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1 Soil treatment with organic non-traditional additives for the improvement of earthworks

2

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16

17 Abstract

18 The aim of this study was to characterise the technical and environmental implications of  
19 non-traditional treatments of a low-plasticity compacted silt ( $PI = 14$ ) and to investigate their  
20 actions both at the micro- and macro-structural scales. Three non-traditional additives  
21 derived from industrial vegetal by-products were studied. These additives are classified as an  
22 acid solution (AS), an enzymatic solution (ES) and calcium lignosulfonate (LS). The first step  
23 was to characterise the effects of these treatments on Proctor compaction, bearing capacity,  
24 unconfined compressive strength and stiffness. The index properties of the treated samples  
25 were also measured to assess changes in the interaction between soil minerals and the  
26 additives. These experimental results showed that the 0.002% ES and 2.0% LS treatments  
27 were the most effective at improving the soil dry density and allowing the soil to reach  
28 optimum density using 3% less water or 25% less compaction energy. In a second step the  
29 mechanisms involved by the treatment were investigated. A microscopic study was  
30 conducted including scanning electronic microscopy (SEM) and mercury injection porosity  
31 (MIP) tests, measurements of the surface tension of non-traditional additives mixed with  
32 water completed the study and demonstrated that the ES treatment has the same surfactant  
33 properties as sodium dodecyl sulfate, a common surfactant. Compaction tests confirmed that  
34 the behaviours of the soil after ES and sodium dodecyl sulfate treatments were similar.

35 Key words: Soil treatment, non-traditional additives, compaction, bearing capacity, surfactant

36

37           **1. INTRODUCTION**

38       In earthworks, sustainable development will play a larger role in future project design and the  
39       assessment of technical solutions by contractors. Currently, the reduction of the  
40       environmental impact of earthworks is a growing issue. One of the major challenges facing  
41       earthworks companies is the need to use a majority of natural materials extracted within the  
42       construction site, including materials with very low geotechnical characteristics. The common  
43       industry solution is to use lime, cement or fly ash to improve the soil geotechnical properties;  
44       however, sustainable development principles also require a reduction in the consumption of  
45       water and non-renewable energies as a way to limit greenhouse gas emissions. In this  
46       context, soil treatment with various non-traditional additives has been tested. These additives  
47       are diverse in nature and some of them derive from the industrial transformation of  
48       renewable raw materials. Due to their organic nature, some products are biodegradable.  
49       Thus, the treated soils can recover their untreated composition after the end of the  
50       biodegradation process. These products are also beneficial due to their efficiency at low  
51       dosages, thereby limiting the environmental impact and cost of their use. Various additives  
52       have been tested for improving the compaction of dry soils to avoid dust generation and for  
53       increasing the durability of uncovered roads [5, 22, 24, 27]. Additionally, additives have been  
54       tested to stabilise erodible soils [33]. Mechanical properties, such as the unconfined  
55       compressive strength [23, 28] or the swelling potential of plastic soils [11, 19], have also  
56       been studied.

57       Tingle and Santoni [28] listed seven non-traditional categories based on the major active  
58       component and classified them as salts, acids, enzymes, lignosulfonates, petroleum  
59       emulsions, polymers and tree resins. Acids are low pH aqueous solutions containing  
60       sulfonated molecules such as naphthalene or limonene [13]. The presence of proteins in  
61       enzymatic solutions was confirmed by Velasquez et al. [32]. Lignosulfonates are organic

62 polymers derived from lignin extracted by the sulfite processing of cellulose in the wood-pulp  
63 and paper industries. According to Tingle et al. [30], petroleum emulsions contain asphalt or  
64 synthetic iso-alkane fluids suspended in emulsions by a surfactant, polymer emulsions  
65 contain vinyl acetates or acrylic copolymers and tree resins are diverse emulsified by-  
66 products of the timber and paper industries. Nevertheless, many of these products also  
67 include secondary additives such as surfactants or ultraviolet inhibitors, which may react with  
68 the soil minerals or modify the soil water properties and thus influence the mechanical  
69 behaviour of the treated soils [30]. The emphasis of this study was put on non-traditional  
70 additives that would lower the environmental impact of soil treatment in the earthworks  
71 industry. Therefore, this study focused on the following products which are derived from the  
72 transformation of renewable organic matters: acids, enzymes and lignosulfonates. For each  
73 of these categories, a review of the literature concerning the effects on geotechnical  
74 properties and suspected mechanisms is presented. Then, geotechnical properties such as  
75 compaction, bearing capacity, unconfined compressive strength and stiffness were  
76 investigated to define the potential application of these treatments. In the last part, the action  
77 of the treatments on the microstructure of the soil was studied by performing scanning  
78 electronic microscopy (SEM) and mercury injection porosity (MIP) tests to identify the  
79 mechanisms involved by the different products.

## 80 **2. LITERATURE REVIEW**

### 81 **2a. Acid solutions**

82 Rauch et al. [20] studied the effects of an acid product on the index properties of three pure  
83 clay phases (kaolinite, illite and montmorillonite) and two plastic soils. They concluded that  
84 the acid treatment had little effect on soil plasticity, swelling potential and shear strength. The  
85 authors explained that the observed modifications were induced by initial differences in  
86 properties of the compacted samples. Tingle & Santoni [28] and Santoni et al. [23] tested the

87 application of an acid solution to three soils ranging from non-plastic to highly plastic in  
88 nature. They found no change in the unconfined compressive strength. The authors  
89 determined that the acid treatment had no effect on the compaction properties of the soils.  
90 However, another study [21] showed that changes in the compaction properties of acid-  
91 treated soils can occur. Scholen [24] reported in situ studies undertaken by the US Forest  
92 Service between 1991 and 1993 which tested the addition of acid solutions. In 1995, no  
93 particular failure or degradation of the treated sections was reported. Scholen [24] proposed  
94 that acidic non-traditional additives act by altering the electrolyte concentration of the pore  
95 fluid, thereby reducing the double layer thickness of clay minerals. Cation exchange was also  
96 suspected to occur and to flocculate the clay minerals. Moreover, important lowering of the  
97 pH of a soil can also lead to the dissolution of clay minerals [26, 36]. For example, to start the  
98 dissolution of kaolinite, the pH values have to be lower than 2 [16, 35]. However, the X-ray  
99 diffraction, specific surface area and cation exchange capacity were not modified by the  
100 treatments, even for dosages as high as 50% [13, 21]. Scanning electron microscope  
101 pictures obtained by Katz et al. [13] showed similar structures for the acid-treated and  
102 untreated soils. According to the literature, acid solutions modify the compaction and bearing  
103 capacity properties of treated soils without modifying the soil mineralogy.

## 104 **2b. Enzymatic solutions**

105 For enzymatic treatments, Rauch et al. [20, 21] concluded that most of the variations in the  
106 Atterberg limits they observed were insignificant compared to the typical variations of such  
107 measurements. Unconfined Compressive Strength (UCS) tests of the treated soils  
108 demonstrated modifications from -30 to +80%, depending on the product and soil tested [18,  
109 28]. Velasquez et al. [32] mentioned an increase in shear strength resistance of 9 to 39% for  
110 two enzymatic treatments but detailed analysis of samples properties showed that  
111 modifications can be attributed to differences in water content and dry density after

112 treatment. For some enzymatic solutions, limited changes in the compaction references were  
113 found [19, 21]. Tingle and Santoni [28] considered that the modifications in the compaction  
114 properties after enzymatic treatments were too small to be significant; however, Velasquez et  
115 al. [32] mentioned that samples treated with enzymatic solutions achieved a higher density  
116 for the same compaction energy. In two of the three field studies reported by Scholen [24],  
117 the treated section of uncovered forest roads showed less degradation than untreated  
118 sections. Visser [34] performed in situ California Bearing Ratio (CBR) measurements on four  
119 soils ranging from sandy to clayey treated with an enzymatic solution. CBR improvement  
120 occurred in 4 cases out of 8. For enzymatic treatment, Scholen [24] hypothesised that  
121 enzymes bond the organic matter of the soil with clay minerals. These additional bonds  
122 should reduce the water affinity of the clay and limit the swelling potential of the soil.  
123 However, Harris et al. [11] showed that the 3-D swelling potential of a plastic clay (PI = 51)  
124 was not modified by the addition of 0.07% of an ES treatment. Another stabilisation  
125 mechanism was suggested by Velasquez et al. [32], who observed that an ES could have  
126 surfactant properties and thus stabilise the soil by improving its compaction ability. This  
127 property was confirmed by Berney et al. [4] and Park et al. [17], who showed that the dry  
128 density of compacted soils is improved by the addition of surfactants. In conclusion, ES  
129 seems to induce compaction modifications, but the stabilisation mechanisms of this family of  
130 products remain in question.

## 131 **2b. Lignosulfonates**

132 The addition of lignosulfonates modified the index properties of treated soils in different  
133 ways. Lambe [14] showed that small quantities of calcium lignosulfonate (< 0.5%) reduce the  
134 liquid limit of a low-plasticity soil. Gow et al. [10] treated a silty sand with 0.25 to 1.0% of  
135 lignosulfonate and observed that the plastic limit was reduced when the dosage increased,  
136 but no consistent trend was found for the liquid limit. A maximum improvement in the UCS

137 was observed for the addition of 5% of lignosulfonates [23, 28]. Compaction properties were  
138 also affected by the treatment. Gow et al. [10] showed that the optimal water content of a  
139 compacted soil was reduced and the maximum dry density increased especially for dosages  
140 between 0.5 and 1.5%. CBR values measured for each dosage at optimal water content and  
141 maximum dry density were improved in comparison to untreated samples but decreased with  
142 increased dosages. In situ evaluations of the mechanical performances of the lignosulfonate  
143 treatment are limited. Surdahl et al. [27] mentioned a stiffness improvement of a granular soil  
144 after treatment. Lignosulfonates are thought to stabilise the soil by coating together its  
145 particles [30, 33]. However, lignosulfonates also have surfactant [31] and dispersive  
146 properties [14, 25]. Thus, the stabilisation mechanism of lignosulfonates is not yet well  
147 understood.

148 In general, soil treatment with non-traditional additives can improve the engineering  
149 properties of soils; however, these results vary depending on the tested products, soil nature  
150 and at times, the dosages. According to Rauch et al. [20] the variability of the engineering  
151 properties may be due to the modification of the optimal density and water content of  
152 compacted soils. Thus, as for traditional treatments such as lime or cement, the addition of  
153 chemical additives may induce modifications of maximum dry density and optimal water  
154 content, which must be determined for accurate field applications. Moreover, the  
155 strengthening mechanisms of non-traditional treatments are not yet well understood and  
156 where data are available, no correlations between microscopic and macroscopic  
157 observations have been established. Therefore, some experiments were conducted to  
158 identify the mechanisms of each additive.



159 **3. MATERIALS AND METHODS**

160 **3a. Non-traditional additives**

161 The acid solution (AS) tested in this study was a concentrated solution of sulfuric acid mixed  
162 with D-limonene, a by-product of citrus processing. For this kind of products, typical dosages  
163 tested in the literature are comprised between 0.01 and 0.2%. As shown by different authors,  
164 UCS, compaction and clay mineralogical properties were not affected by dosage increase  
165 [13, 21, and 28]. Thus, a dosage of 0.01% of concentrated product by dry weight of soil was  
166 tested in this study (Tab. 1).

167 The tested enzymatic solution (ES) is a biodegradable brown aqueous solution with an acidic  
168 pH of 4.6. For this type of product, typical dosages are comprised between 0.002% and  
169 0.1%. However, according to Tingle & Santoni [28] and Velasquez et al. [32], the  
170 geotechnical properties of soils treated with an enzymatic additive were independent of the  
171 dosage. Therefore a dosage of 0.002% was selected.

172 The tested lignosulfonate (LS) was a biodegradable, organic, water soluble calcium salt used  
173 in powder form. The insoluble components were less than 0.5%, the moisture content was  
174 approximately 7% and the reducing sugar content was approximately 7% by dry weight of  
175 product. Experimental results indicated an optimal additive content of 5% [23, 28] for  
176 improving the UCS. On the other hand, Gow et al. [10] had demonstrated that maximum  
177 density and bearing capacity values were obtained for a dosage of 0.5%. For that reason,  
178 three dosages (0.5, 2.0 and 5.0%) were tested.

179 The tested surfactant was a powder with a SDS content of more than 95 % by dry weight.  
180 The required quantity of SDS for a 0.01% dosage was diluted in distilled water and then  
181 added to the soil during mixing operations.

182 **3b Tested soil**

183 A natural fine-graded silt of low plasticity often encountered in earthworks projects in the  
184 Paris Basin was tested in this study (Tab. 2). X-ray diffraction investigations indicated that  
185 the main minerals were quartz and calcite (8.0% by weight determined by calcimetry). The  
186 <2 µm fraction contains quartz and clay minerals (chlorite, illite, kaolinite and smectites) and  
187 the sulphur content of the soil was less than 0.02%.

188 Before testing, the soil was dried at 60°C and pulverised to pass through a 2 mm sieve.  
189 Depending on the liquid or solid nature of the treatment product, a specific protocol was  
190 followed to ensure the application was representative of field application procedures. For the  
191 AS and ES treatments, the initial quantity of water was mixed with the dry soil material to  
192 achieve a water content 3% lower than the target moisture content for compaction. The  
193 samples were placed in a container at 20°C for 24 hours to reach the equilibrium of moisture  
194 content. Then, the additive was mixed with the quantity of water required to obtain the total  
195 desired water content and added during the mixing operation. For LS treatment, the soil was  
196 directly brought to the desired water content and set aside for 24 hours to equilibrate. Finally,  
197 the powder was added to the soil during mixing. For all treatments, a curing time of one hour  
198 in sealed containers was chosen before conducting further tests.

199

## 200 **4. ALTERATION OF GEOTECHNICAL CHARACTERISTICS AFTER TREATMENT**

### 201 **4a. Index properties**

202 The liquid limit (LL) and plastic limit (PL) of the treated soil were determined (Tab. 3). For the  
203 AS, ES and SDS treatments, the Atterberg limits were not modified; however, a reduction in  
204 the LL and PL occurred after the addition of lignosulfonate (LS) at dosages of 2.0 and 5.0%.  
205 Despite these changes in the consistency limits, the plasticity index remained constant for all  
206 tested treatments and dosages.

207 **4b. pH**

208 The pH of the treated soil was determined according to ISO 10390 [12]. Measured values  
209 showed that the pH of the AS and ES treated soil was not affected despite the acidic features  
210 of both products. This phenomenon might be related to the high dilution of the products  
211 combined with the increased buffering capacity of the soil due to the presence of calcium  
212 carbonate. In contrast, the addition of the LS treatment reduced the pH by approximately one  
213 pH unit for the 5.0% dosage. This action is due both to the acidic carboxylic and sulfonate  
214 functions of lignosulfonate and to the high dosages.

215 **4c. Compaction**

216 Compaction tests were performed according to ASTM D 698-91 [1] using a standard Proctor  
217 test. After compaction, the instant bearing index (IPI) was measured following the same  
218 procedure as for the un-soaked Californian bearing ratio (ASTM D 1883-92 [2]) but without  
219 placing surcharge weights. A penetration piston of 49.6 mm diameter is used to apply the  
220 load on the compacted soil. The penetration rate was 1.27 mm/min. The compaction and  
221 instant bearing index curves are plotted in Fig. 1 for AS and ES treated soil. Fig. 2 shows the  
222 results for the addition of LS at 0.5, 2.0 and 5.0%. The optimum water content ( $w_{opt}$ ) and the  
223 maximum dry unit weight ( $\gamma_{dmax}$ ) for each treatment are summarised in Tab. 4.

224 Fig. 1 shows that for the AS treatment, no changes in the compaction and IPI curves  
225 occurred. After ES treatment, the maximum dry unit weight increased and the optimum water  
226 content reduced from 18.2 kN/m<sup>3</sup> and 15.5% to 18.6 kN/m<sup>3</sup> and 14.5%, respectively. On the  
227 dry side of the compaction curve, a maximum increase in the dry unit weight of 0.8 kN/m<sup>3</sup>  
228 was measured. This means that the compaction ability of the treated soil was improved over  
229 a range of water contents from 8 to 15.5%. The IPI curve was also affected by the treatment  
230 and shifted towards lower water contents. Thus, the bearing capacity of the soil was reduced

231 for water contents in the vicinity of  $w_{opt}$ . However, the measured IPI at the respective  
232 optimum water content for both ES treated and untreated soil were roughly equal (Tab. 4).  
233 Fig. 2 shows that the LS treatment increased the dry unit weight and reduced the optimum  
234 water content of the soil for the three dosages. A maximum densification was obtained with  
235 the addition of 2.0% LS, which corresponded to a reduction of 2% in the optimum water  
236 content (13.5% for 2.0% LS treatment as compared to 15.5% for untreated soil). For this  
237 same treatment, dry unit weights were increased on the dry side of the compaction curve and  
238  $\gamma_{dmax}$  increased from 18.2 to 18.5 kN/m<sup>3</sup>. Despite an improvement in density, the addition of  
239 lignosulfonate reduced the IPI at constant water content, and the IPI reduced further with  
240 higher doses of LS. If the soil was compacted at  $w_{opt}$  to reach the maximum dry density, the  
241 IPI measured for the 2.0% LS treatment was 14, compared to a value of 15 measured at  $w_{opt}$   
242 for the untreated soil (Tab. 4). These results illustrate the importance of considering the  
243 entire compaction curve when assessing the effects of ES and LS treatments.

244 After 0.002% ES and 2.0% LS treatments, an increase in dry unit weight and a reduction in  
245  $w_{opt}$  were observed, implying that a minimum dry density may be achieved for lower water  
246 contents. For example, the  $\gamma_{dmax}$  of untreated soil may be achieved at 3% lower water  
247 content. Moreover, both treatments could also reduce the energy needed to compact the soil  
248 to a minimum desired density. An estimation of the potential energy savings was made  
249 according to the results and is presented on Figs 3 and 4. These graphs show the  
250 compaction and bearing capacity curves for the ES and LS treated soil compacted at 100%  
251 and 75% of the standard Proctor energy. For samples compacted on the dry side of the  
252 compaction curve, the measured dry unit weights were the same as for untreated soil despite  
253 the 25% energy reduction. For the ES and LS treatments, however, the bearing capacity of  
254 the soil compacted at 75% of the standard Proctor energy was reduced compared to the  
255 untreated soil.

#### 256 **4d. Unconfined compressive strength**

257 To study the evolution of the mechanical behaviour of treated soil, UCS values were  
258 determined at 7 and 28 days for samples compacted at their respective  $w_{opt}$  and  $\gamma_{dmax}$  after  
259 treatment (Tab. 4). Three replicate samples were statically compacted in one layer in a  
260 cylindrical mould 100 mm high and 50 mm in diameter. Samples were wrapped in a plastic  
261 film, covered by an aluminium sheet and stored in a plastic container at 20°C. Geometrical  
262 parameters, sample weight and water content were controlled after the sample compaction  
263 and just before testing. The results of the UCS tests at 7 and 28 days are presented in Fig. 5.  
264 In comparison with the untreated soil, the resistance of the AS and 2.0% LS treated soil was  
265 not significantly modified. The UCS of ES treated soil was slightly improved due to a higher  
266 maximum dry unit weight obtained after treatment, but the addition of 0.5 and 5.0% of  
267 lignosulfonate reduced the unconfined compressive strength by 20 and 50%, respectively.  
268 Moreover, UCS values only displayed minor changes as a function of curing time, implying  
269 that the treatments mainly have short term effects. For samples compacted at  $w_{opt}$  and  $\gamma_{dmax}$   
270 determined for each treatment after 7 days of curing, the mean modulus for untreated, ES  
271 and 2.0% LS treated soils were respectively 13, 15 and 22 MPa (Tab. 5). Samples were also  
272 compacted at a target water content of 12.5% and a dry unit weight of 18.5 kN/m<sup>3</sup>. The  
273 results showed that only the 2.0% LS treatment significantly reduced the stiffness of the soil.

#### 274 **4e. Swelling potential and hydraulic conductivity**

275 The determination of one dimensional free swell potential of treated soil was carried out on  
276 samples of 70 mm diameter and 15 mm thickness statically compacted in oedometric cells at  
277 different initial compaction states. The samples were saturated with distilled water.

278 Fig. 6 shows the one dimensional swelling potential ( $\Delta H/H_0$ ) for untreated, 2.0% LS and ES  
279 treated samples. For each treatment, at optimal conditions of compaction, the swelling

280 potentials were not significantly modified. Moreover, when the untreated soil was compacted  
281 at the same dry density and initial water content than treated samples, the swelling potentials  
282 were of the same order of magnitude. Therefore, it could be concluded that the studied  
283 products did not significantly alter the swelling potential of the tested soil.

284 After the stabilisation of swelling, the saturated hydraulic conductivities of the samples were  
285 determined (Fig. 7). Treated samples compacted at their respective maximum dry densities  
286 showed a small decrease of the hydraulic conductivity. For treated and untreated samples on  
287 the dry side of the compaction curves, the hydraulic conductivities were very similar.

288 This section showed that the non-traditional additives led to several modifications of the  
289 geotechnical behaviour of the tested material. The modifications did not appear to be time-  
290 dependant since no significant modification of unconfined compressive strength occurred  
291 over the curing period considered. Nevertheless, a key question is the permanence of the  
292 improvement brought by these products over a long period of time. In this context, the  
293 interaction mechanisms between the soils particles and the additives were investigated.

## 294 **5. THE STUDY OF INTERACTION MECHANISMS**

### 295 **5a. Microstructure of compacted soil**

296 The investigation of the microstructure of compacted soil was conducted using a scanning  
297 electron microscope equipped with an energy dispersive X-ray spectrometer (EDS). Images  
298 obtained by collecting the secondary electrons were taken at different magnifications  
299 between 100 and 10,000x. All samples were compacted at their respective optimum water  
300 content and maximum dry unit weight. After compaction, they were wrapped in a plastic film,  
301 covered by an aluminium sheet and stored in a plastic container at 20°C for 28 days. Then,  
302 samples 2 to 3 cm<sup>3</sup> in volume were carefully extracted and freeze-dried to remove the water

303 and minimise sample deformation. Fragments of the freeze-dried samples were extracted  
304 and coated with gold before the SEM analyses.

305 To study the organisation of the internal structure of the compacted soil, SEM investigations  
306 were conducted with mercury injection porosity (MIP) tests. Tests were conducted on freeze-  
307 dried samples compacted at the maximum dry density and optimal water content for each  
308 treatment following the same procedure as for SEM preparation. The pore size distribution is  
309 obtained by derivation of the injected mercury volume ( $V_{inj}$ ) function. To compare the four  
310 samples, the injected volume was normalised such that the surface under the pore size  
311 distribution curve was equal to 1. The void ratio investigated by the mercury ( $e_{inv}$ ) can be  
312 defined as the ratio between the injected volume of mercury and the volume of solids. When  
313  $e_{inv}$  is equal to  $e_{tot}$ , the entire porosity of the sample is filled by mercury. Thus, the calculated  
314  $e_{inv}$  (Tab. 6) reveals that the majority of the existing pores in the samples were filled with  
315 mercury during MIP tests.

316 Fig. 8 shows the surface of the untreated soil. The mineralogical nature of the bigger grains,  
317 generally measuring 10 to 50  $\mu\text{m}$ , was determined by the EDS. These grains were identified  
318 to be quartz associated with smaller clay minerals of 1 to 5  $\mu\text{m}$  diameter. No preferential  
319 orientations of the clay minerals were detected. MIP results indicated that the pore size of  
320 untreated soil presents a uni-modal distribution with a dominant radius of 0.6  $\mu\text{m}$ .

321 Results obtained by SEM showed a slightly more aggregated structure and a face to edge  
322 organisation of clay minerals for the soil treated with 0.01% AS (Fig. 9). Individual clay  
323 minerals are less visible but inter-aggregates pores size remained similar after treatment as  
324 confirmed by MIP data (Figs 12 and 13). Therefore, the structure of the treated soil was not  
325 significantly altered in this case.

326 After 0.002% ES treatment, the same structure as for the untreated soil is observed from the  
327 SEM images (Fig. 10). The MIP study (Fig. 12) showed that less mercury was injected in the  
328 ES treated sample. This phenomenon is linked with the higher dry unit weight of the ES  
329 treated sample, which reduced its total void ratio ( $e_{tot}$ ) and thus the total injected volume  
330 ( $V_{inj}$ ). This result is associated to a reduction of the amount of pores comprised between  
331 3 and 10  $\mu\text{m}$  (Fig. 13).

332 Fig. 11 shows the soil after the addition of 2.0% of lignosulfonate. In the picture, additional  
333 structures can be observed. These structures resemble fibres and are probably  
334 lignosulfonate associations forming a network between the soil minerals (arrows). Point 1 on  
335 Fig. 11 was identified by the EDS to be a clay mineral, but as the fibres were too small to be  
336 analysed by the EDS, their exact nature is unknown. The SEM results also showed larger  
337 inter-aggregates pores, large clay minerals were organised in face to face structures  
338 orientated in the plane of the picture. Compared to untreated samples, MIP results confirmed  
339 the increase of the frequency of pores having radii between 0.3 and 5  $\mu\text{m}$  and also showed a  
340 reduction of pores with a radius  $<0.3 \mu\text{m}$ . The increase in larger pores radius might be  
341 caused by aggregation of clay mineral and dispersion of the created structure due to  
342 adsorption of lignosulfonates molecules. The reduction in intra-aggregates pores may be  
343 explained by the clogging of the smallest pores by lignosulfonates.

#### 344 **5b. Comparison of the treatment additives with a common surfactant**

345 Some authors, for example, Velasquez et al. [32] demonstrated that enzymatic solutions  
346 could have surfactant properties. As enzymatic solutions, lignosulfonates also have  
347 surfactant properties, which could alter the mechanical behaviour of soils [10, 31]. To  
348 determine if the tested non-traditional additives have surfactant properties, the surface  
349 tension was measured using the Du Noüy ring method [9]. The results were compared with



350 the surface tensions obtained for aqueous solutions of sodium dodecyl sulfate (SDS)  
351 (Fig. 14).

352 Similar to SDS, low dosages of the enzymatic solution reduced the surface tension of the  
353 water. At a concentration of 1.0 g/L the surface tension was reduced by more than 50%.  
354 Lignosulfonate also exhibited surfactant properties, but the additive was less efficient in  
355 reducing the surface tension of the solution. At higher concentrations, the surface tension of  
356 the LS solution stabilised approximately 42 mN/m. Only the acid additive did not exhibit any  
357 surfactant properties.

358 To study the influence of the addition of a surfactant on the compaction behaviour, 0.01% of  
359 SDS by dry matter weight was added to the soil (Fig. 15). The required quantity of SDS was  
360 first diluted in water, and the same protocol used for liquid non-traditional treatments was  
361 followed. The resulting compaction and IPI curves were compared to those obtained after the  
362 ES treatment.

363 With regard to the ES treatment, the SDS addition improved the dry unit weight on the dry  
364 side of the compaction curve. The IPI curve for the SDS-treated soil was also slightly shifted  
365 towards smaller water contents as was observed after the ES-treatment. Thus, the SDS and  
366 ES treatments have a similar effect on the mechanical behaviour of soils. This finding implies  
367 that both products may utilise the same stabilisation mechanism. Indeed, the reduction of the  
368 surface tension of the soil's interstitial water should lower the matrix suction according to the  
369 Jurin law.

370 The suction of the treated soil was investigated for the three non-traditional treatments. The  
371 filter paper methodology was used to measure matrix suction of compacted soil according to  
372 ASTM D 5298-94 [3]. After compaction of two 70 mm diameter for 15 mm thickness samples,  
373 three filter papers were inserted between each sample. Thereafter, the samples were kept in

374 sealed container for 7 days to achieve the suction equilibrium. As the calibration curve of the  
375 filter paper was known, the measurement of its water content gave the matrix suction of the  
376 soil.

377 The results showed that the acid solution (AS) did not change the suction of the soil. This is  
378 consistent with the absence of surfactant features of the additive.

379 As ES treatment has surfactant properties, a reduction of the suction could be expected. In  
380 this work, the addition of the enzymatic solution (ES) did not lead to suction modifications  
381 (Tab. 7). This can be explained by the biodegradability of the additive. An equilibrium period  
382 of seven days may allow the additive to be totally degraded before suction measurements.

383 The LS treatment led to an increase in the matrix suction of the soil. As LS is an organic  
384 calcium salt, the increase of the suction might be explained by the hygroscopic nature of  
385 lignosulfonates.

## 386 **6. DISCUSSIONS**

387 Herein, the effects, mechanisms and applications of the three studied treatments are  
388 discussed.

389 AS treatment did not change geotechnical properties of the soil but small modifications in the  
390 clay minerals organisation were observed in SEM pictures (Fig 9). The observed  
391 modifications could be due to changes in the ionic composition of the pore fluid after addition  
392 of the product since this parameter is known to induce fabric changes according to the  
393 literature [6-8, 15, 16]. No particular application was evidenced for AS treatment of the  
394 studied soil.

395 Treatment with ES demonstrated an increase in the compaction ability of the soil. The  
396 increase in maximum dry density induced small modifications of the UCS, stiffness, swelling

397 potential and permeability of the soil. Moreover, the microstructure of the soil was not  
398 modified. Therefore, it can be concluded that the observed hydro-mechanical modifications  
399 are mostly related to the effect of treatment on compaction behaviour of the soil. This  
400 hypothesis was confirmed by the absence of pH and index properties changes. As shown on  
401 the Fig. 15, the compaction behaviour of the ES treated soil is similar to the behaviour  
402 observed after a treatment with a surfactant. Results shown on Fig. 3 indicated that treated  
403 soil had a smaller bearing capacity than the untreated soil at given water content and dry  
404 density. This phenomenon may be explained by the surfactant properties of the ES. Strong  
405 modifications of the surface tension for low additive dosages may also explain why the  
406 compaction ability of the ES treated soil was improved despite a low dosage (0,002%). As  
407 the enzymatic solution is biodegradable, the product has disappeared in a few days. During  
408 the degradation stage of the ES product, increase in bearing capacity may be expected and  
409 after degradation of the additive, no long term modifications are expected beyond those  
410 caused by the initial densification. According to the experimental results, the ES treatment  
411 can be used to increase the compaction ability on the dry side of the compaction curve and  
412 thus compact the soil at higher densities for lower water contents.

413 LS treatment reduced the pH and modified the pore-fluid composition, thus, the organisation  
414 and interactions between soil particles were changed as shown by mechanical results and  
415 SEM pictures. Experimental results showed dosage dependent effects on geotechnical  
416 characteristics. The 2% amount of lignosulfonate added to the soil also reduced the stiffness  
417 and the index properties. This observation is consistent with dispersive properties of  
418 lignosulfonates which tend to increase repulsive interactions between soil constituents [5, 14,  
419 25]. LS treatment also reduced the friction between the grains of the soil, which facilitate its  
420 deformation under loading, and thus explain the observed reduction in bearing capacity  
421 (Figs. 2 and 4). However, surfactant properties and suction modification after treatment  
422 cannot explain alone why the bearing capacity was reduced by an increase in the

423 lignosulfonate dosage (Fig. 2). This observation is probably due to the formation of an  
424 organic film increasing lubrication between the particles after the addition of lignosulfonate  
425 [9]. An increase in dosage led to thicker films, reducing the bearing capacity of the LS treated  
426 soil by facilitating the deformation under loading. As for ES, addition of the product modified  
427 the behaviour of the soil during the compaction. Nevertheless, field and laboratory testing  
428 confirmed the ability of lignosulfonate to be leached out of the soil [11, 22, 27]. So, it is to be  
429 expected that the soil will recover its initial characteristics after a certain period of time. The  
430 uses of LS treatment are similar to those of ES but higher dosages are needed and attention  
431 must be paid on bearing capacity which is altered by the treatment.

## 432 **7. CONCLUSION**

433 This paper presented an experimental multi-scale study of a low-plasticity soil treated with  
434 three non-traditional organic products classified as an acid solution, an enzymatic solution  
435 and lignosulfonate. Hydro-mechanical investigations including compaction, bearing capacity,  
436 UCS, free swelling and hydraulic conductivity tests were conducted. The effect of the  
437 treatments on the microstructure of the compacted soil was investigated using SEM and MIP  
438 tests. The study also proposed a reflexion on the modification of the interactions between soil  
439 particles induced by the treatments. The following conclusions can be drawn based on the  
440 obtained results.

- 441 - To evaluate the performances of non-traditional products used for soil treatment, it is  
442 important to consider the entire compaction and bearing capacity curves and not only  
443 the values obtained at the optimal conditions. In fact, the water content of compacted  
444 soil has a strong influence on the dry unit weight obtained after treatment.
- 445 - The ES and LS treatments reduced the optimal water content and increased the dry  
446 density of the compacted soil allowing the use of 3% lower water content and a 25%

447 reduction in the compaction energy, required to achieve the same density when  
448 compared to untreated soil. Therefore, the treatments can improve the environmental  
449 impact of earthwork projects and are particularly well suited for dry soil compaction in  
450 areas where the access to water resources is limited.

451 - Before in situ applications, careful laboratory investigations should be conducted to  
452 determine if the treatment genuinely improves the soil behaviour; some non-  
453 traditional products have little or no impact on the mechanical properties of treated  
454 soils. The changes in compaction parameters must be characterised to determine the  
455 optimal application conditions in terms of dosage, desired water content and bearing  
456 capacity.

457 - Treatments with organic products have showed immediate modification of the  
458 compaction parameters; however, no evolution of the resistance of the soil over time  
459 was observed. Thus, treatments essentially act through a physical modification of the  
460 soil behaviour. Moreover, as AS and ES were added at low dosages and were  
461 biodegradable, intrinsic characteristics of the soil are not changed and the long term  
462 behaviour of the soil should not be modified. For LS treatment, leaching of the  
463 additive can be expected in case of water transfer in the compacted structure.

464 - Some non-traditional products act like surfactants. This property appears to be an  
465 important parameter of the effectiveness of non-traditional products when they are  
466 used as compaction aids.

## 467 **ACKNOWLEDGMENTS**

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1 **Tab. 1** Dosages tested for the three non-traditional additives

2

Non-traditional additives	Dosages by dry weight of soil
Acid solution (AS)	0.01%
Enzymatic solution (ES)	0.002%
Calcium lignosulfonate (LS)	0.5 ; 2.0 and 5.0%

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4

5 **Tab. 2** Properties of the tested soil

6

Property	Low plastic clay
Passing 2 mm (%)	100
Passing 80 µm (%)	87
Passing 2 µm (%)	19
Specific gravity	2.67
Liquid limit (%)	34
Plastic limit (%)	20
Plasticity index	14
pH	9.2

7

8

9 **Tab. 3** Index properties and pH of the treated soil

10

Treatment	pH (+/- 0.1)	PL (%)	LL (%)	PI
Untreated	9.2	20	34	14
0.01% AS	9.2	19	33	14
0.002% ES	9.3	19	33	14
0.5% LS	8.8	19	32	13
2.0% LS	8.4	17	30	13
5.0% LS	8.1	13	27	14
0.01% SDS	9.1	19	34	15

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14 **Tab. 4** Geotechnical properties of the treated soil

15

Treatment	$w_{opt}$ (%)	$\gamma_{dmax}$ (kN/m <sup>3</sup> )	IPI at $w_{opt}$	UCS (MPa)	
				7 days	28 days
Untreated	15.5	18.2	15	0.44	0.46
0.01% AS	15.0	18.3	19	0.40	0.41
0.002% ES	14.5	18.6	14	0.53	0.54
0.5% LS	15.0	18.4	13	0.36	0.40
2.0% LS	13.5	18.5	14	0.45	0.49
5.0% LS	14.0	18.3	5	0.22	0.24

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19 **Tab. 5** Deformation modulus at 50% of maximal strength

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Treatment	w (%)	$\gamma_d$ (kN/m <sup>3</sup> )	E <sub>50</sub> (MPa)	Standard deviation (MPa)
Untreated	15.0	18.2	13	2
	12.5	18.4	56	7
0.002% ES	14.0	18.5	15	1
	12.5	18.3	50	4
2.0% LS	12.7	18.5	22	2

21

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24 **Tab. 6** Total void ratio for the compacted samples and calculated mercury investigated  
25 maximum void ratio during the MIP tests

26

Treatment	$e_{tot}$	$e_{inv}$	$\gamma_d$ (kN/m <sup>3</sup> )
Untreated	0.47	0.44	18.3
0.01% AS	0.46	0.44	18.3
0.002% ES	0.43	0.42	18.7
2.0% LS	0.44	0.41	18.6

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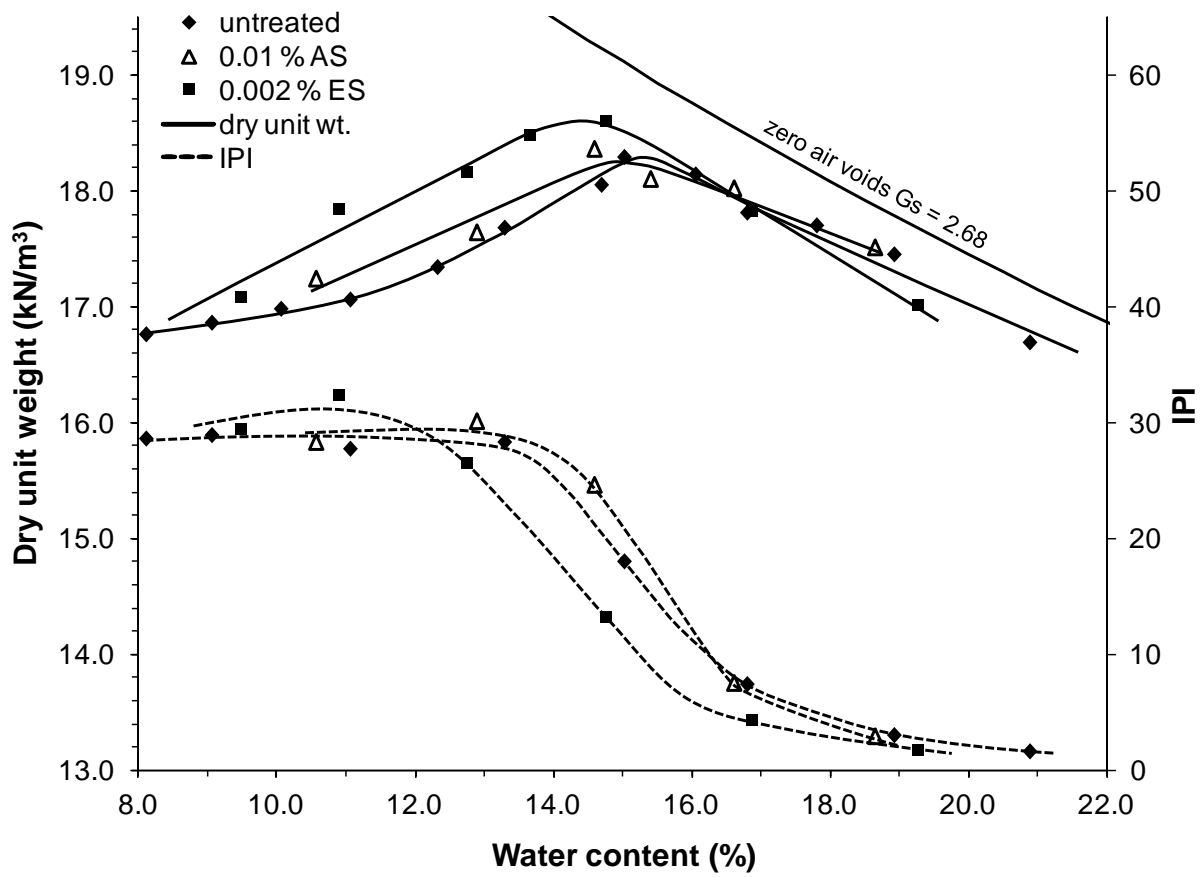
29 **Tab. 7** Matrix suction of treated soil

30

Treatment	w (%)	$\gamma_d$ (kN/m <sup>3</sup> )	Matrix suction (kPa)
Untreated	14.7	18.0	56
0.01% AS	15.4	18.0	54
0.002% ES	14.7	18.2	58
2.0% LS	15.2	18.2	225

31





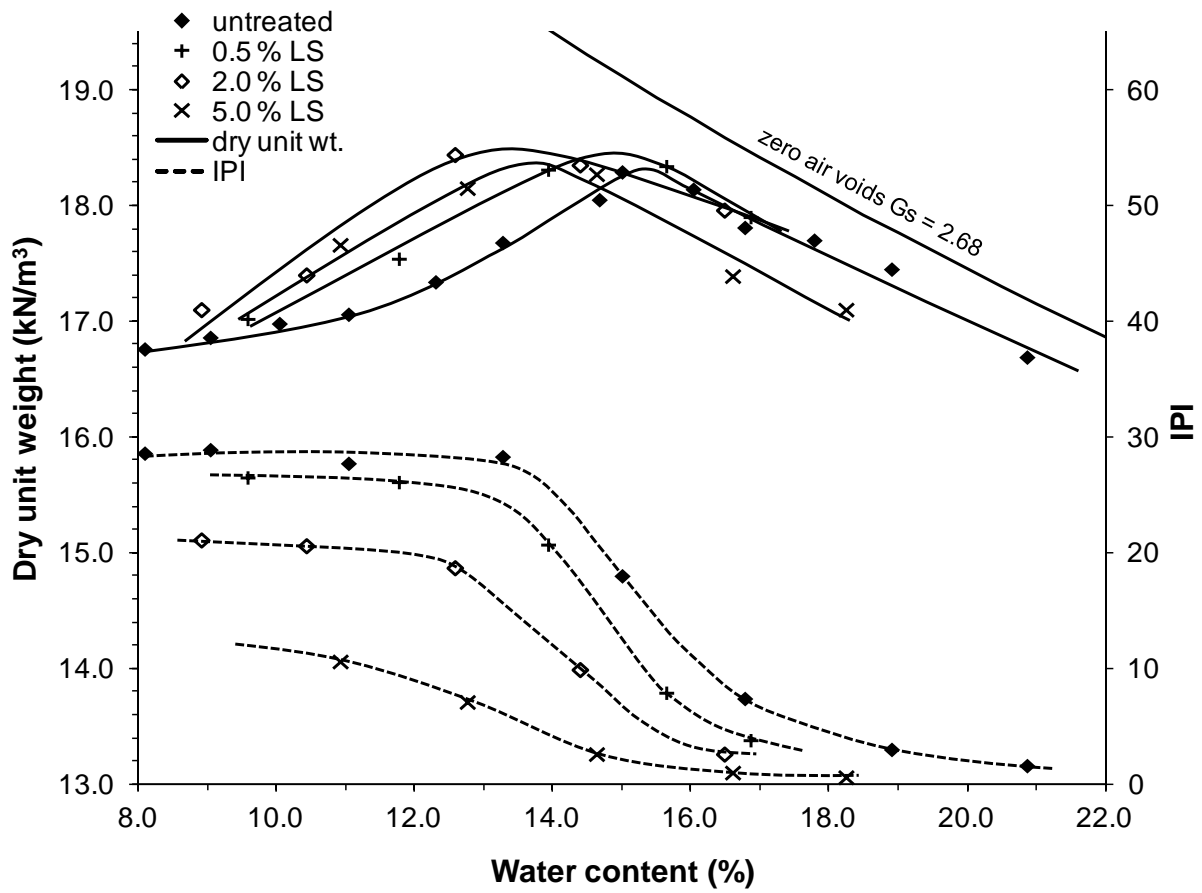
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2 **Fig. 1** Compaction and IPI curves for the untreated, 0.01% AS and 0.002% ES treated soil

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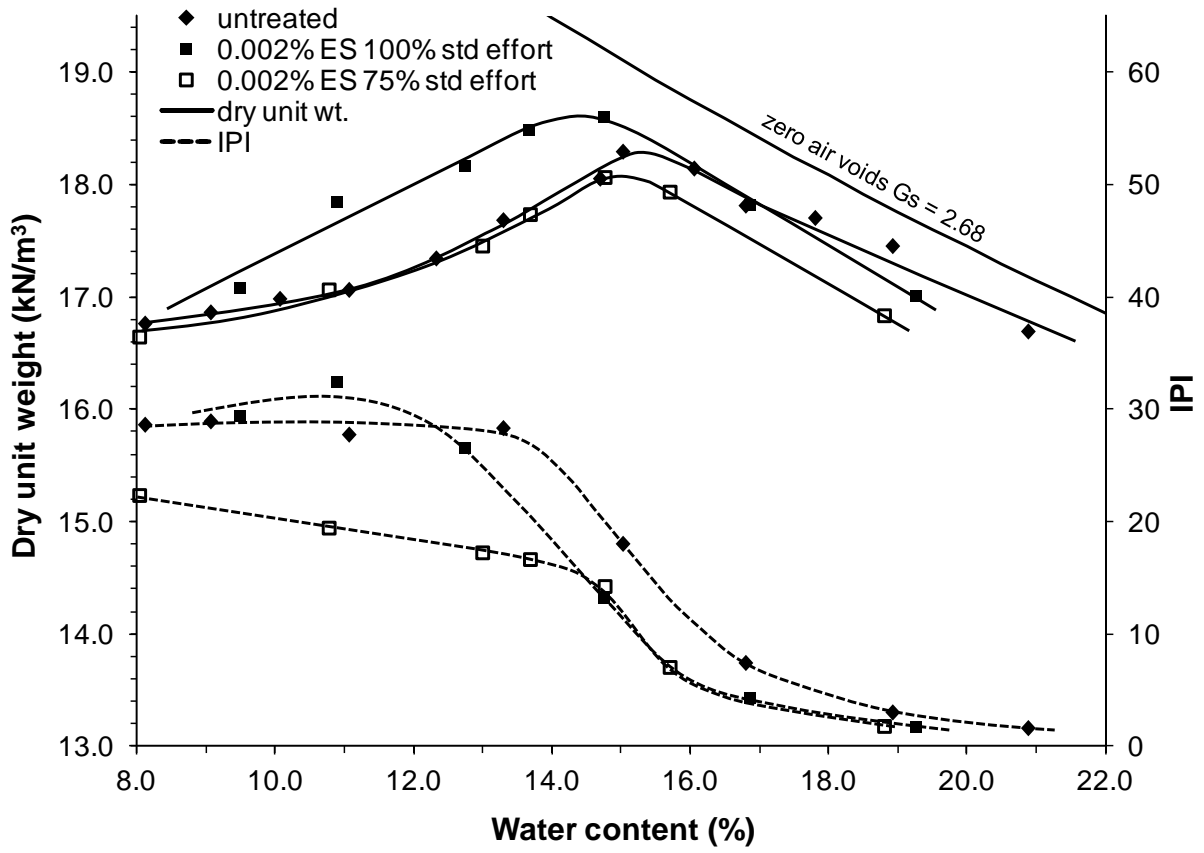
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7 **Fig. 2** Compaction and IPI curves for the LS treatment at 0.5, 2.0 and 5.0%

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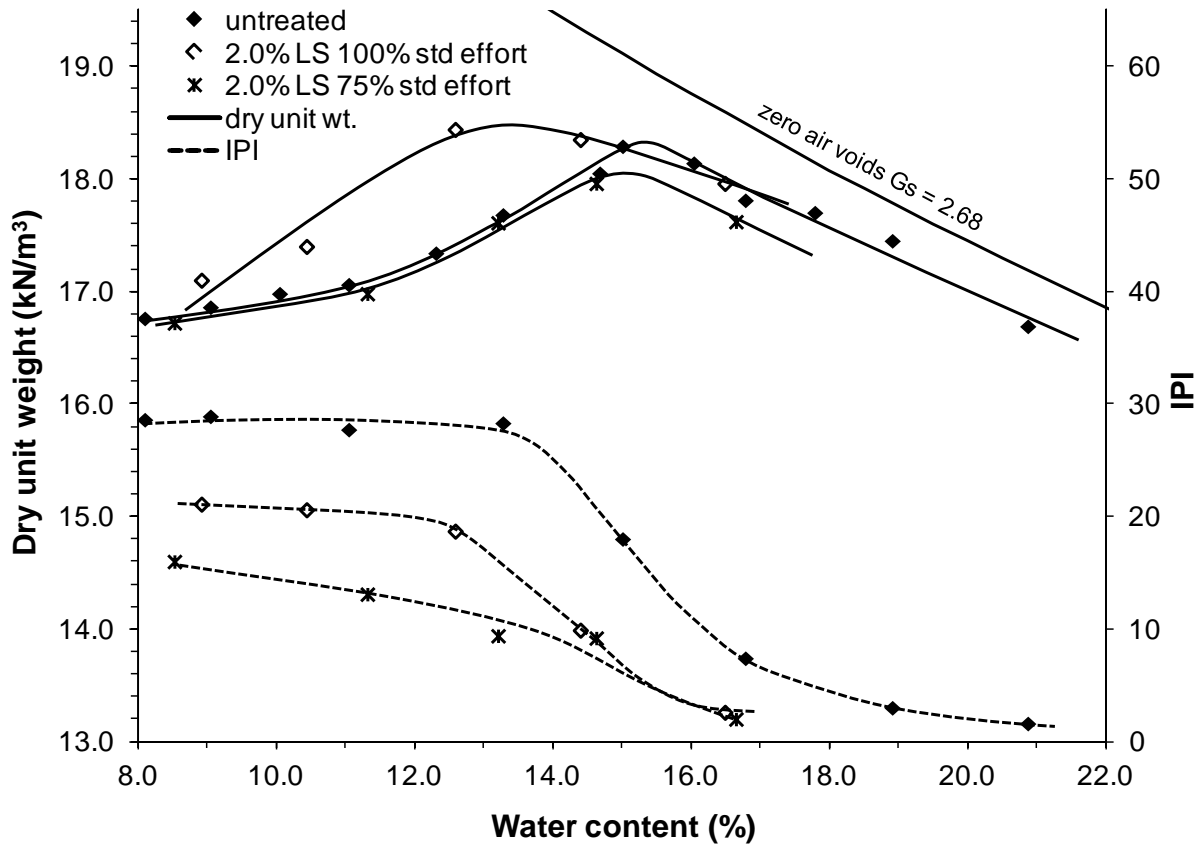


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13 **Fig. 3** Compaction and IPI curves of the ES treatment at 75 and 100% of the standard  
14 Proctor energy

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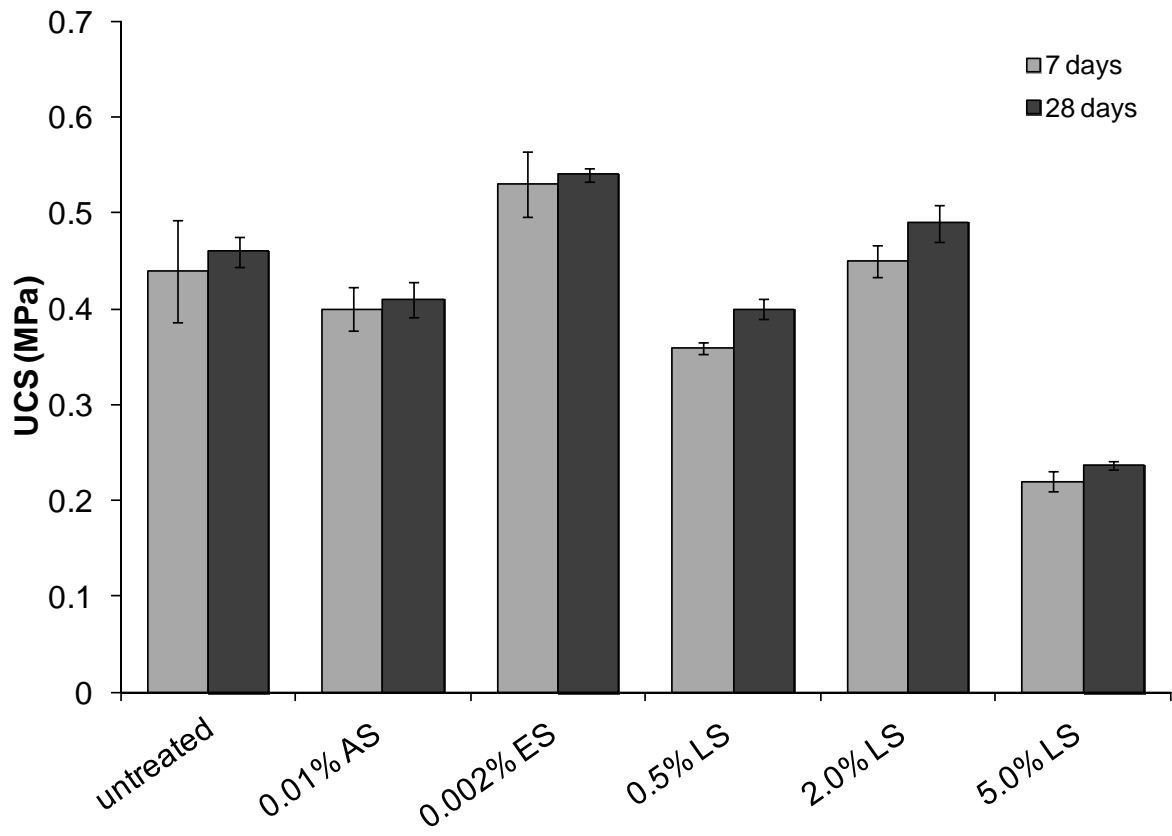
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18 **Fig. 4** Compaction and IPI curves of the 2.0% LS treatment at 75% and 100% of the  
 19 standard Proctor energy

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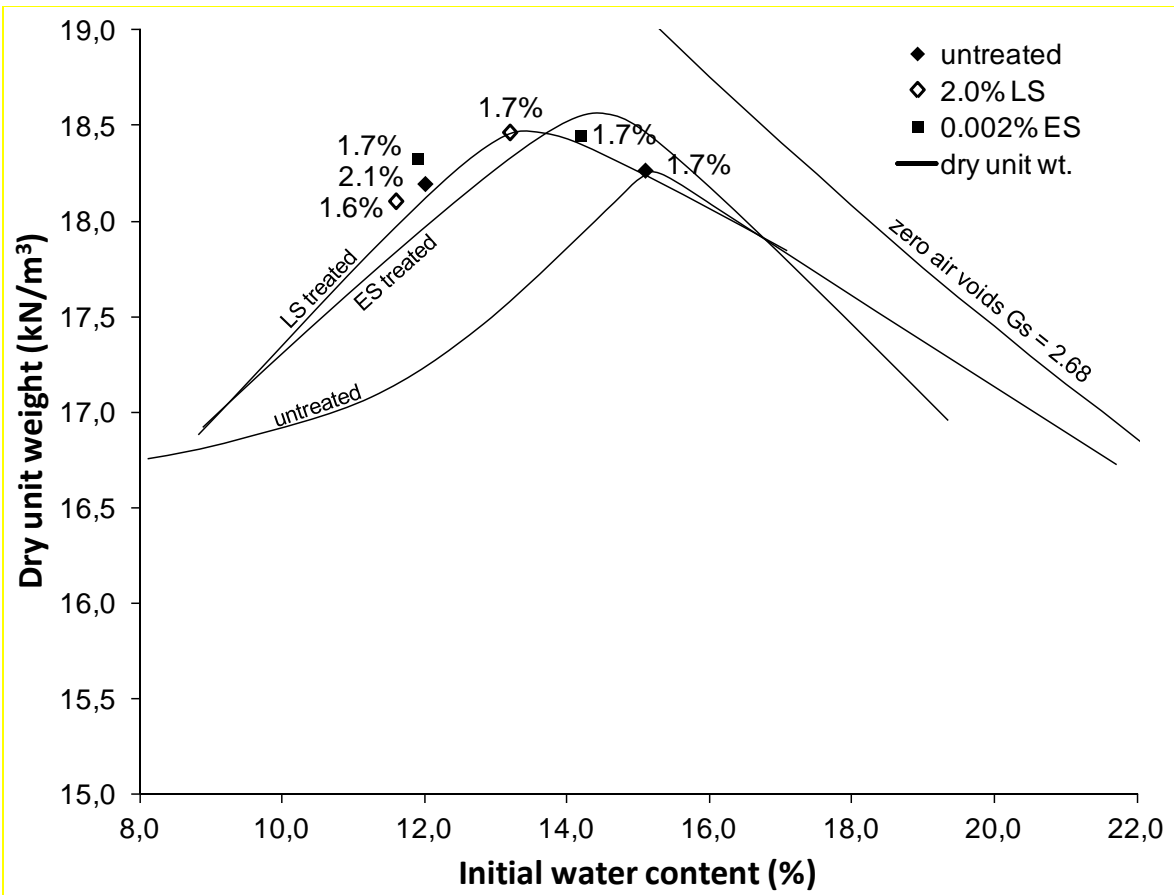
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24 **Fig. 5** Unconfined compressive strength at 7 and 28 days for the studied non-traditional  
 25 treatments compacted at their respective optimum Proctor

26

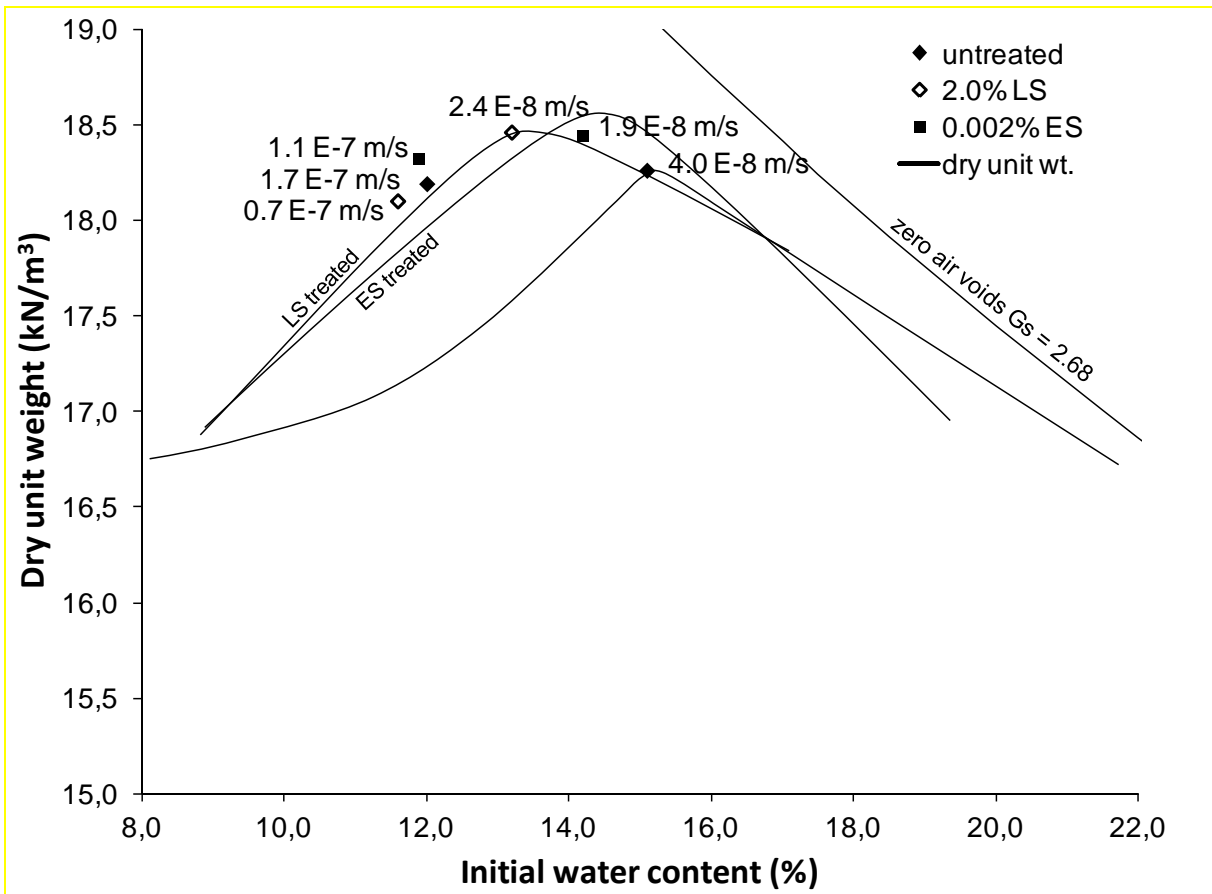


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28 **Fig. 6** One-dimensional free swelling potential for the ES and LS treatments at different initial  
 29 compaction states

30

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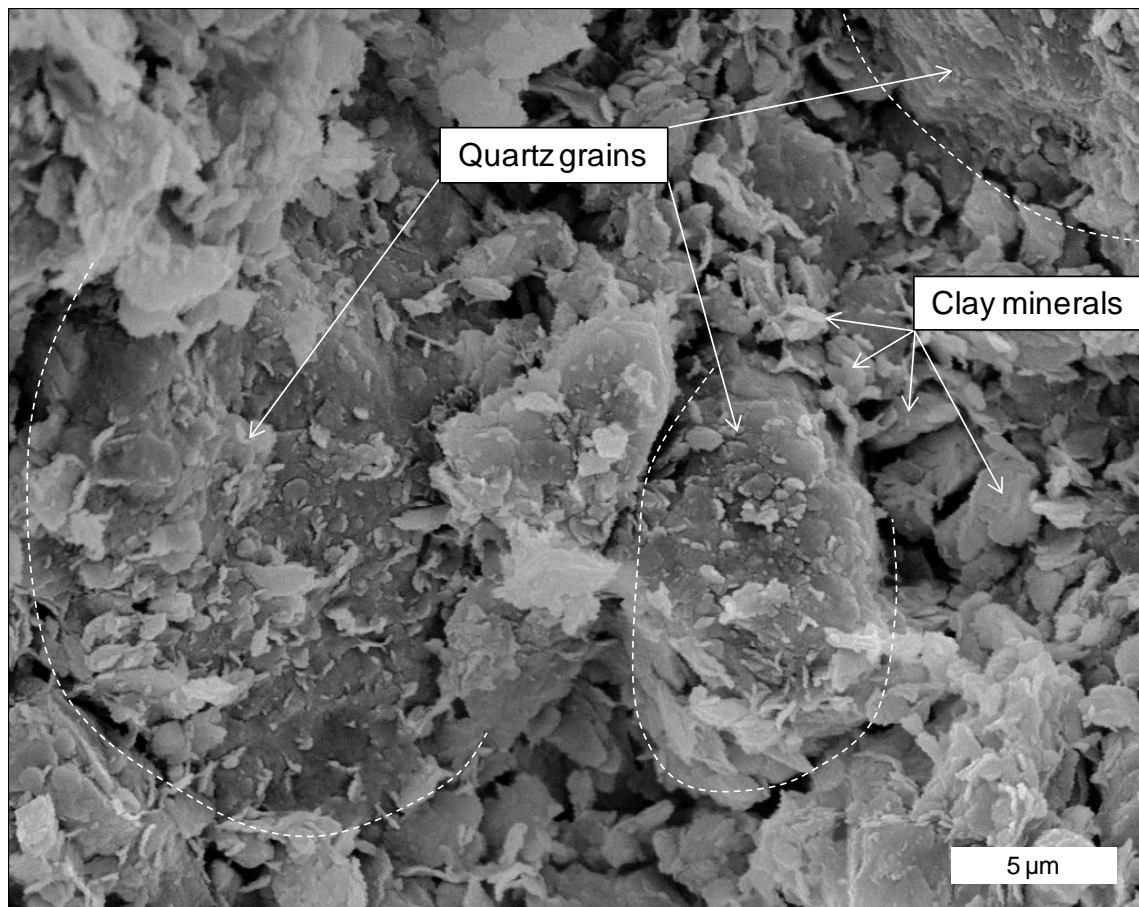


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34 **Fig. 7** Hydraulic conductivity coefficient for the ES and LS treatments in different initial  
 35 compaction states

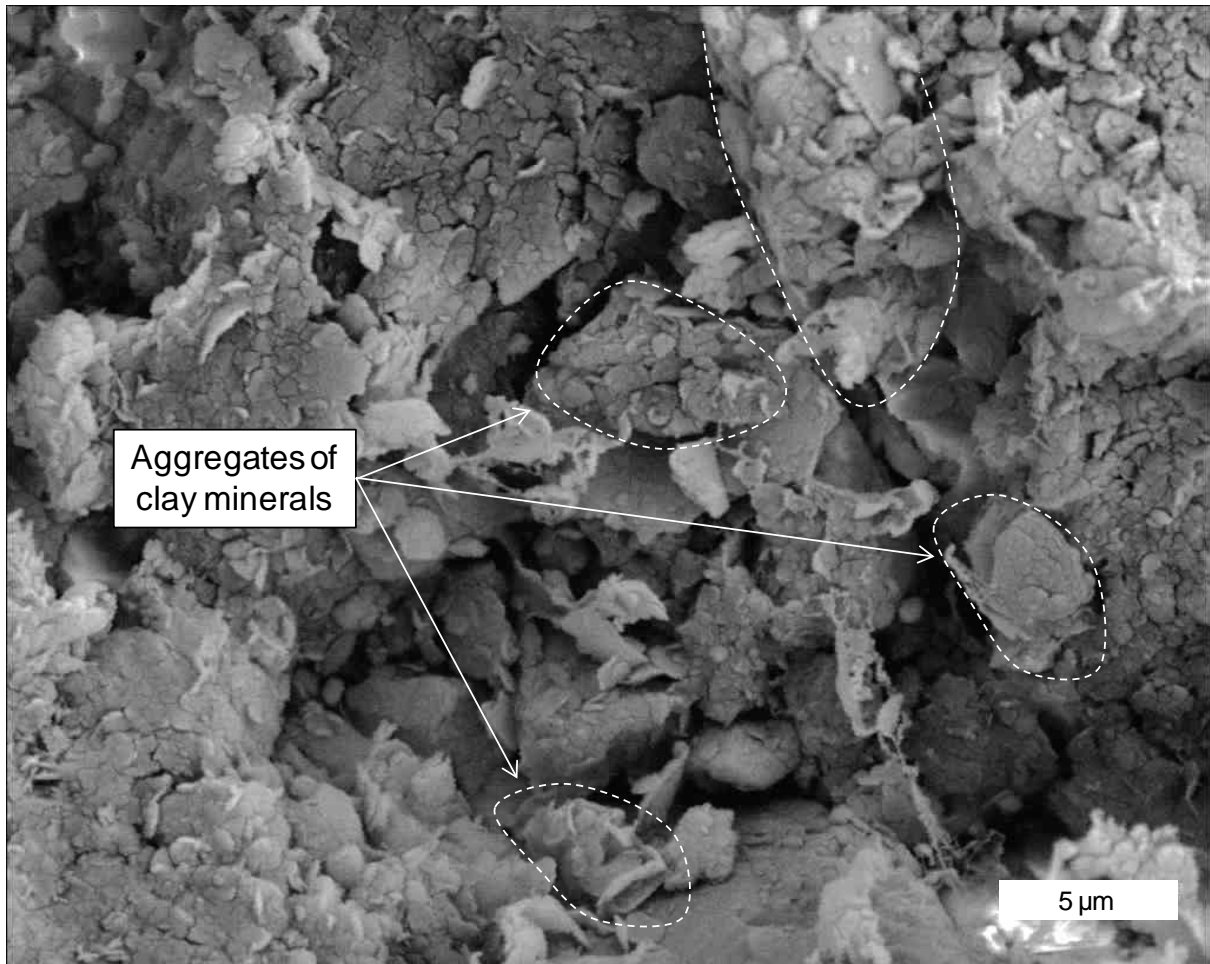
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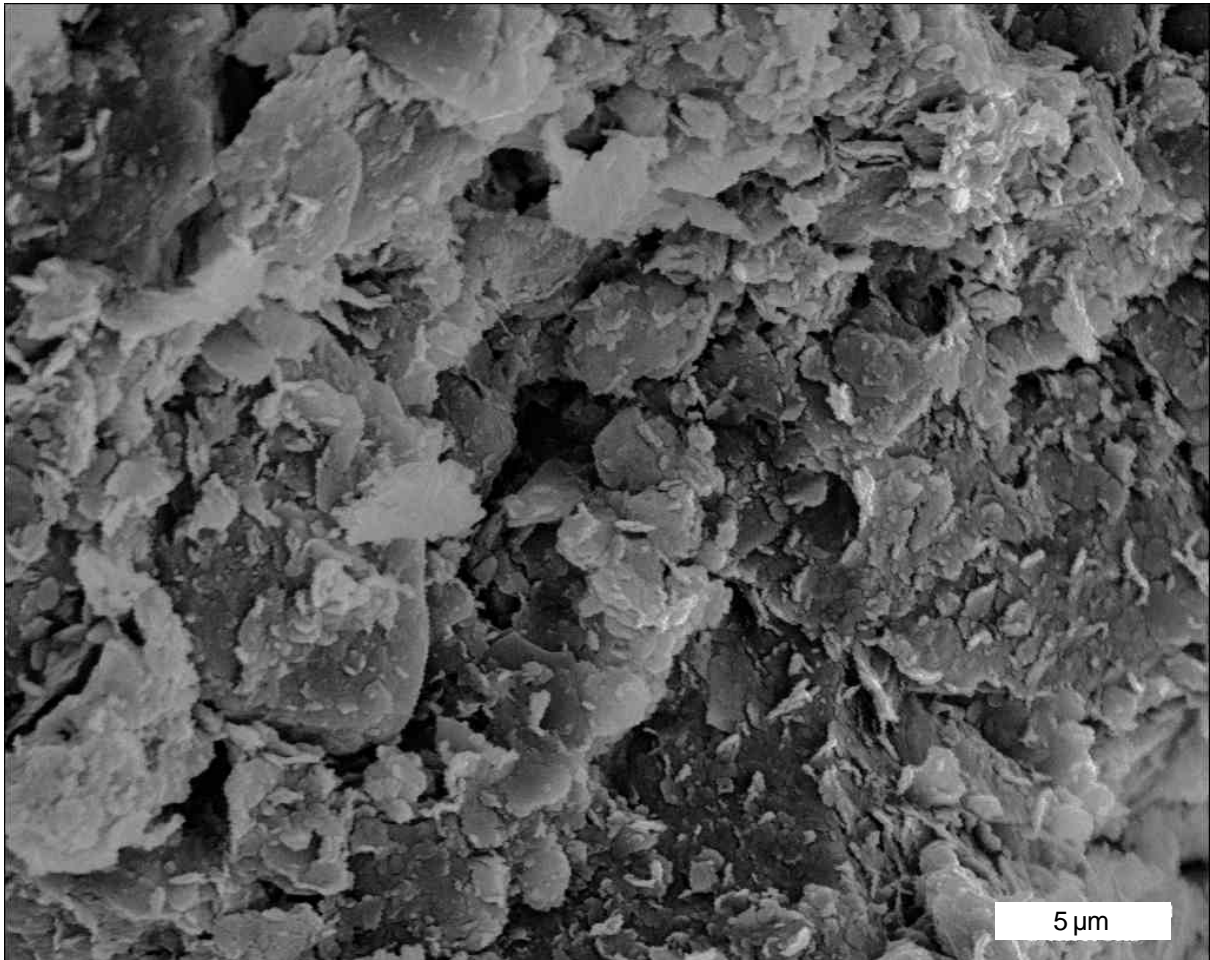
40 **Fig. 8** SEM image of the untreated soil





45 **Fig. 9** SEM image of the 0.01% AS treated soil

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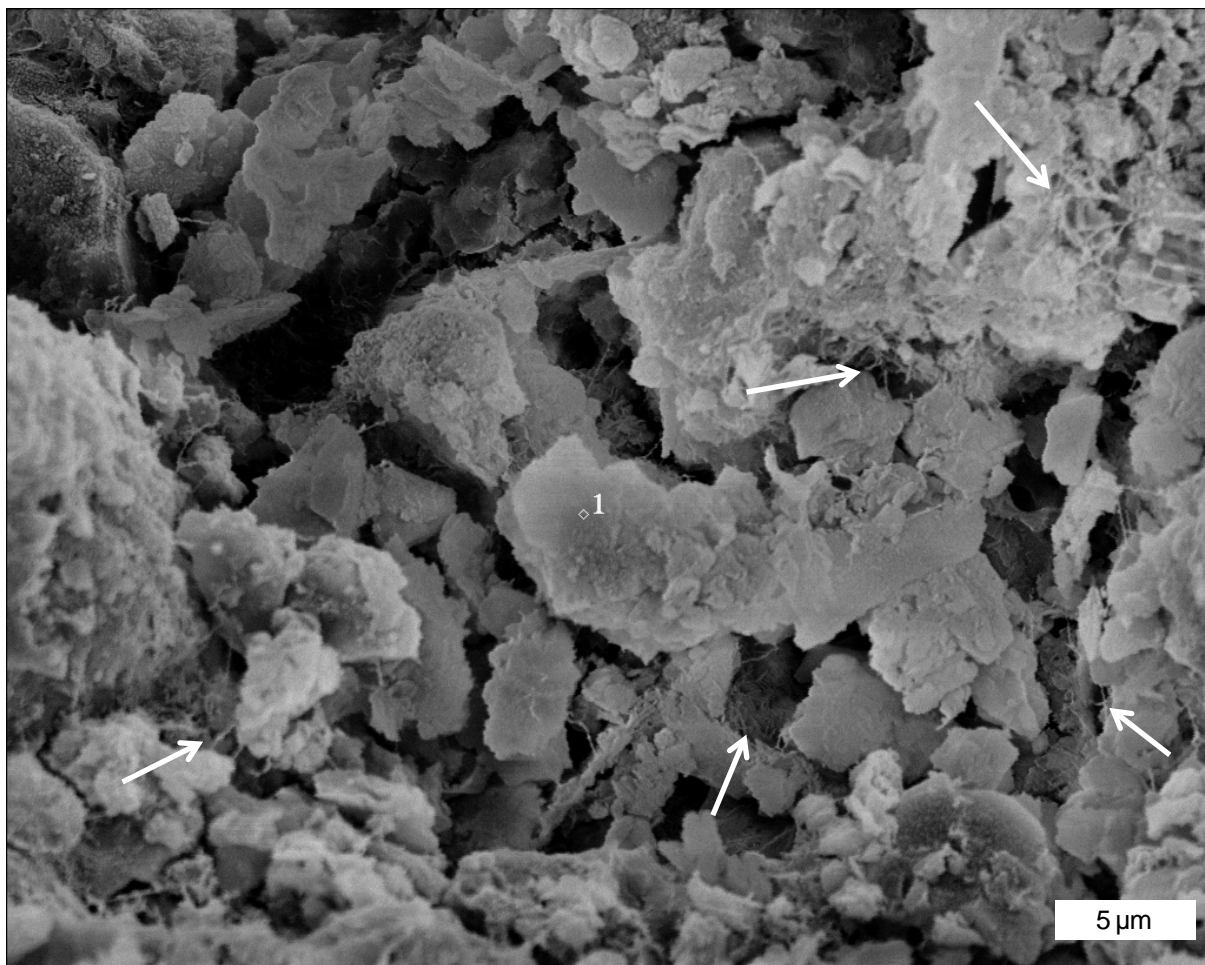
49 **Fig. 10** SEM image of the 0.002% ES treated soil

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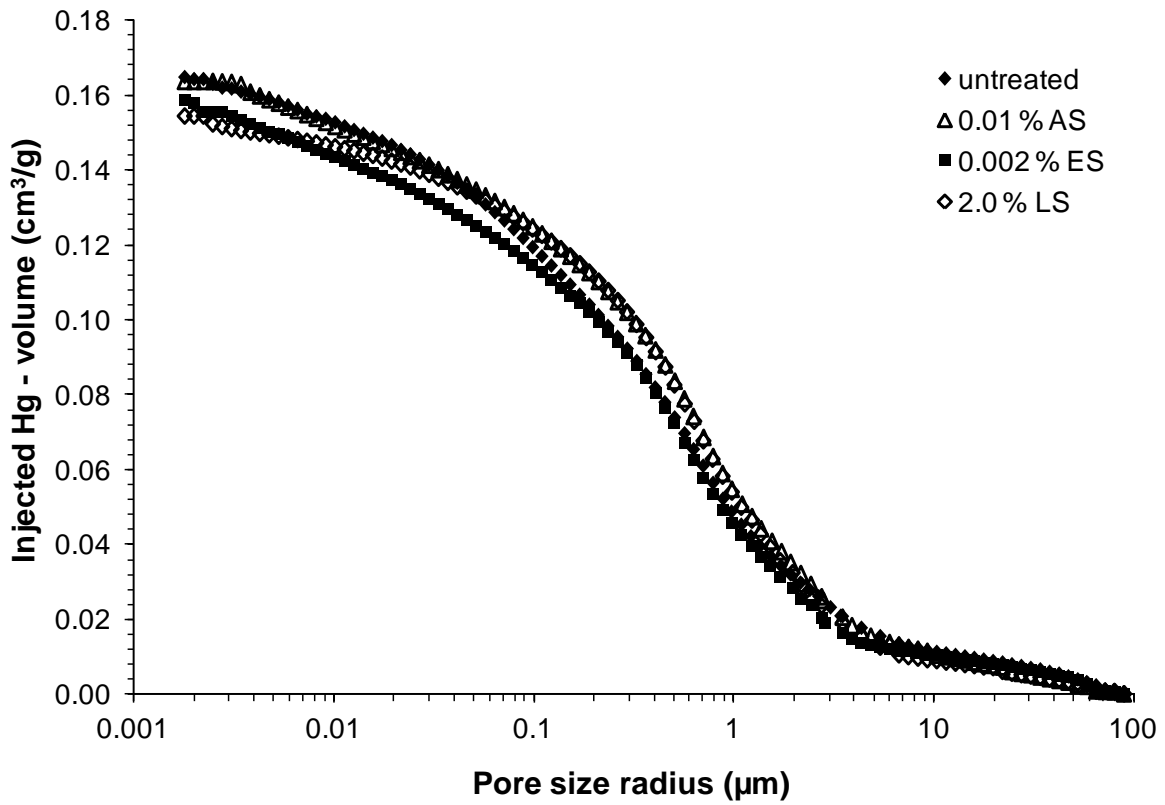


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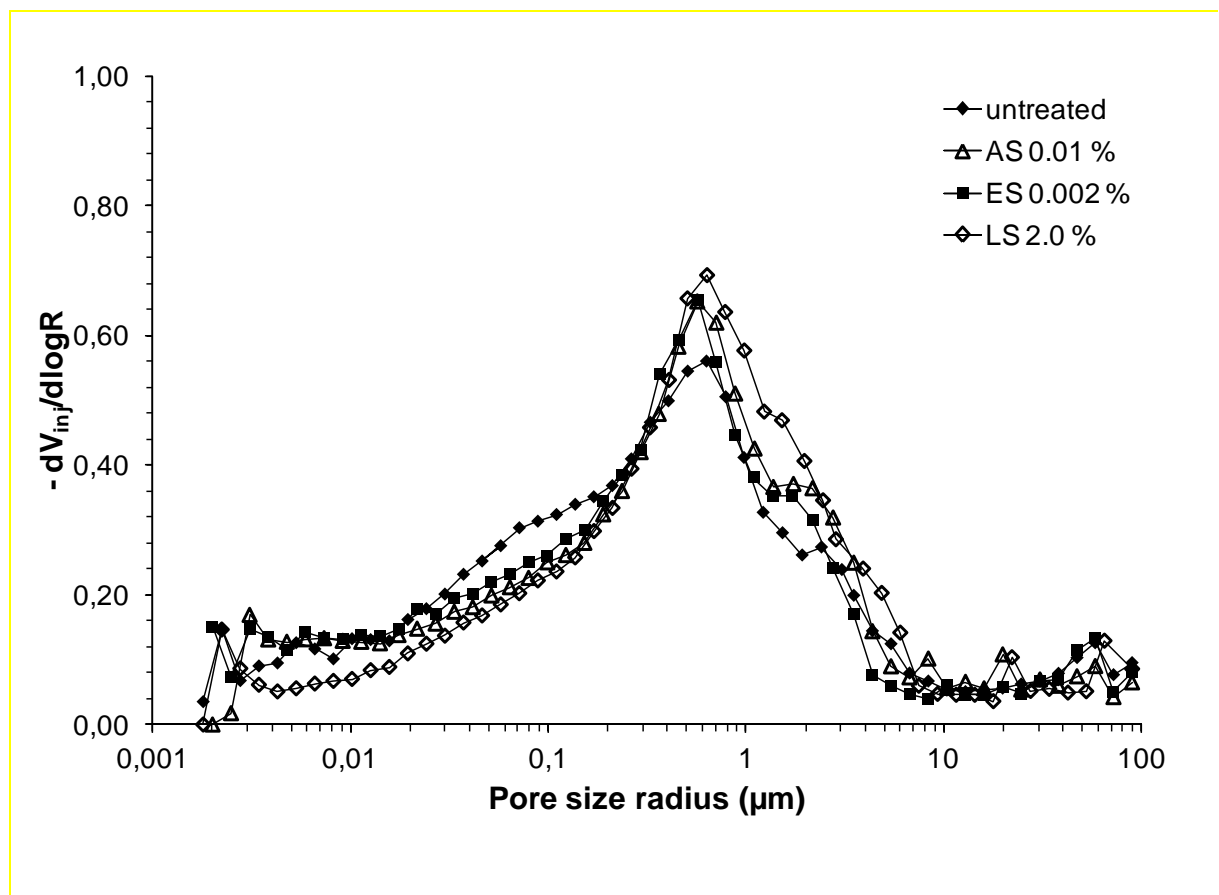
55 **Fig. 11** SEM image of the 2% LS treated soil

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57



60 **Fig.12** Injection curves for the untreated, 0.01% AS, 0.002% ES and 2.0% LS treated soil



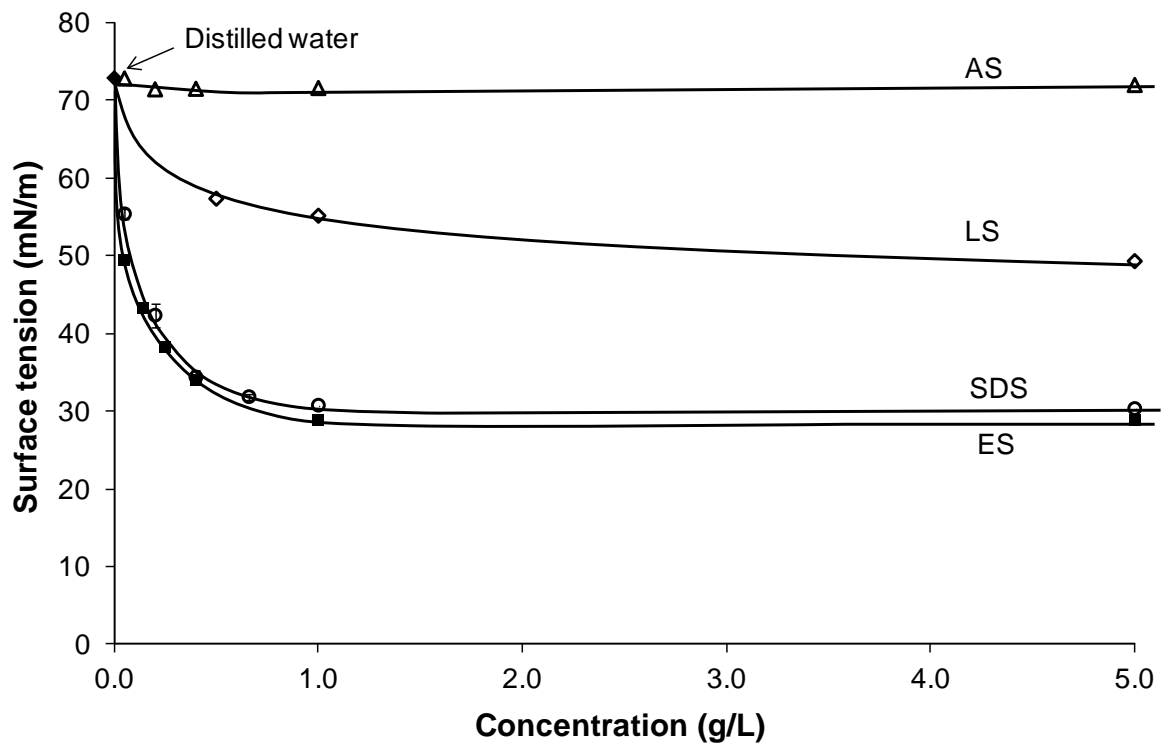
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64 **Fig. 13** Pore size distribution for the untreated, 0.01% AS, 0.002% ES and 2.0% LS treated

65 soil

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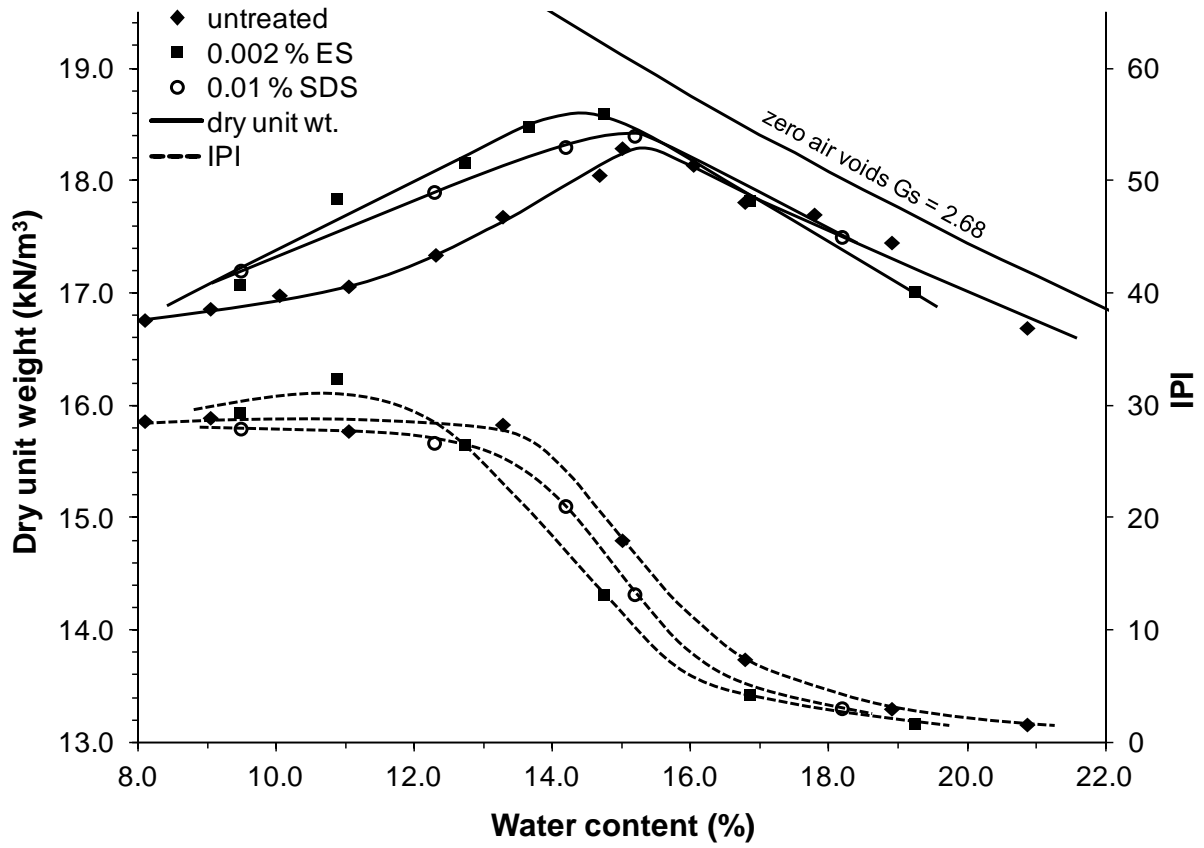
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69 **Fig. 14** Surface tension of the three non-traditional additives and SDS in aqueous solution

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74 **Fig. 15** Compaction and IPI curves for 0.01% SDS and 0.002% ES treated soil

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