



**HAL**  
open science

## Solidification Study of an Organic Fine Sediment Using Geopolymer Binders Properties

Ishak Moghrabi, Harifidy Ranaivomanana, Fateh Bendahmane, Ouali Amiri,  
Dimitri Deneele

► **To cite this version:**

Ishak Moghrabi, Harifidy Ranaivomanana, Fateh Bendahmane, Ouali Amiri, Dimitri Deneele. Solidification Study of an Organic Fine Sediment Using Geopolymer Binders Properties. International Journal of Geomechanics, 2023, 23 (10), 10.1061/IJGNALGMENG-8187 . hal-04193796

**HAL Id: hal-04193796**

**<https://hal.science/hal-04193796>**

Submitted on 6 Sep 2023

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial - NoDerivatives 4.0  
International License

# Solidification study of an organic fine sediment using geopolymer binders properties

Ishak MOGHRABI, PhD. <sup>1</sup>; Harifidy RANAIVOMANANA, PhD. <sup>2</sup>; Fateh BENDAHMANE, PhD. <sup>3</sup>; Ouali AMIRI, PhD. <sup>4</sup>, Dimitri DENELEE, PhD. <sup>5</sup>

<sup>1</sup> Structural design engineer, Chabanne Architecte, 38 Quai de Pierre-Scize, 69009 Lyon, France.

Email: moghrabi.ishak@gmail.com

<sup>2</sup> Associate Professor, Nantes Université, Ecole Centrale Nantes, CNRS, GeM, UMR 6183, F-44600 Saint-Nazaire, France (corresponding author).

Email: harifidy.ranaivomanana@univ-nantes.fr

<sup>3</sup> Associate Professor, Nantes Université, Ecole Centrale Nantes, CNRS, GeM, UMR 6183, F-44600 Saint-Nazaire, France

Email: fateh.bendahmane@univ-nantes.fr

<sup>4</sup> Full Professor, Nantes Université, Ecole Centrale Nantes, CNRS, GeM, UMR 6183, F-44600 Saint-Nazaire, France.

Email: ouali.amiri@univ-nantes.fr

<sup>5</sup> Senior Researcher, GERS-GIE, Université Gustave Eiffel, IFSTTAR, F-44344 Bouguenais, France.

Email: dimitri.deneele@univ-eiffel.fr

## Abstract

The present paper addresses the characterization of long-term and durability properties for employment in road subgrade construction of geopolymer-treated sediments previously studied in Part I of this work. Four mixtures are presently selected: untreated sediment (F<sub>0</sub>), sediment treated with 2% lime and 7% OPC (2L7OPC) and two geopolymer-treated mixtures using 5 and 10% of GGBFS/sodium-silicate based geopolymers (5GP2 and 10GP2 respectively). Long-term properties are investigated through unconfined compressive strength

26 and tensile strength tests. The resistance of designed materials against weathering conditions  
27 are tested through freeze-thaw cycles, humidification-drying cycles, and immersion at short  
28 term. 10GP2 mixtures showed long-term properties superior to that of 2L7OPC mixtures.  
29 Specimens treated using 10% GP2 are resistant against weathering conditions.

30

## 31 **Keywords**

32 Long-term properties, durability, geopolymer binder, sub-grade recovery

33

## 34 **1 Introduction**

35 The recovery of dredged sediments in civil engineering has been the subject of numerous  
36 studies. Earthwork structures, *e.g.*, *fill*, *roads*, *dykes*, allow to recover important quantities of  
37 sediments, with minimal environmental and economic impacts, compared to other recovery  
38 methods like substitution in concrete and cement that requires thermal/chemical pre-treatment  
39 of sediments. Following the traditional method, in order to use sediments in road construction  
40 and assimilated recovery methods, these are solidified using cement and hydraulic admixtures.  
41 However, the consumption of cement worldwide has important environmental impacts due to  
42 the high CO<sub>2</sub> emissions and consumption of energy and natural resources.

43 In the present study, an innovative method which consists of solidifying sediments using  
44 geopolymer binders (GP) is presented. The used GP binders are user-friendly and fabricated  
45 basing on industrial by-products, *i.e.*, grain granulated blast furnace slag (GGBFS) and fly ash  
46 (FA) respectively. Two geopolymers are tested: a first one denoted as GP1 composed of  
47 FA/GGBFS/K-silicate and a second one denoted as GP2 made using GGBFS/Na-silicate. In  
48 part I of the present study, short-term properties of treated sediments were investigated using  
49 aptitude for treatment test and unconfined compressive resistance (UCS). Basing on results of  
50 part I, the experimental program is pursued on 4 formulations (see section 2).

51 The investigation of short-term properties is important regarding the all-weather  
52 trafficability of the earthmoving machinery but also for the protection of earthworks against  
53 climatic agents during construction. Nevertheless, short term properties are not sufficient to  
54 assess the sustainability of designed materials for subgrade recovery. It is therefore required to  
55 examine long-term properties of materials in order to obtain homogeneous bearing capacity of  
56 designed materials and to maintain over time, despite fluctuations in the hydric state of water-  
57 sensitive subsoils, an acceptable bearing capacity. The durability properties against weathering  
58 conditions, freezing-thaw, wetting drying and immersion, should be as well tested. The  
59 destructive durability testing techniques are usually employed to evaluate the durability  
60 characteristics of stabilised soils (NF P 94-102-2 AFNOR 2001; NF P 234-1 AFNOR 1992;  
61 Stegemann and Cote 1996). Although the durability of geopolymers binders has been widely  
62 studied (Firdous et al. 2018; Nawaz et al. 2020) very few studies are available in literature that  
63 focused on durability properties of soil treated using geopolymers. These are mainly focused  
64 on the improvement of mechanical properties and mainly concern fly ash geopolymer-treated  
65 soils (Abdullah et al. 2021; Cristelo et al. 2011; Ghadir and Ranjbar 2018; Khadka et al. 2020;  
66 Phummiphan et al. 2018; Sukmak et al. 2013).

67 In terms of wetting–drying durability cycles, (Rios et al. 2017) observed low volumetric  
68 change on Class (F) fly-ash geopolymer-treated sand in addition to reasonable residual strength.  
69 Similar low volumetric change results were obtained by (Sargent et al. 2013) on an activated  
70 fly ash geopolymer-treated clay, despite a low residual UCS performance. The authors explain  
71 this result due to the lack of clay content in the stabilised soil limiting the cation exchange  
72 capacity. Recently, (Rivera et al. 2020) observed on an A4 type soil stabilized using fly ash  
73 based geopolymer, a low acceptable mass loss after twelve wetting-drying cycles.

74 In terms of freezing–thawing durability cycles, (Abdullah et al. 2019) observed on a Class  
75 (F) fly-ash geopolymer-stabilised kaolin clay a lower mechanical resistance in a freezing

76 climate than that in a tropical climate. The authors reported that low temperatures delay  
77 geopolymerization reactions.

78 It is worth noting that no study is available in literature concerning long-term and  
79 durability properties of dredged sediments treated using user-friendly geopolymers. Available  
80 studies in literature were mainly focused on drying-wetting cycles of treated soils.

81 For the above reasons, long term and durability properties of treated sediments are  
82 investigated in the present study with a particular attention paid to microstructural aspects.  
83 Long-term properties are tested using UCS and DCS tests over 4 curing periods (60, 90, 180  
84 and 360 days), followed by thermogravimetric analysis test (TGA). Three durability tests are  
85 performed, namely, freeze-thaw cycles, humidification-drying cycles and immersion at long-  
86 term.

87

## 88 **2 Tested mixtures**

89 In part I of the present study, the composition of used hydraulic and geopolymer binders  
90 as well as their characteristics were presented. These included the mechanical resistance,  
91 chemical activity, mineralogical composition and its pore size distribution. The characteristics  
92 of used sediments were investigated as well. Note that two geopolymers were used, GP1:  
93 FA/GGBFS/K-silicate and GP2: GGBFS/Na-silicate. 18 mixtures were tested using 5, 10 or 15  
94 % of binders, 9 without granular corrector and 9 with the addition of dredged sand. Aptitude  
95 for treatment test results showed that without a granular corrector, any of the tested binders is  
96 apt due to unmeasurable swelling and very low diametral compressive resistance. However, the  
97 addition of dredged sand with sediments-to-sand ratio of 0.3, allowed to limit organic matter  
98 content in the overall mixtures below 3 % and enhanced its granular skeleton. Thus, the  
99 experimental investigation was pursued using sediments with granular corrector.

100 Firstly, short-term properties were tested using unconfined compressive strength (UCS)  
101 at 7- and 28-days curing periods, then the mineralogical and microstructural properties were  
102 investigated using thermogravimetric analysis, X-ray diffraction and mercury intrusion  
103 porosimetry. Results showed that GP1 treatment does not allow to solidify the sediment-sand  
104 mixture. Thus, long-term and durability properties of GP1 are not tested herein. Sediments  
105 treated using 10 and 15% of GP2 showed acceptable results with low swelling and high  
106 mechanical performance. Specimens treated using 5% of GP2 showed an improvement in its  
107 mechanical performance (300 kPa) with respect to reference non-treated sediments (100 kPa),  
108 yet not sufficient for road subgrade use. Yet, it is interesting to investigate the potential of  
109 mechanical properties enhancement at long-term. Sediments treated using 2% of hydraulic lime  
110 and 8% of OPC showed acceptable mechanical resistance as well. As the objective herein is to  
111 optimize the used binder dosage, and in order to reduce the time needed to perform long-term  
112 and durability tests over 4 curing periods (60, 90, 180 and 360 days), 4 formulations are selected  
113 to pursue the experimental campaign. Untreated sediment-sand mixture ( $F_0$ ) is used as reference  
114 to be compared with treated formulations. As the effect of geopolymers on treated sediments  
115 mechanical properties at long term is still obscure, treatment using 5 and 10% of GP2 is  
116 considered. Despite the low mechanical efficiency of 5% GP2 treatment at short-term, the  
117 experimental program is pursued on 5GP2 in order to investigate a potential long-term  
118 enhancement. Therefore, 4 mixtures are selected, namely, one untreated ( $F_0$ ), one treated using  
119 OPC/L (2L7OPC) and two treated using GP2 geopolymer (5GP2 and 10GP2). Tested  
120 formulations are given in Table 1.

121

### 122 **3 Methods**

123 Long-term properties investigation consists of performing UCS and DCS tests and  
124 TGA/DTG analysis at 60-, 90-, 180- and 360-days curing periods. The resistance of designed

125 materials against weathering conditions are tested through 3 tests: freeze-thaw cycles,  
126 humidification-drying cycles, and immersion at short term.

127 Cylindrical specimens, of 5cm diameter, dedicated for UCS and durability tests are  
128 fabricated with a height-to-diameter ratio of 2, while those dedicated for DCS test are fabricated  
129 using a height-to-diameter ratio of 1. Specimens are compacted in order to obtain a density  $\rho =$   
130  $0.96 \cdot \rho_d$  (NF P 98-114-3 AFNOR 2001). Compaction parameters are presented after fabrication,  
131 specimens are conserved at temperature of 20 °C and 90 % humidity.

132

### 133 **3.1 Unconfined compressive resistance (UCS)**

134 The unconfined compressive resistance (UCS) is a reliable test used to evaluate the  
135 effectiveness of using designed materials in a civil engineering application *e.g., road*  
136 *embankments, fill materials*. For instance, in case of a road sub-grade application, UCS helps  
137 to evaluate the applicability of designed materials, and allows assessing the effect of applied  
138 treatment on the evolution of the mechanical resistance at long term as well.

139

### 140 **3.2 Tensile resistance (TS)**

141 Long term performance of treated materials is evaluated through the tensile strength (TS)  
142 and elasticity modulus (E) after a curing period of 90 days at least on cylindrical specimens of  
143 5 cm diameter and 5 cm height. Plotting (*E, TS*) couple, allows classifying treated materials.  
144 Then the required subgrade layer thickness can be determined as function of obtained  
145 mechanical classification, road bed classification and required road support platform ranking  
146 (SETRA-LCPC 2000a). Tensile strength TS can be estimated through direct tensile test or  
147 diametral compression test. However, direct tensile test is not applicable for class A soil,  
148 therefore diametral compression test is used to find TS with  $TS = 0.8DCS$  (NF P 98-114-3  
149 AFNOR 2001). This test is performed with charging speed less than 0.2 MPa/sec. Following

150 NF P 98-114-3 (NF P 98-114-3 AFNOR 2001), the modulus of elasticity (E) corresponds to 30  
151 % of maximal tensile stress on UCS stress-strain curve. Afterwards, E is obtained using Hook's  
152 law.

153

### 154 **3.3 Thermogravimetric analysis (TGA)**

155 TGA test allows identifying the presence/absence of organic matter and inorganic  
156 minerals in sediments as well as it enables to detect the potential new formed geopolymerization  
157 and OPC hardening products. It consists of increasing the ignition temperature of a sample from  
158 25-to-1025 °C with a heating rate of 5 °C /min, while measuring the evolution of its mass.

159

### 160 **3.4 Durability tests**

161 The strength of designed materials against weathering conditions are characterized  
162 through 3 tests: freeze-thaw and humidification-drying cycles, and immersion at short term. In  
163 fact, earthworks structures (roads, dykes, fill embankments) are subject to potential extreme  
164 natural conditions due to important temperature changes and floods followed by draining and  
165 appearance of sun. These phenomena induce freezing-thawing, wetting-drying and total  
166 immersion for long durations. Thus, to avoid severe failure, earthworks materials should be  
167 resistant in terms of durability properties. These potential extreme conditions can be simulated  
168 in the laboratory in order to evaluate the sustainability of designed materials.

169

#### 170 **3.4.1 Freeze-thaw cycles**

171 When the temperature drops under 0 °C, the water in the capillary pores freezes and its  
172 volume increases, which provokes cracking and swelling. Frost-induced swelling is a  
173 cumulative phenomenon that increases as the material is subjected to repeated freeze-thaw  
174 cycles. The damage of materials depends on several factors, like the cooling rate, the number



175 of cycles, the minimum temperature reached and the duration of the freezing. Thawing-freezing  
176 test is carried out according to the French standard (NF P 234-1 AFNOR 1992). The fabricated  
177 specimens ( $\Phi$ : 5 cm, h: 10 cm) are conserved at a room temperature of 20 °C for 40 days.  
178 Afterwards, these are subjected to 20 freeze-thaw cycles. A freezing-thawing cycle lasts 24 h  
179 and consists of 2 stages. During the first stage, specimens are maintained at 10 °C for 4 hours,  
180 while the second consists of keeping specimens at - 10 °C for 14 hours. The remaining time  
181 allows temperature alteration. The resistance of samples to freeze and thaw is evaluated through  
182 the UCS of specimens subjected to freeze-thaw cycles to that conserved at 20 °C.

183

#### 184 3.4.2 Wetting-drying cycles

185 The test methodology of wetting-drying durability test was proposed by (Stegemann and  
186 Cote 1996). It consists of comparing the UCS of samples submitted to 10 wetting-drying cycles  
187 with that of specimens kept at ambient temperature for 60 days. The fabricated specimens ( $\Phi$ :  
188 5 cm, h: 10 cm) are conserved for 40 days at room temperature of 20 °C to be submitted to  
189 wetting-drying cycles for 20 days. Specimens are immersed for 24 hours allow total saturation,  
190 to be dried later at 50 °C for 24 hours.

191

#### 192 3.4.3 Immersion in water at short term

193 The water immersion test is carried out according to the French standard (NF P 94-102-2  
194 AFNOR 2001). After fabrication, the cylindrical samples ( $\Phi$ : 5 cm, h: 10 cm) are conserved at  
195 a room temperature of 20 °C for 28 days. Then these are immersed in water for 32 days then  
196 removed to perform UCS test. To be resistant against immersion, UCS ratio of specimens after  
197 immersion to that cured at ambient conditions should be higher than 0.8. Durability tests are  
198 performed on 3 specimens per mixture in order to acquire the mean values.

199

## 200        **4 Long term properties**

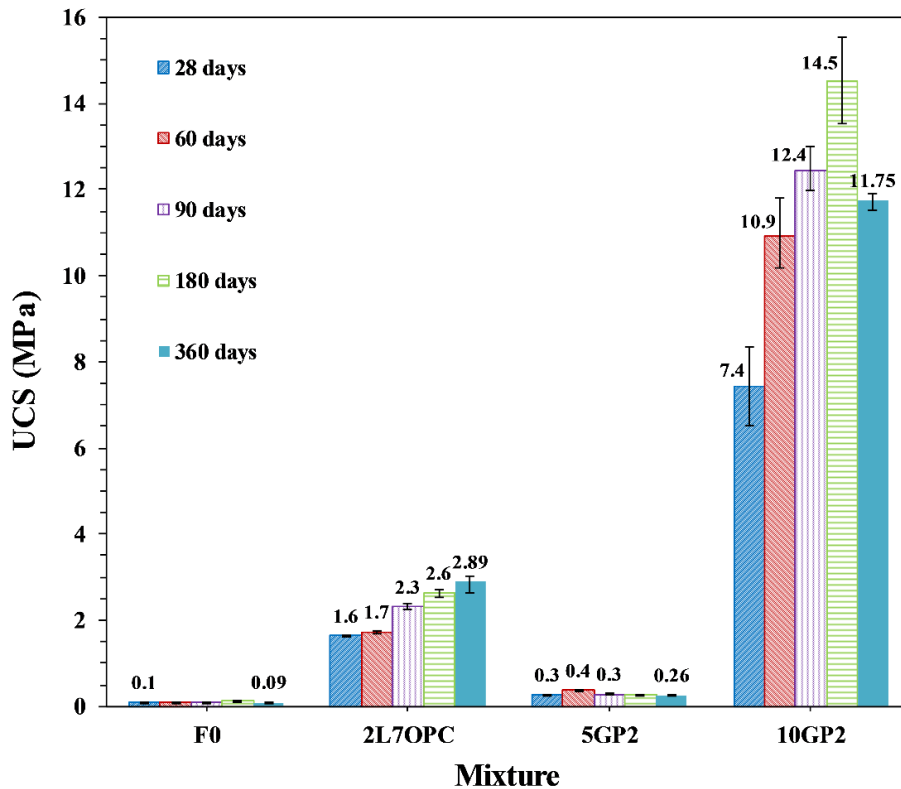
201            Long-term properties of treated sediments are investigated through unconfined  
202 compression test and diametral compression test. These are performed over the following  
203 curing periods: 60, 90, 180 and 360 days.

204

### 205        **4.1 Unconfined compressive strength**

206            Unconfined compressive strength results are presented in Fig. 1, indicating the average  
207 of 3 tested specimens along with the maximum and minimum values. Fig. 1 shows progressive  
208 increase of UCS till 360 days for 2L7OPC mixture. The obtained strength at 28 days is  
209 acceptable for road sub-grade application ( $> 1$  MPa) (SETRA-LCPC 2000b).

210            No significant UCS development is observed in the case of 5GP2 mixture, even after 360  
211 days curing period. With such UCS, 5GP2 cannot be employed in subgrade construction. Yet,  
212 it can be used as fill material since 300-400 kPa is largely sufficient for such application  
213 (SMOCS 2013; Stabcon 2011). This result can be explained by the fact that the quantity of  
214 calcium ions present in 5GP2 mixture is not sufficient to encapsulate the negative effects of  
215 OM and reacts with Na-silicate solution afterwards. GGBFS contains 43.2 % by mass of  
216 calcium oxide. Thus, adding 5 % of GGBFS includes  $0.05 * 43.2 = 2.16$  % of  $\text{Ca}^{2+}$  ions. This  
217 percentage is merely higher than the needed quantity of hydraulic lime to hinder the negative  
218 effects of OM. Thus, the lack of free  $\text{Ca}^{2+}$  in 5GP2 mixtures available for geopolymerization  
219 results in poor solidified materials mechanical strength.



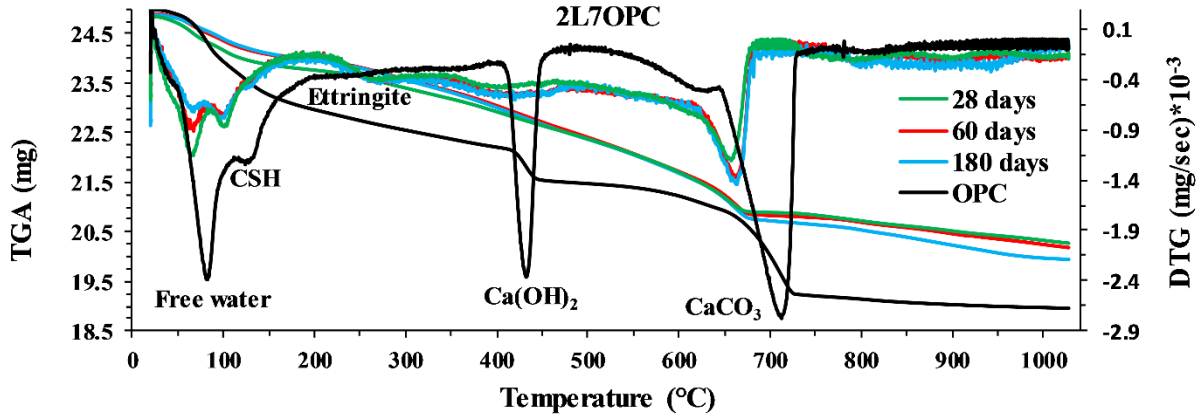
**Fig. 1. Unconfined compressive strength of designed materials as function of curing period**

10GP2 mixtures showed high UCS and high stability at short term curing period. Fig. 1 shows that the UCS of 10GP2 mixtures is superior to that of 2L7OPC. Moreover, the development of UCS continues over the tested curing periods. Yet a decrease in the UCS at 360 days is observed. Given that the diametral compressive strength increases between 180 and 360 days (Fig. 1), the decrease of UCS is supposedly due to an experimental default like a change of the humidity of the conservation chamber. 2L7OPC and 10GP2 mixtures can be employed as subgrade and sub-base materials according to its tensile strength (TS). The progressive increase of UCS can be explained by the fact that OM retards, and sometimes hinders (case of 5GP2), hardening reactions.

#### 4.2 Mineralogical analysis

Fig. 2(a) shows the TGA/DTG analysis of 2L7OPC sample over time. Fig. 2(a) shows 4 peaks at 80 °C, 130 °C, 430 °C and 710 °C which correspond to the release of free water,

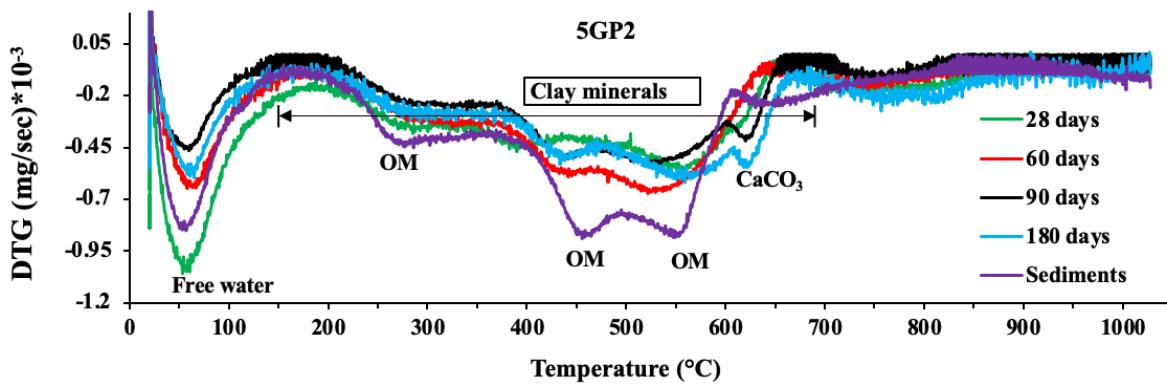
235 dehydration of OPC hydrates (*calcium silicate hydrate C-S-H, calcium aluminates C-A-H*),  
 236 dehydroxylation of portlandite  $\text{Ca(OH)}_2$  and decarbonation of calcium carbonates  $\text{CaCO}_3$   
 237 respectively (Bhatty 1986; Monteagudo et al. 2014).



238

239

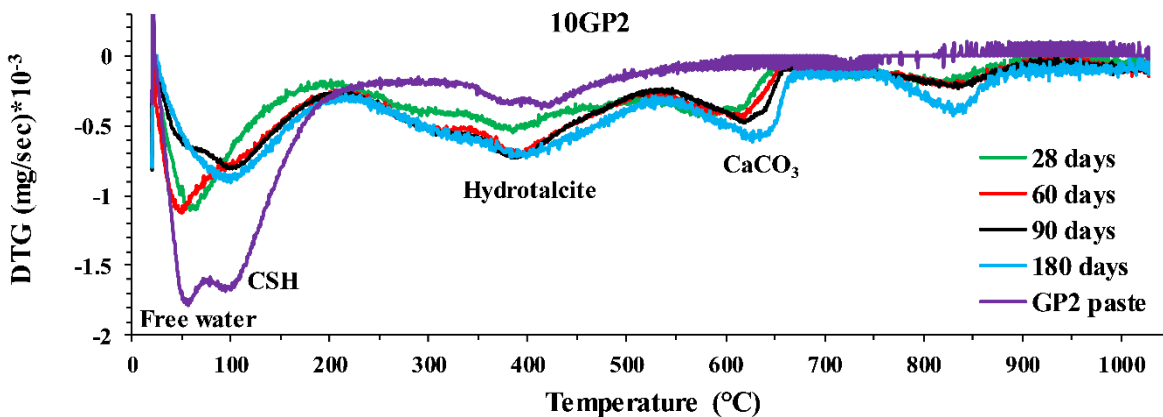
(a)



240

241

(b)



242

243

(c)

244 Fig. 2. TGA/DTG results as function of curing period of a) 2L7OPC b) 5GP2 c) 10GP2

245 TGA curves as well show that the decomposed mass increases with time knowing that the initial  
246 mass of all specimens is of 20mg. Thus, the increase of UCS can be correlated with the  
247 hydration of lime and OPC and thus the formation of new hydrates (Ranaivomanana et al.  
248 2013).

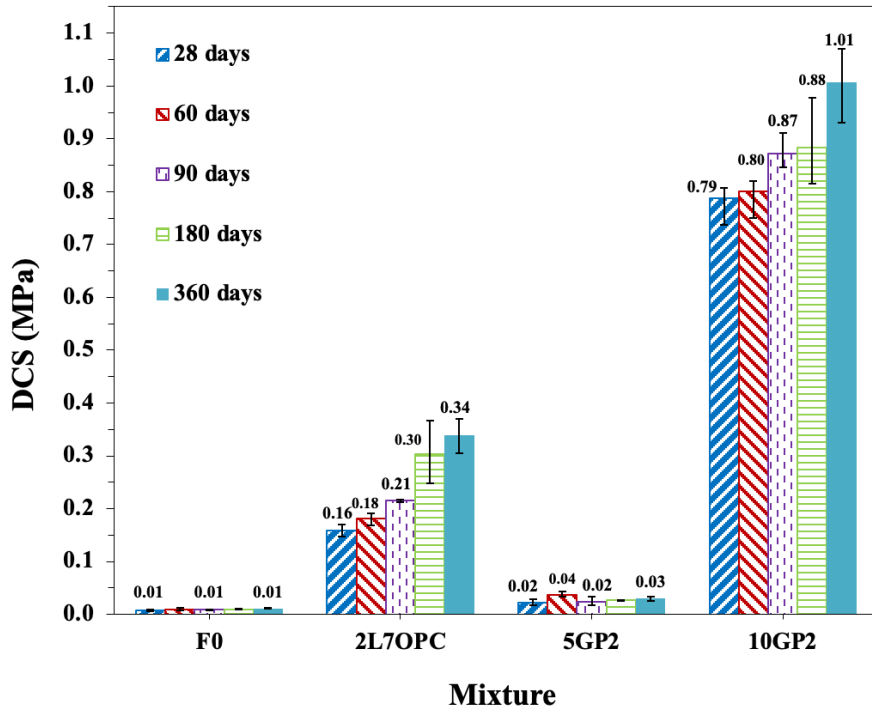
249 5GP2 DTG curves (Fig. 2(b)) show that the principle GP2 binder products, C-S-H and  
250 hydrotalcite that decompose at 105 °C and 400 °C respectively, cannot be identified. DTG  
251 peaks correspond to the materials contained in sediments but with lower amplitudes due to the  
252 dilution effect induced by GGBFS.

253 10GP2 DTG results in Fig. 2(c) show that the quantities of hydrotalcite (at 400 °C) and  
254 CaCO<sub>3</sub> increase with curing period. Which explains the mechanical improvement observed in  
255 Fig. 1.

256

### 257 **4.3 Diametral compressive strength (DCS)**

258 Fig. 3 shows a very low DCS of F<sub>0</sub> mixture. Similarly, in the case of 5GP2, where a slight  
259 increase of 10 kPa with respect to F<sub>0</sub> is observed. DCS of 2L7OPC and 10GP2 mixtures are in  
260 correlation with UCS results. In order to classify designed materials, it is necessary to determine  
261 their elastic modulus (E) in addition to tensile strength (TS), knowing that TS = 0.8 DCS.  
262 According to the obtained classification, the dimensioning of a subgrade or sub-base layer  
263 basing on designed materials can be performed.



264

265 **Fig. 3. Diametral compressive strength of designed materials as function of curing period**

266

267 **4.4 Classification of designed materials**

268

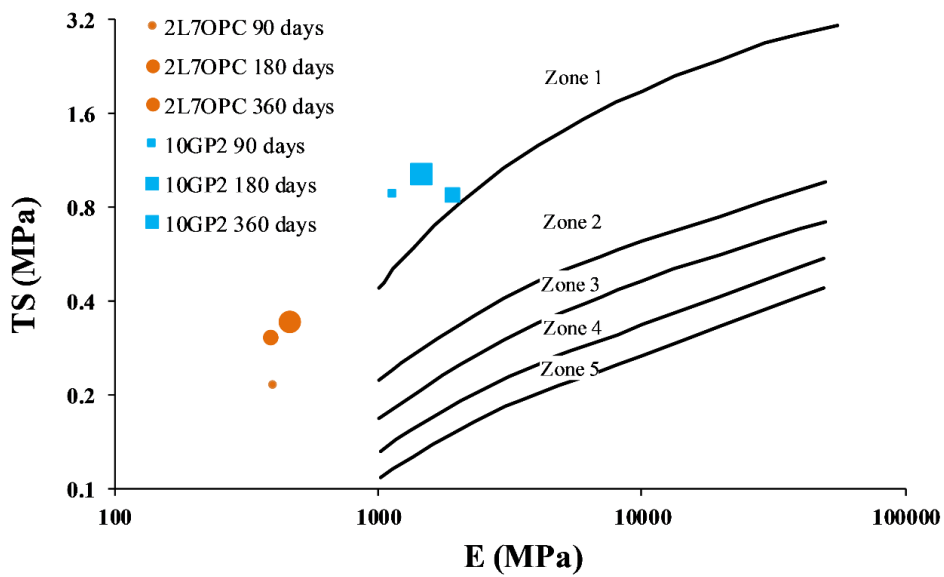
The mean values of (E, TS) couple of 2L7OPC and 10GP2 mixtures are presented in Fig.

269

4. Both TS and E values of tested mixtures increase with curing period due to the formation of

270

new hydration products.



271

272 **Fig. 4. Evolution of designed materials long term properties**

273 2L7OPC and 10GP2 mixtures are located in the zone 1 on (E, TS) diagram for the 3 curing  
274 periods: 90, 180 and 360 days. Solidified sediments are considered as materials of class 2 and  
275 thus can be used for sub-base as well as subgrade construction.

276  
277 **5 Durability properties**

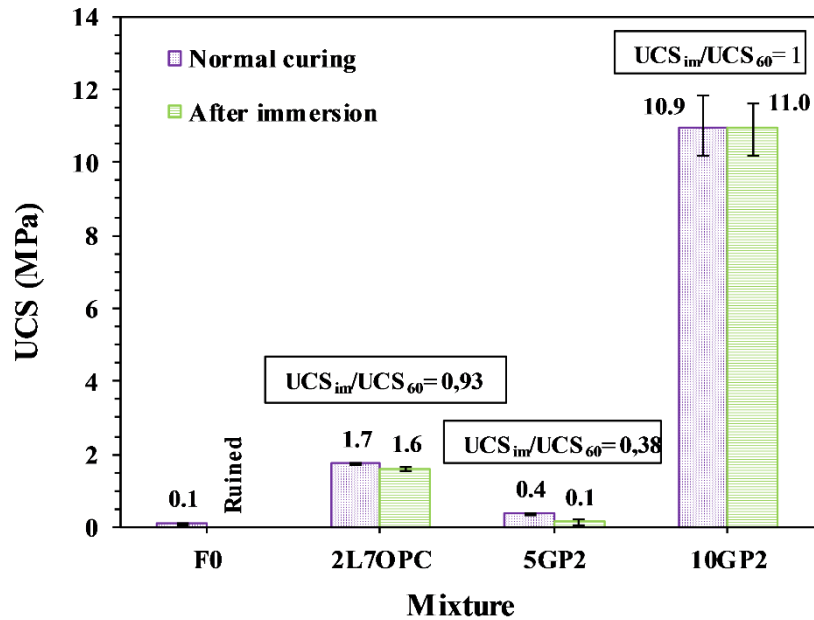
278 Testing durability properties is essential in order to determine the feasibility of employing  
279 designed materials in subgrade construction. Durability properties of designed materials are  
280 evaluated by considering 3 weathering tests, namely, immersion at short term, freeze-thaw  
281 cycles and humidification-drying cycles.

282 **5.1 Immersion in water at short term**

283 Water immersion test is carried out according to the French standard (NF P 94-102-2  
284 AFNOR 2001). After 28 days curing period, 3 specimens of each mixture, are immersed in  
285 water for 32 days. Three other specimens are left at 20 °C and 90 % humidity for 60 days. After  
286 60 days, immersed specimens are removed from water then tested for UCS, to be compared to  
287 that cured at normal conditions. For subgrade application, materials are considered resistant to  
288 immersion if the ratio  $UCS(\text{immersed specimens})/UCS(\text{normally cured specimens})$  is less than  
289 0.8 for materials having a methylene blue value (MBV) less than 5 g/100 g, and less than 0.6 if  
290 MBV greater than 5 g/100g. UCS results of specimens cured at normal conditions and those  
291 immersed in water are presented in Fig. 5.

292 Untreated specimens ( $F_0$ ) are deteriorated few days after immersion in water. Even if 100 kPa  
293 is sufficient for certain applications such as fill embankments, untreated sediments would not  
294 resist to rainy conditions. By comparing UCS results with and without immersion, 2L7OPC  
295 and 10GP2 mixtures are considered to be insensible against immersion, given that  
296  $UCS_{im}/UCS_{60}$  ratio remained higher than 0.6, even higher than 0.8 knowing that raw sediment

297 MBV is of 5.8 g/100 g. UCS of 2L7OPC decreased slightly while that of 10GP2 is not affected.  
 298 5GP2 mixture is not resistant against immersion with a  $UCS_{im}/UCS_{60}$  ratio equal to  $0.38 < 0.6$ .  
 299



300

301

Fig. 5. UCS results of sensibility to water test

302

$UCS_{im}/UCS_{60}$ : UCS (immersed specimens)/ UCS(normally cured specimens for 60 days)

303

304

## 5.2 Freeze thaw cycles

305

When the temperature drops under  $0\text{ }^{\circ}\text{C}$ , the water in the capillary pores freezes and is accompanied by a volume increase which provokes swelling and cracking. Frost-induced swelling is a cumulative phenomenon that evolves as the material is submitted to repeated freeze-thaw cycles. The damage of materials depends on several factors, like the cooling rate, the number of cycles, the minimum temperature reached and the duration of the freezing. Thawing-freezing test is carried out according to (NF P 234-1 AFNOR 1992).

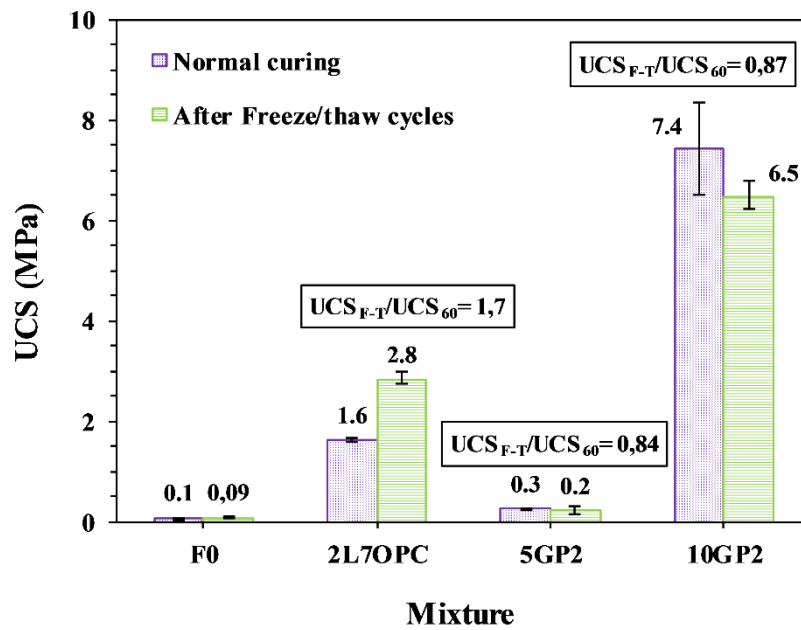
311

After fabrication, specimens are conserved for 28 days at normal conditions and then subjected against 20 freeze-thaw cycles afterwards. UCS of specimens cured at  $20\text{ }^{\circ}\text{C}$  ( $UCS_{60}$ ) is

312



313 compared to that subjected to freeze-thaw cycles ( $UCS_{F-T}$ ). As  $UCS_{F-T}/UCS_{60}$  ratio of the 4  
 314 tested mixtures is higher than 0.8, these are considered to be resistant to freeze-thaw cycles.



315

316

**Fig. 6. UCS results of freeze/thaw test**

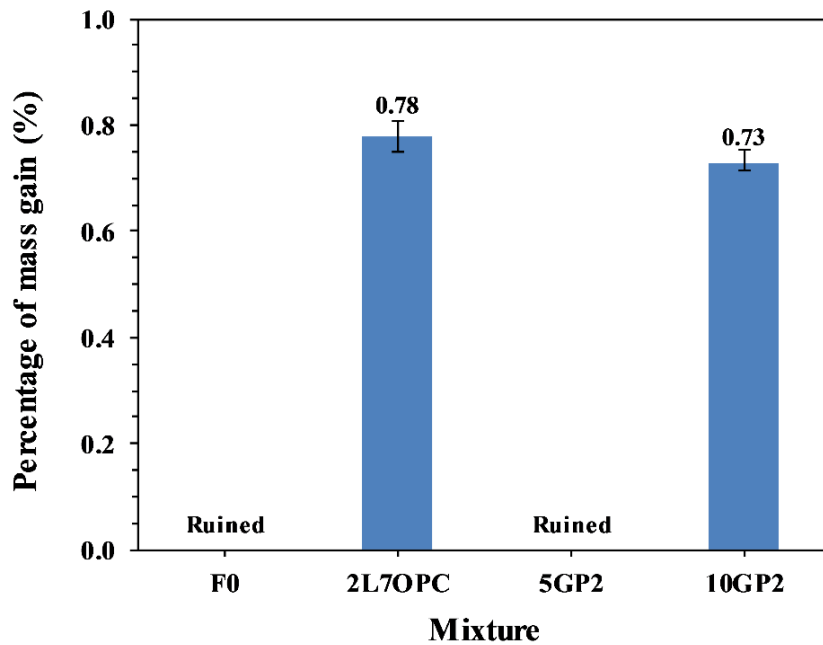
317  $UCS_{F-T}/UCS_{28}$ :  $UCS(\text{after freeze-thaw cycles}) / UCS(\text{normally cured for 28 days})$

318

### 319 5.3 Humidification-drying cycles

320 The test methodology of wetting-drying test was proposed by (Stegemann and Cote  
 321 1996). Humidification-drying cycles could be the most intense weathering conditions on treated  
 322 sediments/soil. 28 days after fabrication, specimens are subjected to 10 humidification-drying  
 323 cycles. Specimens are weighed before and after the experiment in order to assess the mass loss  
 324 afterwards. The percentages of mass change are given in Fig. 7. Untreated specimens ( $F_0$ ) and  
 325 5GP2 are partially deteriorated after 1 humidification cycle, then destroyed few hours after the  
 326 beginning of the second humidification cycle. On the other hand, 2L3OPC and 10GP2  
 327 specimens remained totally intact after 10 humidification-drying cycles. Contrariwise, mass  
 328 measurements after the end of the experiment reveal a slight gain in specimen's mass. Fig. 8  
 329 and Fig. 9 show the TGA/DTG curves of both 2L7OPC and 10GP2 mixtures. This result is due

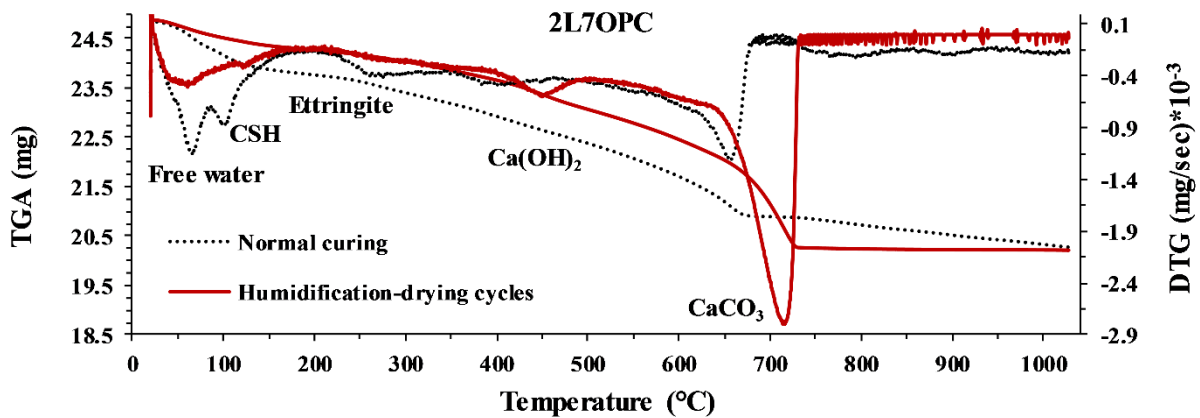
330 to the formation of  $\text{CaCO}_3$  upon contact with tap water during 10 humidification phases. In  
 331 2L7OPC, the decomposed mass between 600 and 750 °C is doubled after immersion. Normally  
 332 cured 10GP2 specimens do not contain  $\text{CaCO}_3$  while the humidification cycles lead to the  
 333 formation of new  $\text{CaCO}_3$  minerals. The formation of  $\text{CaCO}_3$  improves the mechanical  
 334 properties of treated materials, and thus increases the strength of specimens to humidification-  
 335 drying cycles. (Ranaivomanana et al. 2013) reported that immersing cementitious materials in  
 336 carbonated water leads to the formation of new calcium carbonates. Durability results are  
 337 consistent with obtained UCS, DCS and  $S_v$  results.



338

339

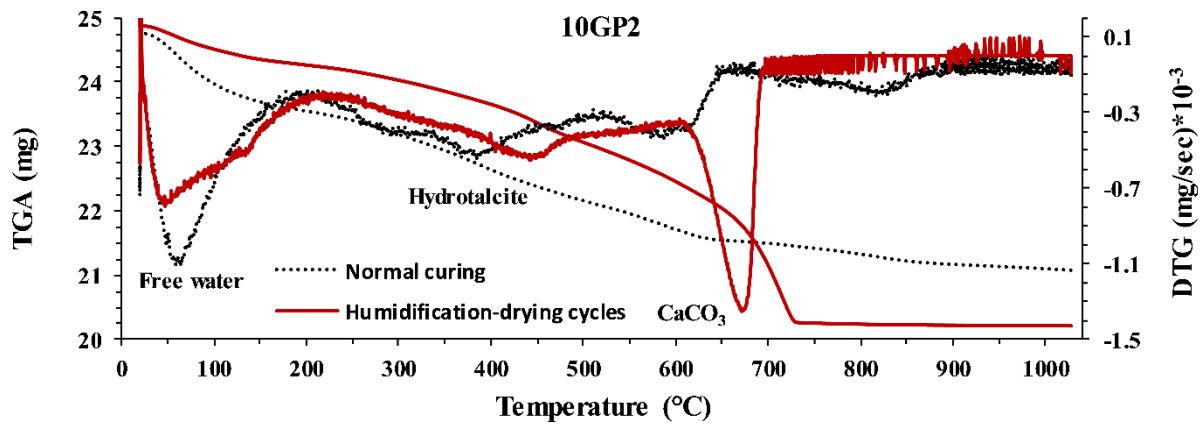
Fig. 7. Humidification-drying cycles results. Percentage of mass gain



340

341

Fig. 8. 2L7OPC TGA/DTG results at normal curing and after humidification/drying cycles



342  
343 **Fig. 9. 10GP2 TGA/DTG results at normal curing and after humidification/drying cycles**

344  
345 **6 Conclusion**

346 In the present study, long term and durability properties of a highly organic sediment  
347 treated using a geopolymer binder, based on GGBFS/sodium silicate solution (GP2), for an  
348 application in road subgrade construction are investigated.

349 Long-term mechanical properties and durability properties are tested on 4 formulations:  
350 untreated-reference mixture (F<sub>0</sub>) and 3 treated mixtures, namely 2L7OPC, 5GP2 and 10GP2.  
351 The main observed results are the following:

- 352 • UCS and TS of 2L7OPC and 10GP2 mixtures increased with time. TGA results showed  
353 that the formation of hydrotalcite in 10GP2 specimens and OPC hydration products in  
354 2L7OPC specimens, is responsible for its mechanical strength enhancement. TGA  
355 curves showed as well an increase in the formation of hydrates/geopolymerisation  
356 products over curing period.

357 UCS and TS results of 5GP2 do not show an improvement even at 360 days curing  
358 period. The quantity of calcium in 5GP2 is not sufficient to saturate the OM and  
359 participate to geopolymerization reactions.

- 360 • F<sub>0</sub> and 5GP2 mixtures are not resistant to any of the 3 weathering tests namely,  
361 immersion in water, freeze-thaw and humidification-drying cycles. The absence of

362 treatment in F<sub>0</sub>, and the weak geopolymer setting reactions in 5GP2, produced materials  
363 of important macro-porosity, and thus high permeability. Furthermore, the absence of  
364 binding between particles, and the high permeability made the tested specimens  
365 unresistant against weathering cycles.

366 2L7OPC and 10GP2 mixtures are very resistant to weathering conditions, with higher  
367 mechanical performance of 10GP2 compared to 2L7OPC. The formation of  
368 hydration/geopolymerisation products induces the agglomeration of particles, and thus  
369 generating larger cluster that increases the mechanical resistance of designed materials.  
370 Furthermore, the formed hydrates decreased the volume of porosity, and replaced the  
371 macro-pores by micro-pores, which decreased its permeability. Stronger binding  
372 between particles and lower permeability reduces the sensitivity of materials against  
373 weathering conditions.

374 • Based on elastic modulus and tensile strength results, geopolymer based mixture  
375 10GP2, can be employed for subgrade and sub-base road layers. However, 2L7OPC  
376 mixtures can be used only for subgrade construction.

377

## 378 **Notation**

379 *The following symbols are used in this paper:*

380	CaCO <sub>3</sub>	Calcium carbonates
381	CSH	Calcium silicate hydrate
382	DCS	Diametral compressive strength
383	DTG	Differential thermogravimetric analysis
384	E	Elastic modulus
385	FA	Fly ash
386	GP	Geopolymer binder

387	GGBFS	Ground granulated blast furnace slag
388	L	Hydraulic lime
389	OPC	Ordinary Portland Cement
390	OM	Organic matter
391	TGA	Thermogravimetric analysis
392	TS	Tensile strength
393	UCS	Unconfined compressive strength

## 394        **7 Data availability statement**

395        The authors confirm that the data supporting the findings of this study are available within the  
396        article (measured by the authors or extracted from their respective published source in the  
397        literature that is listed in the reference section).

398

## 399        **8 References**

- 400        Abdullah, H. H., M. A. Shahin, and P. Sarker. 2019. "Use of Fly-Ash Geopolymer  
401        Incorporating Ground Granulated Slag for Stabilisation of Kaolin Clay Cured at  
402        Ambient Temperature." *Geotech. Geol. Eng.*, 37 (2): 721–740.  
403        <https://doi.org/10.1007/s10706-018-0644-2>.
- 404        Abdullah, H. H., M. A. Shahin, M. L. Walske, and A. Karrech. 2021. "Cyclic behaviour of clay  
405        stabilised with fly-ash based geopolymer incorporating ground granulated slag."  
406        *Transp. Geotech.*, 26: 100430. <https://doi.org/10.1016/j.trgeo.2020.100430>.
- 407        Bhatta, J. I. 1986. "Hydration versus strength in a portland cement developed from domestic  
408        mineral wastes — a comparative study." *Thermochim. Acta*, 106: 93–103.  
409        [https://doi.org/10.1016/0040-6031\(86\)85120-6](https://doi.org/10.1016/0040-6031(86)85120-6).
- 410        Cristelo, N., S. Glendinning, and A. Teixeira Pinto. 2011. "Deep soft soil improvement by  
411        alkaline activation." *Proc. Inst. Civ. Eng. - Ground Improv.*, 164 (2): 73–82.  
412        <https://doi.org/10.1680/grim.900032>.
- 413        Firdous, R., D. Stephan, and J. N. Y. Djobo. 2018. "Natural pozzolan based geopolymers: A  
414        review on mechanical, microstructural and durability characteristics." *Constr. Build.*  
415        *Mater.*, 190: 1251–1263. <https://doi.org/10.1016/j.conbuildmat.2018.09.191>.
- 416        Ghadir, P., and N. Ranjbar. 2018. "Clayey soil stabilization using geopolymer and Portland  
417        cement." *Constr. Build. Mater.*, 188: 361–371.  
418        <https://doi.org/10.1016/j.conbuildmat.2018.07.207>.

- 419 Khadka, S. D., P. W. Jayawickrama, S. Senadheera, and B. Segvic. 2020. “Stabilization of  
420 highly expansive soils containing sulfate using metakaolin and fly ash based  
421 geopolymer modified with lime and gypsum.” *Transp. Geotech.*, 23: 100327.  
422 <https://doi.org/10.1016/j.trgeo.2020.100327>.
- 423 Monteagudo, S. M., A. Moragues, J. C. Gálvez, M. J. Casati, and E. Reyes. 2014. “The degree  
424 of hydration assessment of blended cement pastes by differential thermal and  
425 thermogravimetric analysis. Morphological evolution of the solid phases.”  
426 *Thermochim. Acta*, 592: 37–51. <https://doi.org/10.1016/j.tca.2014.08.008>.
- 427 Nawaz, M., A. Heitor, and M. Sivakumar. 2020. “Geopolymers in construction - recent  
428 developments.” *Constr. Build. Mater.*, 260: 120472.  
429 <https://doi.org/10.1016/j.conbuildmat.2020.120472>.
- 430 NF P 94-102-2 AFNOR. 2001. “Sols : Reconnaissance et essais — Sol traité au liant  
431 hydraulique, éventuellement associé à la chaux, pour utilisation en couche de forme —  
432 Partie 2 : Méthodologie des études de formulation en laboratoire. [Soils: Investigation  
433 and testing — Soil treated with hydraulic binder, possibly combined with lime, for use  
434 as a selected fill — Part 2: Methodology of laboratory formulation studies].”
- 435 NF P 98-114-3 AFNOR. 2001. “Assises de chaussées — Méthodologie d’étude en laboratoire  
436 des matériaux traités aux liants hydrauliques — Partie 3 : Sols traités aux liants  
437 hydrauliques éventuellement associés à la chaux. [Roadway foundations —  
438 Methodology for laboratory study of materials treated with hydraulic binders — Part 3:  
439 Soils treated with hydraulic binders possibly combined with lime].”
- 440 NF P 234-1 AFNOR. 1992. “Essais relatifs aux chaussées - Comportement au gel des matériaux  
441 traités aux liants hydrauliques - Partie 1 : essai de résistance au gel-dégel des graves et  
442 sables traités [Tests relating to pavements. Freezing behaviour of material treated with  
443 hydraulic binders. Part 1: freezing–thawing test of stabilized gravel or sand].”
- 444 Phummiphan, I., S. Horpibulsuk, R. Rachan, A. Arulrajah, S.-L. Shen, and P. Chindaprasirt.  
445 2018. “High calcium fly ash geopolymer stabilized lateritic soil and granulated blast  
446 furnace slag blends as a pavement base material.” *J. Hazard. Mater.*, 341: 257–267.  
447 <https://doi.org/10.1016/j.jhazmat.2017.07.067>.
- 448 Ranaivomanana, H., J. Verdier, A. Sellier, and X. Bourbon. 2013. “Sealing process induced by  
449 carbonation of localized cracks in cementitious materials.” *Cem. Concr. Compos.*,  
450 Complete (37): 37–46. <https://doi.org/10.1016/j.cemconcomp.2012.12.008>.
- 451 Rios, S., N. Cristelo, A. Viana da Fonseca, and C. Ferreira. 2017. “Stiffness Behavior of Soil  
452 Stabilized with Alkali-Activated Fly Ash from Small to Large Strains.” *Int. J.*  
453 *Geomech.*, 17 (3): 04016087. American Society of Civil Engineers.  
454 [https://doi.org/10.1061/\(ASCE\)GM.1943-5622.0000783](https://doi.org/10.1061/(ASCE)GM.1943-5622.0000783).
- 455 Rivera, J. F., A. Orobio, N. Cristelo, and R. Mejía de Gutiérrez. 2020. “Fly ash-based  
456 geopolymer as A4 type soil stabiliser.” *Transp. Geotech.*, 25: 100409.  
457 <https://doi.org/10.1016/j.trgeo.2020.100409>.
- 458 Sargent, P., P. Hughes, M. Rouainia, and M. White. 2013. “The use of alkali activated waste  
459 binders in enhancing the mechanical properties and durability of soft alluvial soils.”  
460 *Eng. Geol.*, 152: 96–108. <https://doi.org/10.1016/j.enggeo.2012.10.013>.
- 461 SETRA-LCPC. 2000a. *GTS. Treatment of soil by lime and/or hydraulic binders: application to*  
462 *the realization of embankments and road sub-grade layer [Traitement des sols à la*  
463 *chaux et/ou aux liants hydrauliques: application à la réalisation des remblais et des*

464 *couches de forme*]. 246. Technical guide. LCPC, Laboratoire central des ponts et  
465 chaussées; SETRA, Service d'études techniques des routes et autoroutes.

466 SETRA-LCPC. 2000b. *GTR. Technical guide for the implementation of fill and road subgrade*  
467 *[Guide technique pour la réalisation des remblais et des couches de forme]*. Fascicules  
468 I - Principes généraux, 211. Technical guide. LCPC, Laboratoire central des ponts et  
469 chaussées; SETRA, Service d'études techniques des routes et autoroutes.

470 SMOCS. 2013. *Sustainable Management of Contaminated Sediments (SMOCS) – Guideline*.  
471 82. Baltic Sea Region Programme 2007-2013.

472 Stabcon. 2011. "Vägledning för nyttiggörande av muddermassor i hamn- och  
473 anläggningskonstruktioner [Recovery guidance of sediments in port and harbors]."  
474 Sweden: Swedish geotechnical institute.

475 Stegemann, J. A., and P. L. Cote. 1996. "A proposed protocol for evaluation of solidified  
476 wastes." *Sci. Total Environ.*, 178 (1–3): 103–110.

477 Sukmak, P., S. Horpibulsuk, and S.-L. Shen. 2013. "Strength development in clay-fly ash  
478 geopolymers." *Constr. Build. Mater.*, Special Section on Recycling Wastes for Use as  
479 Construction Materials, 40: 566–574.  
480 <https://doi.org/10.1016/j.conbuildmat.2012.11.015>.  
481

## 482 Tables

483 **Table 1. Composition of tested mixtures**

Designation	Binder(s)	Sediment + dredged sand <sup>(1)</sup>	Water-to-dry ratio
<b>F<sub>0</sub></b>	-	100%	11.4%
<b>2L7OPC</b>	2% lime + 7% OPC <sup>(2)</sup>	91%	11.4%
<b>5GP2</b>	5% GP2 <sup>(3)</sup>	95%	11.4%
<b>10GP2</b>	10% GP2	90%	11.4%

484 <sup>(1)</sup> dredged sediments to dredged sand ratio = 3

485 <sup>(2)</sup> Ordinary Portland cement: CEM II 32.5

486 <sup>(3)</sup> GP2: GGBFS + 35% sodium silicate solution + 7.7% water (the composition of GP2 binder is  
487 given in part I of the present study)