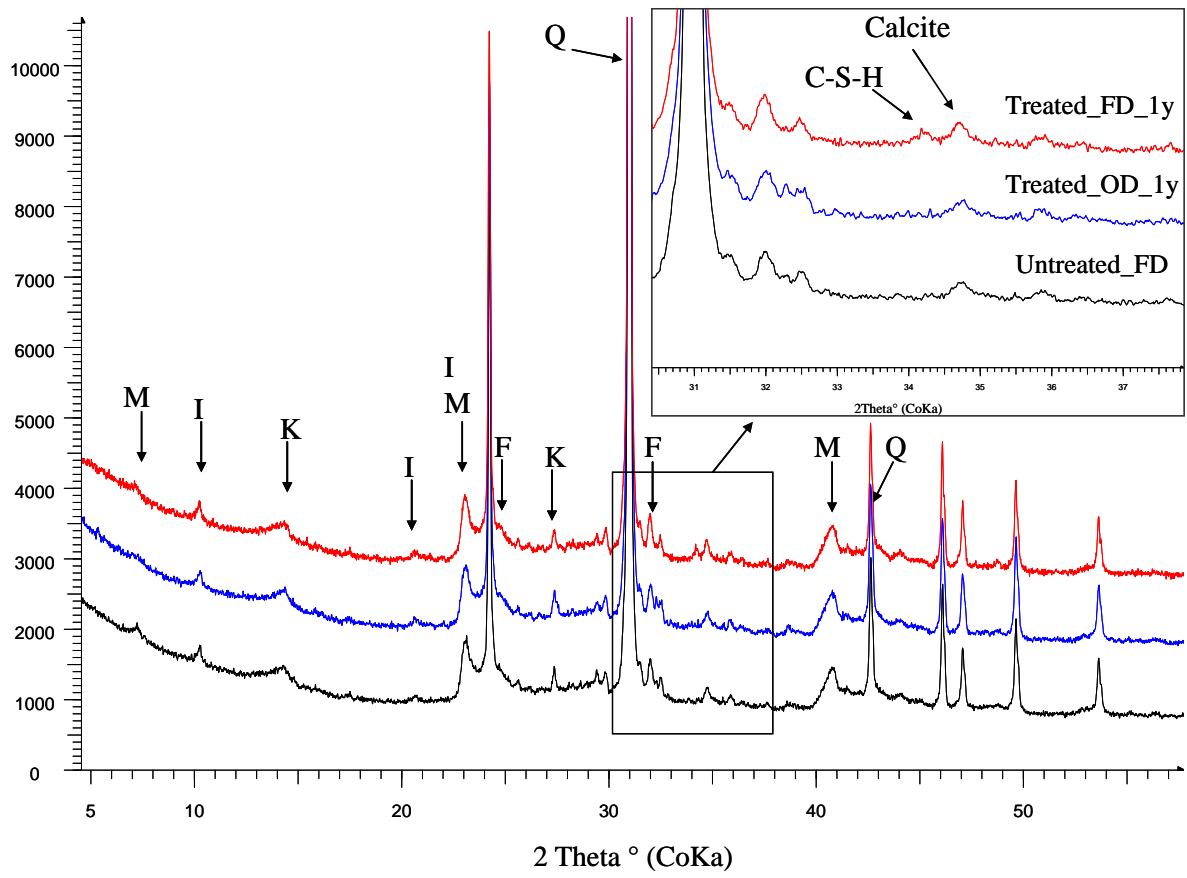


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555 **Figure 2 X-ray diffraction patterns of the untreated soil S1**



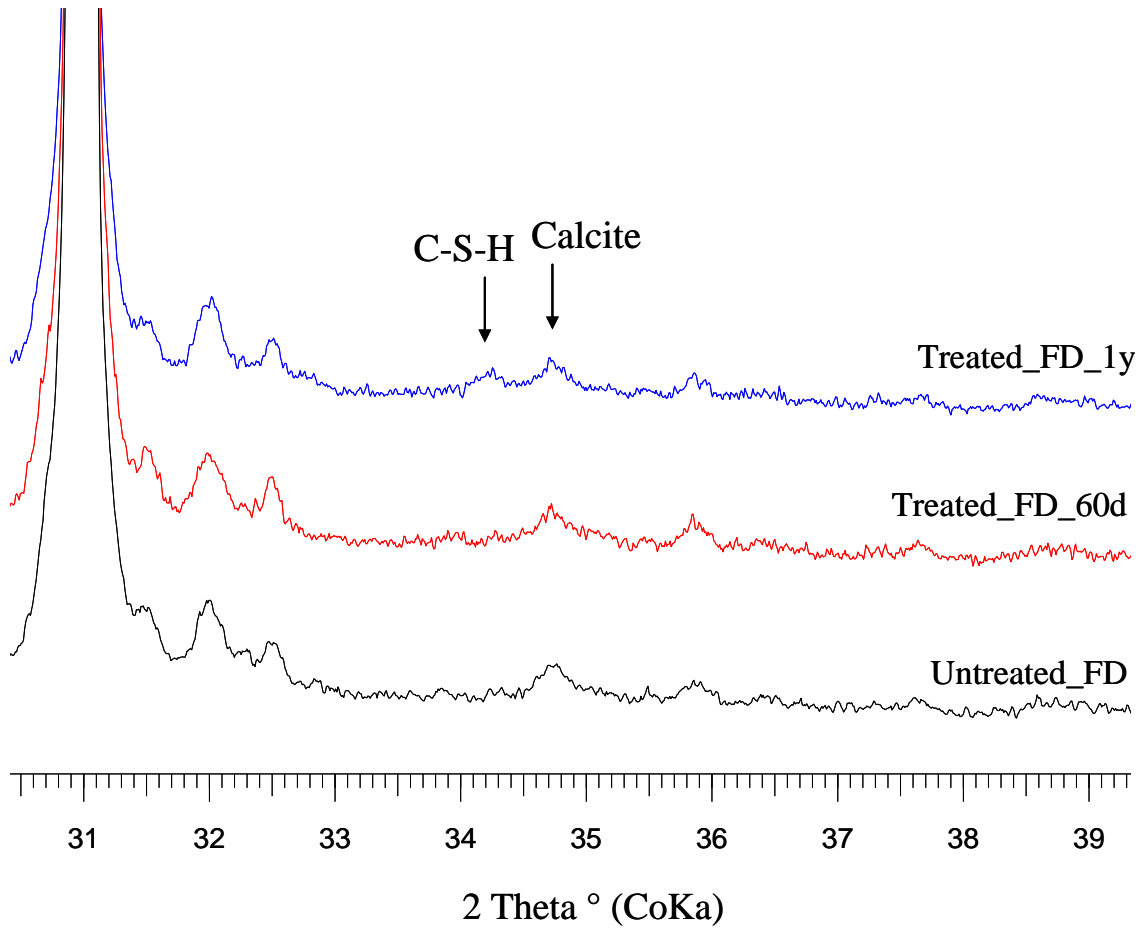
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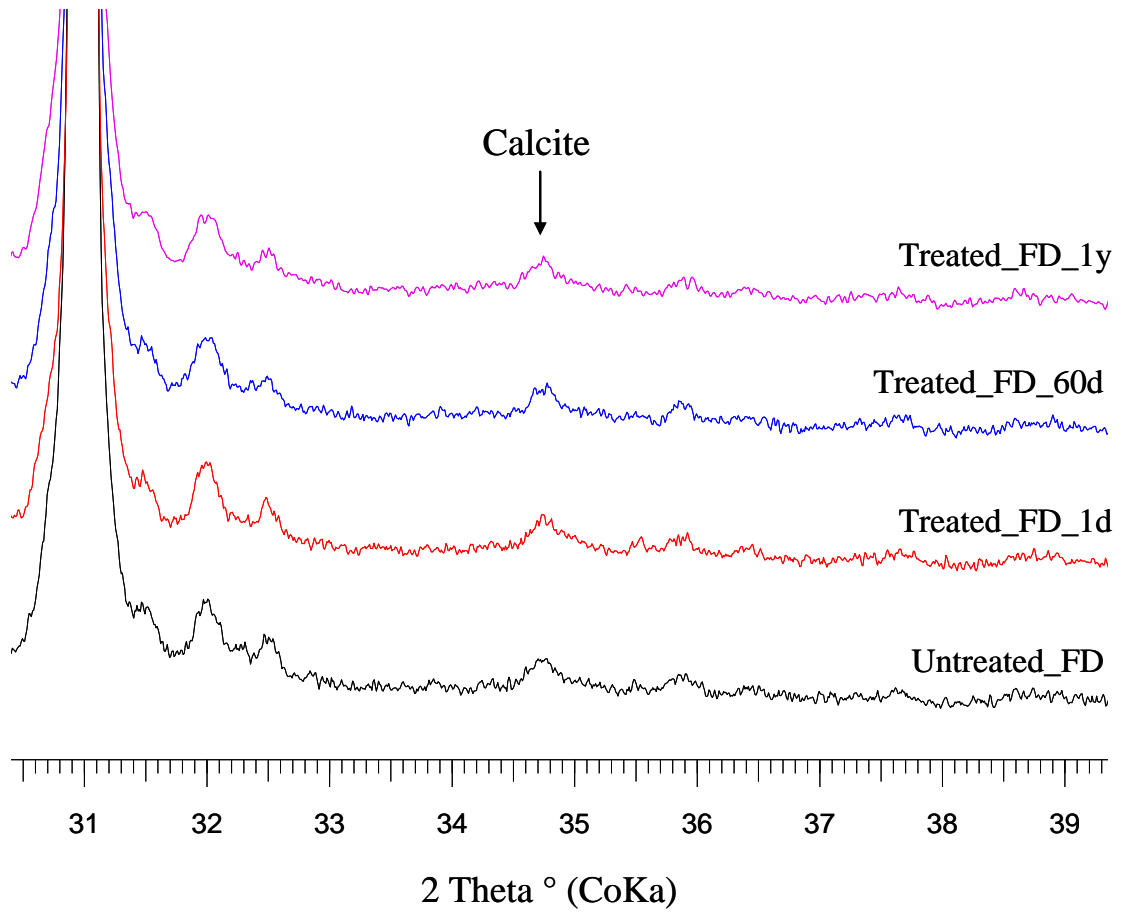
559 **Figure 3 X-ray diffraction patterns of lime-treated soils during curing: (a) S5; (b) S0.4**

(a)



560

(b)



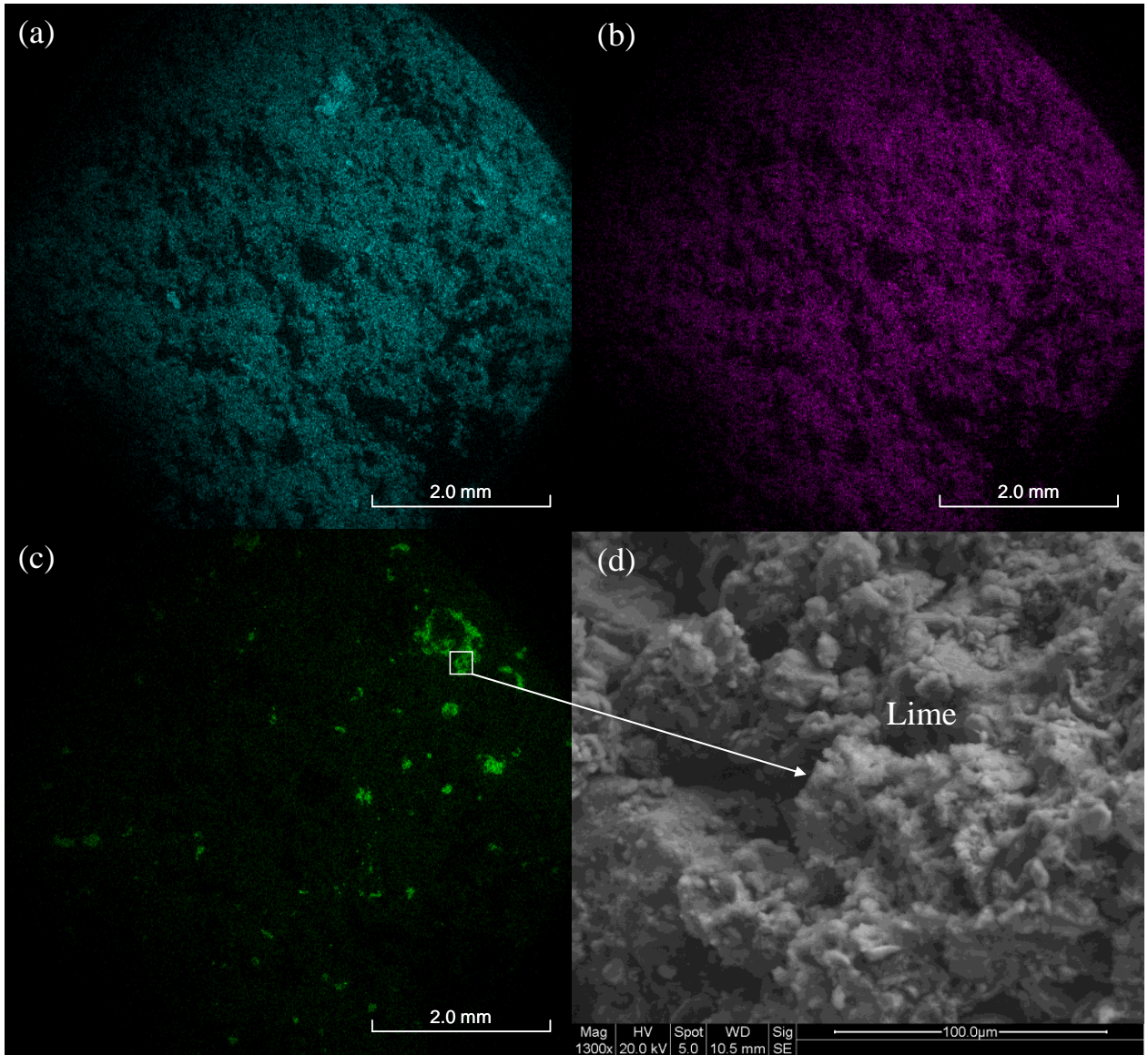
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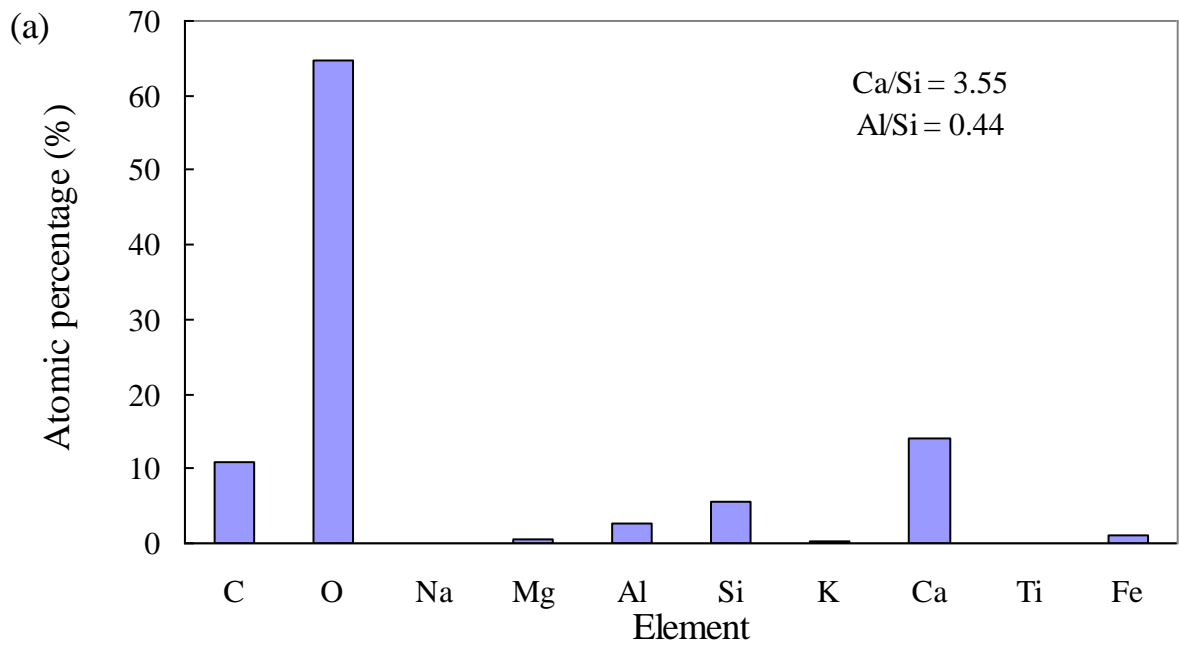
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565 **Figure 4 Elementary distribution maps of a lime-treated soil, S1, at a curing time of $t = 1$ day: (a) Silicium;**
566 **(b) Aluminium; (c) Calcium, and SEM picture of the spot in the calcium concentration area**

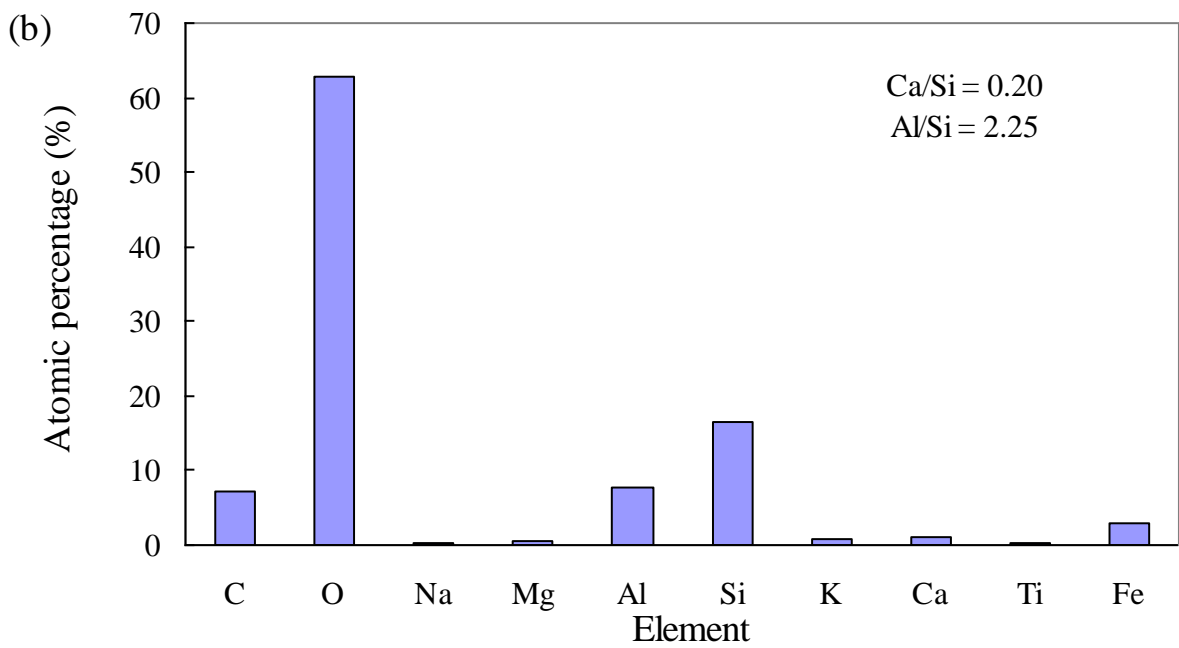


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568 **Figure 5** Element examination by EDAX on lime-treated soil S1 at a curing time of $t = 1$ day: (a) on the
569 spot in the calcium concentration area; (b) on the spot in the clay particle area



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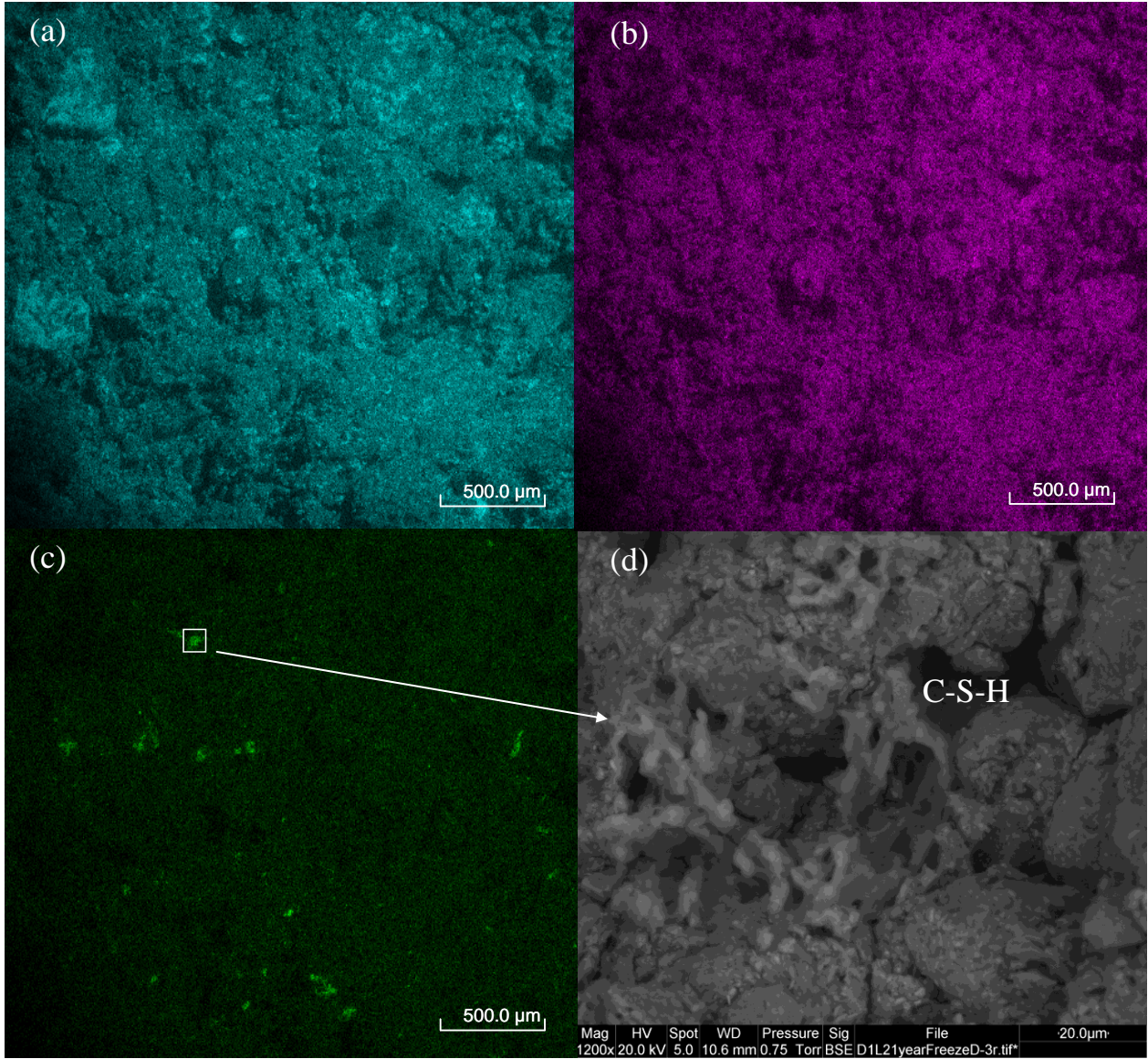
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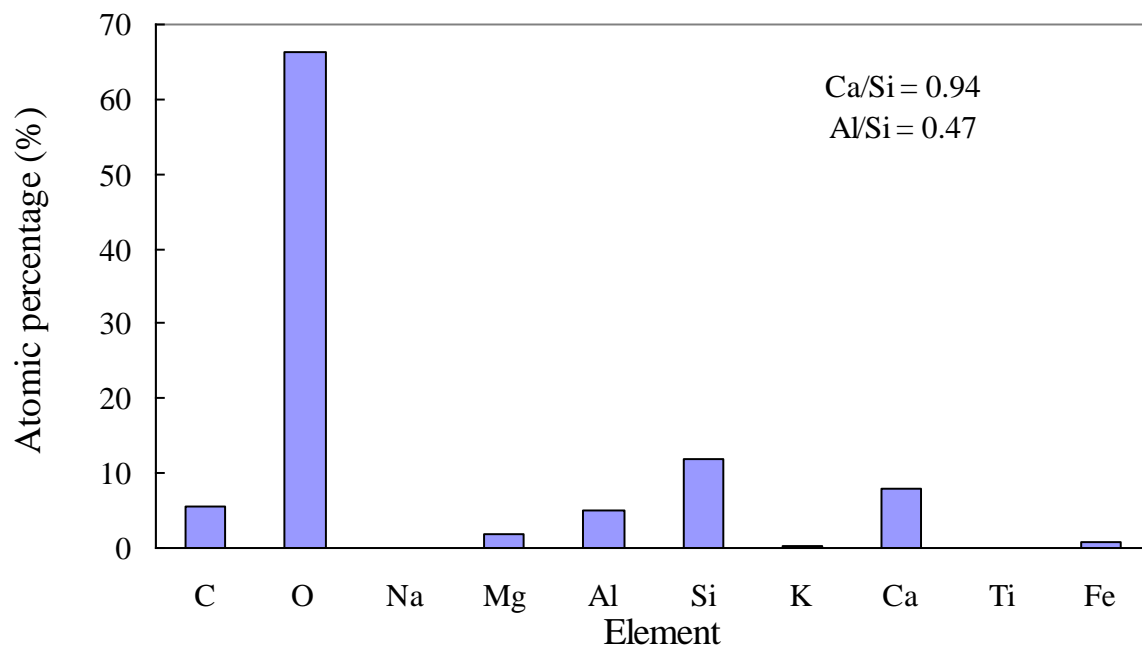
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575 **Figure 6 Elementary distribution maps of a lime-treated soil, S1, at a curing time of $t = 1$ year: (a) Silicium;**
576 **(b) Aluminium; (c) Calcium, and SEM picture of the spot in the calcium concentration area**



577

578 **Figure 7 Element examination by EDAX on lime-treated soil S1 at a curing time of $t = 1$ year, on the spot**
579 **in the calcium concentration area**



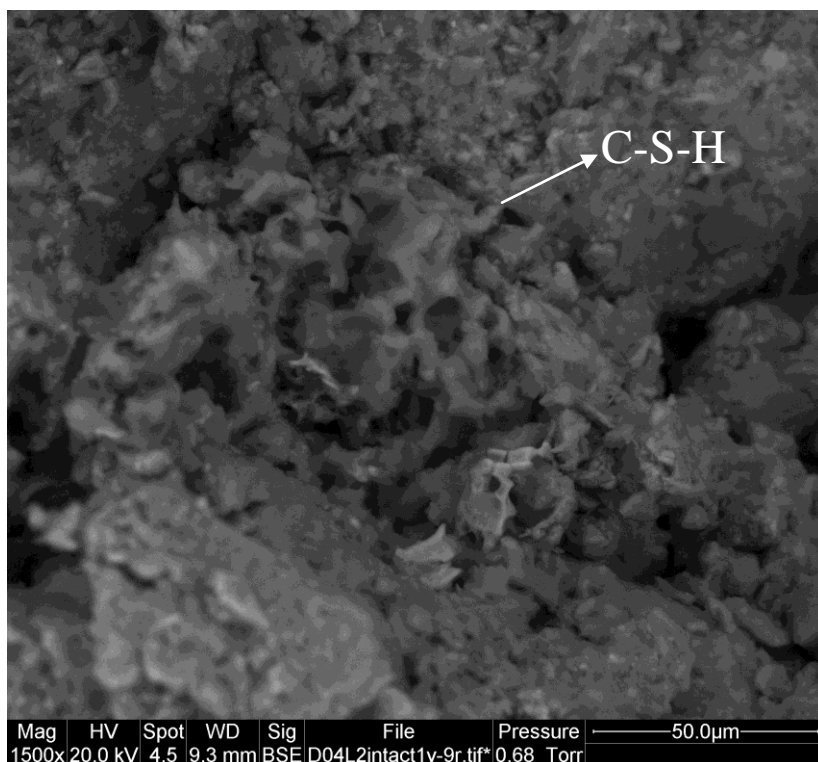
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584 **Figure 8 SEM picture of the lime-treated soil, S04, at a curing time of $t = 1$ year**

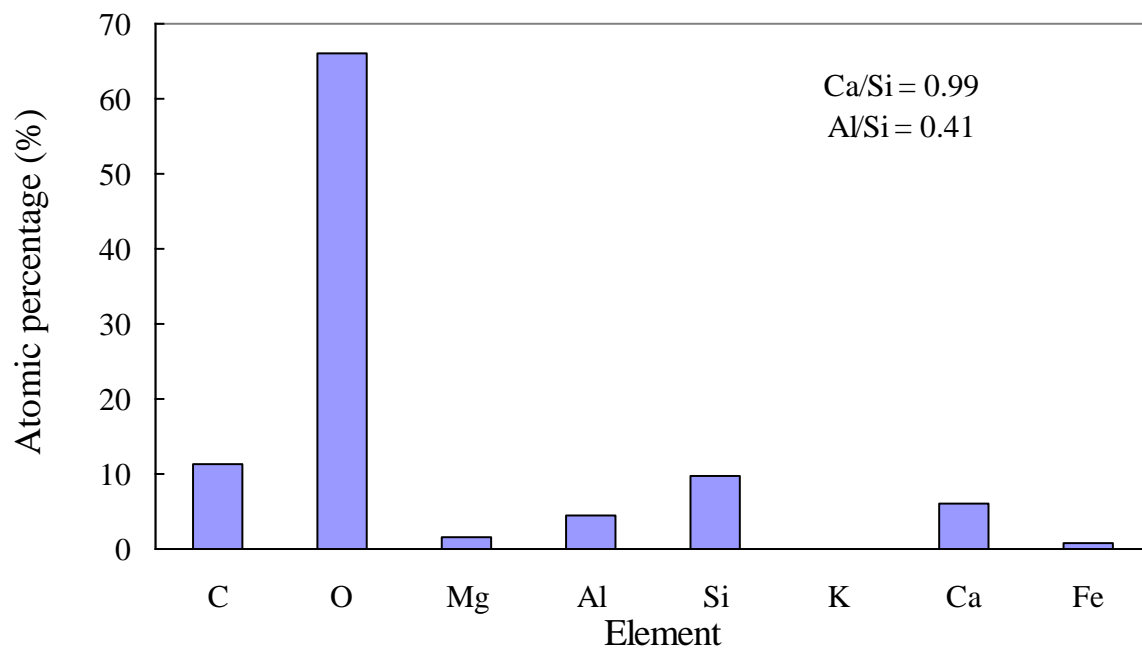


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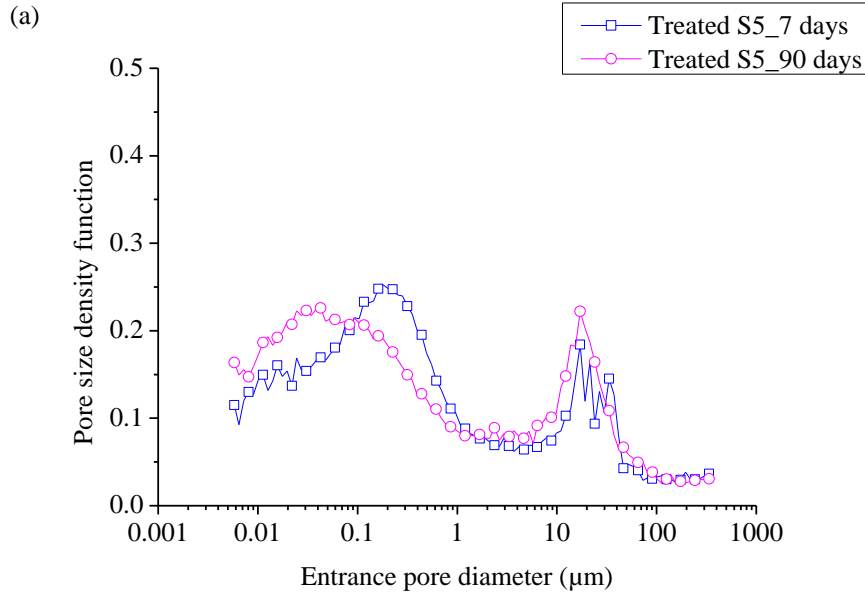
588 **Figure 9** Element examination by EDAX on lime-treated soil S0.4 at a curing time of $t = 1$ year, on the spot
589 **in the calcium concentration area**



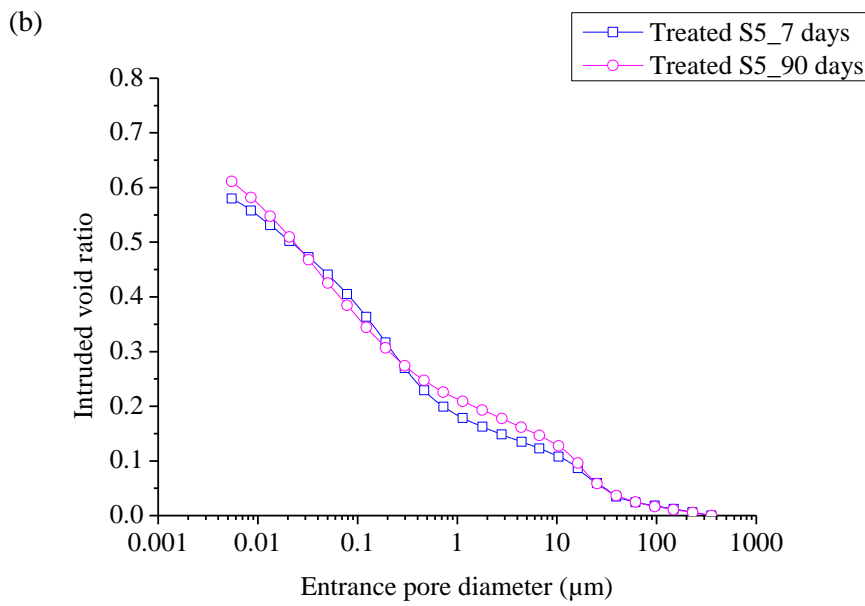
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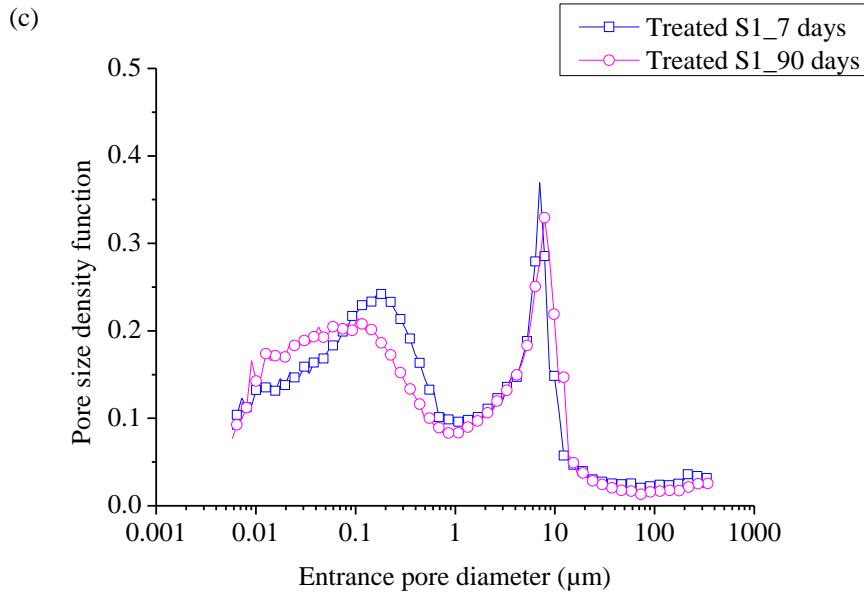
592 **Figure 10 Pore size distributions and cumulative intrusion curves of lime-treated soils with different**
593 **aggregate sizes (S5, S1 and S0.4), obtained by MIP**



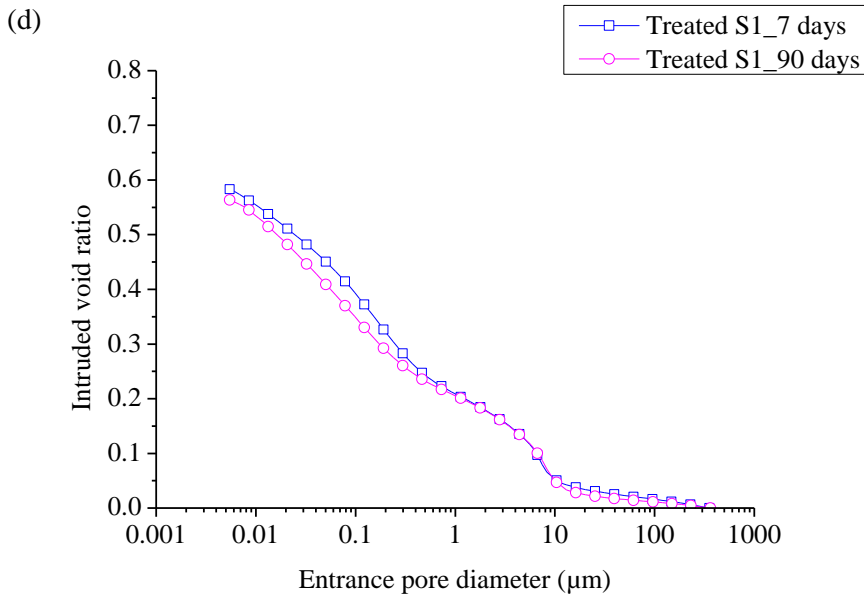
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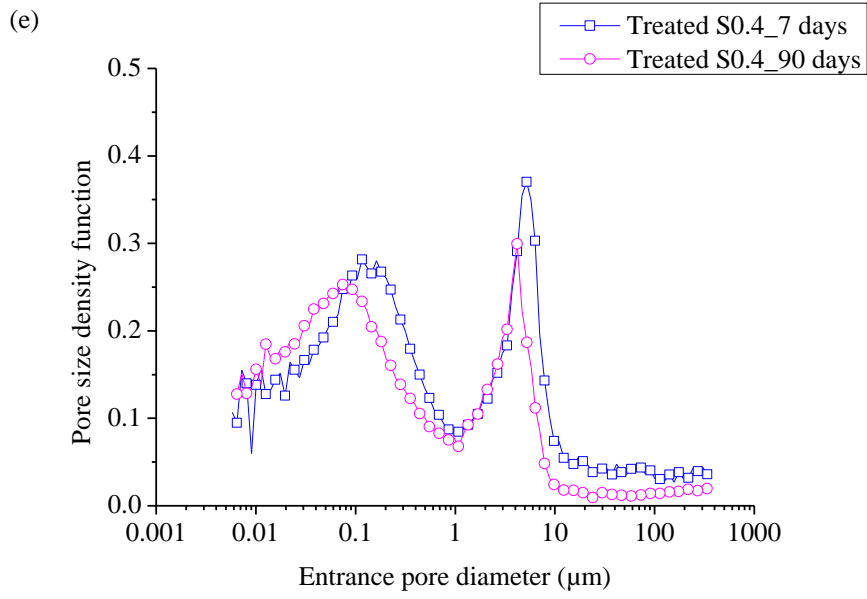
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