

# Comparative study of stabilization/solidification of dredged sediments with ordinary Portland cement and calcium sulfo-aluminate cement in the framework of valorization in road construction material

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1	Comparative study of Stabilization/Solidification of dredged sediments with Ordinary
2	Portland cement and Calcium Sulfo-Aluminate cement in the framework of
3	valorization in road construction material
4	
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#### 23 Abstract

Stabilization/solidification (S/S) of dredged sediments is an environmentally friendly, 24 25 low-cost and time-efficient way to manage this waste (according to the European waste classification) in the context of valorization as novel resources. In this study, 26 27 the main objective was to evaluate the efficiency of using a novel and green binder Calcium Sulfo-Aluminate (CSA) cement to solidified Dunkirk sediments. For 28 comparison, the Ordinary Portland Cement (OPC) binder was used as a reference. For 29 this purpose, several tests were performed on samples containing various types and 30 31 amounts of binders. The experimental program included: modified Proctor compaction test, immediate Californian Bearing Ratio (I-CBR) index test, unconfined 32 compressive strength test, splitting tensile strength test, measurements of elastic and 33 34 secant modulus, and mineral and micropore structure analysis. It is realistic to conclude that the compaction performance of the CSA-/OPC-solidified sediments is 35 significantly improved. The mechanical performance of compressive, tensile strength 36 37 and elastic modulus increases with binder content and curing time. The simple model 38 obtained to relate the compressive and tensile strength with elastic modulus allow predicting easily, for a given suitable set of characteristics, the type or the amount of 39 binder needed. This model allows optimizing the amount of binder to reach a given 40 properties of solidified sediments and hence improves the cost of developed material. 41 The results also demonstrate the ability of solidified sediment to be valorized in road 42 43 construction.

44 Keywords: Sediments; Valorization; Ordinary Portland Cement;

2

Calcium

45 Sulfo-Aluminate cement; Mechanical strength; Road material

#### 46 **1. Introduction**

To prevent flood risks and ensure a sufficient draft for navigation, regular sediment 47 dredging is carried out every year. However, the dredging process produce a large 48 amount of sediments. In Europe, the annual volume of dredged sediments is estimated 49 at 300 million tons [1]. Fine dredged sediments is regarded as waste with high 50 moisture content, complex components (as contaminants), high organic matter content, 51 high compressibility and low mechanical strength [2], [3], [4], [5]. Conventional 52 53 management of sediments as ocean dumping, land deposit, and blow-filled piling, are not sustainable and environmentally friendly as it can cause serious secondary 54 pollution [6], [7]. Therefore, try to find an alternative method for sediments 55 56 management is the core of current research.

The valorization of sediments in road construction by stabilization/solidification (S/S) 57 method has received considerable interest in recent years [8], [9], [10], [11], [12]. 58 Stabilization/Solidification (S/S) is considered as an environmentally friendly, 59 low-cost and time-efficient way for the treatment of sediments. It can significantly 60 relieve environmental impacts and improve the mechanical properties of sediments 61 [13]. The reuse of S/S treated sediments in the field of construction can be considered 62 63 as a promising method not only to solve problems related to sediment management but also to provide a new source of construction materials [14], [15]. The traditional 64 binder used for S/S is Ordinary Portland Cement (OPC), and the justification has been 65 proved by efficiency shown over time, low cost, availability and reliability [16], [17]. 66

However, the manufacturing process of OPC has significant environmental issues, i.e., 67 high consumption of energy (5000 MJ/t cement), non-renewable resources (1.5 t 68 limestone and clay/t cement) and high CO<sub>2</sub> emissions (0.95 t CO<sub>2</sub>/t cement). OPC 69 manufacturing acts as a significant contributor to anthropogenic  $CO_2$  emission (5-7 %) 70 71 [18], [19]. Due to the increasing demand for construction, the share of  $CO_2$  emissions attributed to the cement production is predicted to increase from 16 % to 24 % by 72 2050, demonstrating the urgent need to improve its sustainability [20]. Therefore, 73 several research works are undertaken to propose alternative cementitious material 74 75 with low energy consumption, less pollution or no pollution and excellent durability, such as reactive MgO [21], magnesia-industrial by products [22], [23], [24], 76 Si-modified MgO cement [16], industrial by products-cement [17], calcined dredging 77 78 sediments-cement [25], basalt fiber-cement [26] uncontaminated sediments-cement [27] and others. 79

The continuous push toward the development of a sustainable cementitious binder has 80 81 resulted in increased research activity on Calcium Sulfo-Aluminate (CSA) cement in 82 the last few decades. CSA cement is produced by the calcination of gypsum, bauxite, and limestone at ~1250 °C, which is 200 °C lower than the temperature used for 83 Portland cement clinker (~1450 °C) [28]. Moreover, the lower limestone demand (35 % 84 - 40 %) for the manufacture of CSA cement reduces its carbon footprint. The 85 production of 1 t of OPC clinker releases a maximum of 0.95 t of CO<sub>2</sub>, whereas the 86 production of 1 t of CSA clinker leads to reduce by one third the CO<sub>2</sub> emissions [29], 87 [30]. The main hydraulic phase of CSA is ye'elimite ( $C_4A_3S$ ), and the secondary 88

phases included belite  $(C_2S)$ , calcium sulfate, and gehlenite  $(C_2AS)$ , etc. The 89 hydration of ye'elimite is used to gain early age strength, and the hydration of belite 90 91 could improve long-term strength [31], [32]. CSA cement exhibits many interesting properties such as short setting time, high early strength, impermeability, sulfate, and 92 chloride corrosion resistance, and low alkalinity [32], [33], [34], [35]. Hence, CSA 93 cement has demonstrated considerable potential in the S/S of hazardous materials, 94 such as low-level radioactive waste, heavy metals waste, sewage sludge, bottom ash, 95 and galvanic sludge [36], [37], [39], [40], [41]. Besides, a few studies use CSA 96 97 cement in geotechnical applications, especially in ground improvement. For example, it is proved that CSA-treated sand has significantly high initial strength, which 98 powerfully indicates that the use of CSA in geotechnical applications may lead to the 99 100 reduced construction period and carbon footprint [42], [43], [44], [45]. In fact, to the best of our knowledge, there remain scientific questions as to reused sediments with 101 CSA cement, no research thus far on the use of CSA cement with sediments in road 102 103 construction. Therefore, this study evaluates the effectiveness of CSA cement on the 104 marine sediment from Dunkirk Port (France) for potential use as road material. In this context, the main objectives of the present research work are: (i) to assess the 105 Proctor compaction properties and immediate Californian Bearing Ratio (I-CBR) 106 index of solidified sediments with the addition of CSA cement; (ii) to validate the 107 efficacy of CSA cement in enhancing the properties (e.g., compressive strength, 108 109 tensile resistance, and elastic modulus) of solidified sediment and (iii) to demonstrate

110 its ability in comparison to OPC treatment. The results of this paper will contribute to

1	11	an investigation on the feasibility of using an eco-friendly CSA binder and dredged
1	12	marine sediments as road material products without complex treatment.
1	13	
1	14	2. Experimental materials and methods
1	15	2.1. Raw Materials
1	16	The sediments (SD) were dredged from Dunkirk Harbour in the north of France. The
1	17	basic physical characteristics of sediments are measured according to the standard
1	18	used in France and/or Europe.
1	19	- The initial water content is measured according to test standard NF P 94-050
1	20	[46].
1	21	- The true density is measured using a helium pycnometer (NF EN ISO 17892-3)
1	22	[47].
1	23	- The organic matter is measured by the ignition method at 450 $^{\circ}$ C (XP P94-047)
1	24	[48].
1	25	- The liquid limit and plastic limit, defined by the percussion-cup method and
1	26	the rolling thread method is measured according to test standard EN ISO
1	27	17892-12 [49].
1	28	- The particle size distribution is measured according to test standard EN ISO
1	29	13320 [50].
1	30	In terms of binders, Ordinary Portland cement (OPC) with CEM I 52.5 R from
1	31	LafargeHolcim Saint-Pierre-La-Cour company and calcium sulfoaluminate cement
1	32	(CSA) from Vicat company were used in this study. The chemical compositions of all

133	these materials were determined by X-ray fluorescence (XRF) analysis with a
134	BRUCKER AXS D8 ADVANCE using cobalt and ka radiation (ka=1.78 Å), the
135	results are listed below.

136 **2.2. Methods** 

In the current study, the mix proportions of different samples are shown in Table 1,
which was designed with reference to our previous research [8], [9], [23]. It should be
noted that the content of OPC and CSA was determined in percentage by mass of dry
sediments. Tap water was used as mixing water.

141 The modified Proctor compaction tests and immediate Californian Bearing Ratio (I-CBR) index tests were carried out according to NF EN 13286-2 [51] and NF EN 142 13286-47 [52], to determine the feasibility of the solidified sediments to be used as a 143 144 filling material in the road. The test carries out in a Proctor mould with a diameter of 150 mm and a height of 120 mm, and the sediment was filled up in five equal layers 145 and 25 strokes were applied to each layer with a 4.5 kg hammer by performing falls 146 147 from a height of 457 mm. Then test the water content and I-CBR index, in order to determine the maximum dry unit weight, optimum water content and I-CBR index 148 values of the solidified sediments. 149

The unconfined compressive strength was measured by using the INSTRON testing machine according to NF EN 13286-41 [53], as shown in Fig. 1 (a). The cylindrical specimens were prepared by static compression at their respective maximum dry density and optimum water content, according to NF EN 13286-53 [54]. Firstly, the dry sediments and cement were mixed in an electric mixer for 5 min. Then the tap

water was added and mixing is performed for another 5 min. Afterwards, the mixtures 155 were transferred into the cylindrical mould with a diameter of 50 mm and a length of 156 100 mm. By using axial compression, the mixtures is compacted to reach the desired 157 height. The compacted specimen was demolded and stored in covered plastic boxes 158 for preventing the moisture losses. The curing is made in a curing room at  $20 \pm 1$  °C 159 for 3 d, 7 d, and 28 d. A group of three same cylindrical specimens was prepared for 160 each mixes, and the results discussed later were the average values measured on three 161 specimens. The unconfined compressive strength is given by the equation (1). 162

163 The elastic modulus is determined in an unconfined compression test in accordance with NF EN 13286-43 [55], which using the secant slope at 30% of peak compressive 164 strength. Firstly, the maximum compression strength of the specimen is measured (for 165 166 a given mix). Then the elastic modulus is measured on another sample. As shown in Fig. 1 (b), to measure the elastic modulus, an extensioneter with three displacement 167 sensors were used. During the elastic modulus measurement, the compressive 168 maximum effort applied corresponds to 30% of the maximum compression strength. 169 The elastic modulus is determined according to equation (2). 170

The indirect tensile strength test was chosen so as to obtain the splitting tensile strength, which determined in accordance with NF EN 13286-42 [56]. For the indirect tensile test, the same specimens ( $\phi$  50 mm × h 100 mm) as those in the unconfined compressive strength test were used. The specimen was placed horizontally between two-plywood band, and a compressive force applied perpendicularly to the axis of symmetry of the cylinder specimen, as shown in Fig. 1 (c)-(d). The splitting tensile strength is calculated by the following equation (3).

178 
$$q_c = \frac{4F_c}{\pi D^2}$$
 (1)

179 
$$\mathbf{E} = \frac{1.2F_c}{\varepsilon_E \pi D^2} \tag{2}$$

$$q_{it} = \frac{2F_{it}}{\pi HD}$$
(3)

181 where  $q_c$  is the unconfined compressive strength;  $q_{it}$  is the splitting tensile strength; 182 F<sub>c</sub> is the maximum force of compressive test; F<sub>it</sub> is the maximum force of tensile test; 183 H and D are the length and diameter of the specimens;  $\varepsilon_E$  is the longitudinal strain of 184 the specimen, when F = 0.3 F<sub>c</sub>.

The crystallographic composition and microstructural properties of typical samples were investigated by X-ray diffraction (XRD) and mercury intrusion porosimetry (MIP) tests. Fragments taken from the broken samples after compressive strength tests were dried at 40 °C for at least 48 h. The XRD analysis was determined by the BRUCKER AXS D8 ADVANCE X-ray diffractometer with a Co Ka radiation source. The micropore structure analysis was defined by the MIP tests with a Micromeritics Autopore V porosimeter.

Samples	OPC (%)	CSA (%)	SD (%)	Curing time (d)
SD	0	0	100	3, 7, 28
SD2OPC	2	-	98	3, 7, 28
SD4OPC	4	-	96	3, 7, 28
SD6OPC	6	-	94	3, 7, 28
SD2CSA	-	2	98	3, 7, 28
SD4CSA	-	4	96	3, 7, 28
SD6CSA	-	6	94	3, 7, 28

**Table 1** Mix proportions of different samples.



199 Fig. 1 A view of the (a) unconfined compressive strength and (b) elastic modulus test;

200 (c) The side view and (d) the main view of indirect tensile strength test.

201

#### 202 **3. Results and discussion**

#### 203 **3.1. Physical and chemical characteristics**

The basic physical characteristics of sediments are shown in Table 2. The initial water content measured is about 5.20 %. It is worth to note that this value is related to the water content of the sediments after 3 months of natural drying. After dredging this later value can exceed 100 %. The true density, measured using a helium pycnometer is about 2.58 g/cm<sup>3</sup>. The organic matter measured by the ignition method is about 7.67 %. The low value of the true density could be explained by the high amount of

210	organic matter content measured. The liquid limit and plastic limit, defined by the
211	percussion-cup method and the rolling thread method are respectively 39.5 $\%$ and
212	28.0 %. The test results induce a plasticity index of 11.5 %. This result is typical of
213	previous studies on sediments dredged in the north part of France. The results for the
214	particle size distribution show that the sediments are composed of 6.97 % clay, 39.48 %
215	silt and 53.55 $\%$ sand. The low proportion of clays particle combined with the high
216	amount of organics matter content could explain the obtained value of liquid limit and
217	plastic limit.

For Ordinary Portland Cement (OPC), Calcium Sulfo-Aluminate cement (CSA) and

sediments used in this study, the chemical compositions are listed in Table 3.

220 Table 2 Basic physical characteristics of sedime	nts.
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Value	Test standard
5.20	NF P 94-050
2.58	NF EN ISO 17892-3
7.67	XP P94-047
	EN ISO 17892-12
39.5	
28.0	
11.5	
	EN ISO 13320
6.97	
39.48	
53.55	
	Value           5.20           2.58           7.67           39.5           28.0           11.5           6.97           39.48           53.55

**Table 3** Chemical composition of the materials. (wt %)

Components	MgO	$Al_2O_3$	SiO <sub>2</sub>	SO <sub>3</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>
SD	1.5	5.4	51.4	traces	1.2	12.1	0.3	4.9
OPC	1.0	4.8	19.9	3.3	1.0	64.1	0.3	2.6
CSA	1.1	17.4	8.5	13.7	0.3	40.5	0.9	7.3

#### **3.2. Compaction properties and I-CBR Index**

The effects of CSA and OPC binders on the compaction curves under the modified Proctor compaction test are shown in Fig. 2. The saturation degree curves for Sr = 80 %and Sr = 100 % are also presented in Fig. 2.

According to European standard NF EN 13286-2 [51], we can easily get the values of 228 maximum dry density and optimum moisture from the Proctor curve, which 229 corresponding to the coordinate of maximum position on the Proctor curve. It can be 230 seen from Fig. 2 that for the raw sediments, the maximum dry density is 1.641 g/cm<sup>3</sup>, 231 232 and the corresponding optimum moisture content is 21.8 %. For solidified sediments with OPC, the maximum dry density is highly reduced after 2 % addition of OPC 233 whereas for 4 % and 6 % addition the changes of dry density are less important 234 (decrease of maximum dry density from 1.578 g/cm<sup>3</sup> to 1.574 g/cm<sup>3</sup>). At the same 235 time with the decrease in the maximum dry density, the optimal moisture content 236 increases in the same way. For solidified sediments with CSA, as for OPC addition, 237 238 the maximum dry density is highly reduced after 2 % addition of CSA and at the same time, the optimal moisture content is increased. However, with an addition of 4 % and 239 6 % of CSA in the mixture, the maximum dry density seems to increase again and the 240 optimal water content decreases (an increase of maximum dry density from 1.575 241 g/cm<sup>3</sup> to 1.615 g/cm<sup>3</sup> and a decrease of optimal moisture content from 23.5 % to 242 20.2 %). These results are well highlighted in Fig. 4 (a)-(c). 243

Fig. 3 shows the I-CBR index curves of the raw sediments and OPC/CSA-solidified sediments. The measured I-CBR index values at the optimal moisture contents for all

246	the mixtures are shown in Fig. 4 (c). The I-CBR index value of raw sediments is about
247	15 % at the optimum moisture content. This value is lower than that for a sub-layer
248	material (I-CBR $\geq$ 25 %) according to the specifications in French standards [57]. The
249	addition of CSA to dredged sediments leads to an increase linearly of the I-CBR index
250	with the increase in the CSA content. After the addition of 2 % of CSA, the I-CBR
251	index value reach 20 $\%$ at the optimum moisture content and then increases to 30 $\%$
252	and 40 % as the CSA content increases to 4 % and 6 %. From this result, it could be
253	reasonable to predict that the treatment of sediments by 3 % of CSA could be
254	sufficient to reach an I-CBR index value of 25 % with is the due value to be used in
255	sub-layer material (I-CBR $\geq$ 25 %). To be used in the foundation layer (I-CBR $\geq$
256	35 %), treatment with 5 % CSA could be a reasonable value according to the
257	specifications in French standards [57].

For the OPC-solidified sediments, its I-CBR index values increase from 30 % to 45 % with the increase of the OPC content from 2 % to 6 %. From this result, it could be also reasonable to predict that the I-CBR index value of treated sediments with 1.5 % OPC could be sufficient to reach 25 %, which is the due value to be used in sub-layer material (I-CBR  $\ge$  25 %). To be used in the foundation layer (I-CBR  $\ge$  35 %), treatment with 4 % OPC could be a reasonable content.

It appears from these results that some of the solidified sediment specimens (include SD2OPC, SD4OPC, SD6OPC, SD4CSA and SD6CSA) present adequate properties for use as road material. However, the results of modified Proctor and I-CBR index tests reflect the behavior of solidified sediment in a short time (curing for less than 1 hour). To examine the mechanical behavior of solidified sediment in the long term
(curing for several days or even longer periods), it is necessary to measure the
mechanical characteristics in terms of compressive strength and/or tensile strength
and elastic modulus at long terms.







Fig. 3. I-CBR index curves of solidified sediments with (a) OPC; (b) CSA.







different samples.

#### **3.3. Unconfined compressive strength**

Fig. 5 shows the variation of unconfined compressive strength ( $q_c$ ) and unconfined compressive strength ratio ( $\eta_c$ , defined as the ration of unconfined compressive strength of solidified sediments  $q_{cn}$  to the unconfined compressive strength of raw sediments  $q_{c0}$  as in equation (4)) with the amount of treatment with OPC.

$$\eta_{\rm c} = \frac{q_{cn}}{q_{c0}} \tag{4}$$

For instance, under the condition of 28 d curing, the raw sediments (SD) display the 296 lowest  $q_c$  value (0.50 MPa), and the  $q_c$  of solidified sediments with 2 %, 4 % and 6 % 297 298 OPC inclusion increases to 0.76 MPa (SD2OPC), 1.40 MPa (SD4OPC) and 1.91 MPa (SD6OPC). Meanwhile, the  $\eta_c$  correspondingly increases to 1.52 (SD2OPC), 2.80 299 300 (SD4OPC) and 3.82 (SD6OPC) as compared with that of SD (1.00). In addition, 301 under the condition of the 4 % OPC content, the q<sub>c</sub> of solidified sediments (SD4OPC) is respectively 1.05 MPa, 1.14 MPa and 1.40 MPa at 3 d, 7 d and 28 d, while the 302 corresponding  $\eta_c$  changes from 2.10 to 2.28 and 2.80. This experimental result is 303 304 consistent with the previous study performed [9], [11].



**Fig. 5.** (a) Unconfined compressive strength and (b) strength ratio of solidified

16

For CSA-solidified sediments specimens, the  $q_c$  and  $\eta_c$  are reported in Fig. 6. It is 310 similar to OPC-solidified sediments specimens, both CSA content and curing time 311 lead to possibly changing trend of the  $q_c$  and  $\eta_c$ . After the same curing time of 28 d, 312 the q<sub>c</sub> of CSA-solidified sediments specimens increases from 0.50 MPa (SD) to 0.59 313 MPa (SD2CSA), 1.20 MPa (SD4CSA) and 1.71 MPa (SD6CSA) with the increase of 314 CSA content from 0 % to 6 %, the  $\eta_c$  correspondingly increases from 1.00 (SD) to 315 316 1.18 (SD2OPC), 2.40 (SD4OPC) and 3.42 (SD6OPC). Under the condition of 4 % CSA inclusion, the qc of SD4CSA specimens is 1.04 MPa (3 d), 1.08 MPa (7 d) and 317 1.20 MPa (28 d) with the curing time changes from 3 d to 28 d, while the 318 319 corresponding  $\eta_c$  is 2.07 (3 d), 2.17 (7 d) and 2.40 (28 d), respectively. However, for solidification of sediments as road material, comparing the effects of 320 OPC and CSA treatment under the same condition (binder content and curing time), it 321 322 can be seen that the CSA performs a little worse than OPC from the point of view of engineering performance capacity. The CSA did not show its high early strength 323 property for solidification of sediments in this study. This may be due to the organic 324 matter and/or metallic pollution in the sediment, which could influence the hydration 325 reaction of CSA [58]. Even the type of CSA cement may decide its different strength 326 performance [44]. Indeed, the compressive strength of solidified sediments with CSA 327 still can be reached to 78.41 - 98.42 % that of using OPC in this study. Thus, 328

- 329 considering the main advantages of CSA green, low carbon, environmentally
  - 17

friendly, and energy saving, we still consider CSA as an effective alternative for 330 solidification of sediments to replace traditional binder OPC. 331



332

Fig. 6. (a) Unconfined compressive strength and (b) strength ratio of solidified 335 sediments with CSA. 336

337

#### 3.4. Elastic modulus and secant modulus 338

Elastic modulus is an important characteristic to evaluate the ability of solidified 339 sediments to resist deformation. As illustrated in Fig. 7, a typical stress-strain curve 340 showing how to determine elastic modulus. It is evident that this typical stress-strain 341 curve can be divided into three stages: (I): initial elastic deformation stage; (II): 342 plastic deformation stage; (III): post-failure stage. The elastic modulus (E) can be 343 obtained from the linear portion (stage I) of the stress-strain curves at 30% of peak 344 stress [55]. Each result consisted of three measurements. 345





Fig. 7. A determined example of elastic modulus and secant modulus in a typicalstress-strain curve.

351 The variations of elastic modulus E and their error bars of solidified sediments with different binder contents and curing time are plotted in Fig. 8. The results presented 352 indicate that the variation in the elastic modulus has an upward trend as the increase 353 354 of binder content and the curing time. After 28 d of curing, the elastic modulus of OPC-solidified sediments increases by 2.33 times (0.50 GPa), 7.87 times (1.33 GPa) 355 and 8.53 times (1.43 GPa) as compared with raw sediment (0.15 GPa), with 356 increasing OPC content from 2 % to 6 %. Additionally, the SD4OPC specimen has a 357 higher E values after 7 d and 28 d curing than that at 3 d curing time, E changed from 358 0.76 GPa (3 d) to 1.18 GPa (7 d) and 1.33 GPa (28 d). A similar conclusion can be 359 observed for CSA-solidified sediment specimens, and both CSA content and curing 360 time had a great influence on the elastic modulus. Higher CSA content and longer 361 curing time benefit for the developing of elastic modulus, such as the specimens cured 362 28 d, they achieved the higher E values of 0.41 GPa (SD2CSA), 0.86 GPa (SD4CSA) 363 and 1.07 GPa (SD6CSA) when the CSA contents were 2 %, 4 % and 6 %, respectively, 364

while the E values was only 0.15 GPa for SD. Besides, with the curing time increase from 3 d to 28 d, the E values of CSA-solidified sediment samples were increases significantly, the E values of SD4CSA still as high as 0.64 GPa (3 d), 0.77 GPa (7 d) and 0.86 GPa (28 d). Achour et al. [59] also reported similar conclusions, elastic modulus increases with the increase of binder content and curing time.





373

Fig. 8. Elastic modulus of solidified sediments with (a) OPC; (b) CSA.



382 (i) Stage I, which corresponding to the initial elastic deformation stage of stress-strain

curves,  $E_{sec}$  increases a little at low strains in this stage. It is worth to note that  $E_{sec}$ should be constant in this stage due to the stress increases linearly with the strain in the initial elastic deformation stage, theoretically. However, because of the contact between specimen and press machine is incomplete at the initial stage, resulting in the stress increases approximately linear with the strain in the experiment, this is the reason that  $E_{sec}$  increases a little at low strains in this stage.

(ii) Stage II,  $E_{sec}$  decreases by the increases of axial strain until the point that corresponding to the peak stress point of stress-strain curves, therefore, it was considered corresponding to the plastic deformation stage of stress-strain curves.

392 (iii) Stage III, which means the post-failure stage of stress-strain curves,  $E_{sec}$  sudden 393 decrease significantly with increases of the strain.

However,  $E_{sec}$  becomes larger for solidified sediments with binder than raw sediments, the higher the binder amount and curing time, the higher the  $E_{sec}$  at the same strain. The reason for this event can be explained as the hydration reaction of binder, as the reaction goes on with more binder amount and curing time, the systems produced a large number of hydration products, include the sample particles interact with each other, increase the  $E_{sec}$ .

It should be noted that the values of elastic modulus and secant modulus differ by an order of magnitude (GPa and MPa). This is because the axial deformation data of the elastic modulus tests comes from three displacement sensors, which were stuck on to the specimens to record the true axial deformation of the specimens. However, the axial deformation data used for the calculation of the secant modulus tests comes from the press machine; the measured axial deformation of the top and bottom surfaces is greater than the actual axial deformation of the specimen, lead to measured secant modulus much smaller than the true elastic modulus of the specimen. This is also why European standard NF EN 13286-43 [55] recommends this method to measure the true elastic modulus of the specimen.



Fig. 9. Secant modulus curves of solidified sediments (a) Sediments with OPC cured
28 d; (b) Sediments with CSA cured 28 d; (c) SD4OPC cured for 3 d, 7 d, and 28 d; (d)
SD4CSA cured for 3 d, 7 d, and 28 d.

#### 419 **3.5. Splitting tensile strength**

Fig. 10 summarizes the effect of binder content and curing time in terms of the splitting tensile force-displacement behavior of solidified sediments. In general, the results reveal some common features: all the curves indicate a brittle behavior, no matter of raw sediment or solidified sediments; the force increases immediately with increasing displacement until reaching the peak splitting tensile force value, then reduce immediately followed by an increase of the displacement.

426 Indeed the peak tensile force and stiffness increase significantly with the increase of binder content and curing time. For 28 d curing specimens, the peak tensile force of 427 SD is 436.54 N, the peak tensile force of solidified sediments increase to 610.41 N 428 429 (SD2OPC), 1214.26 N (SD4OPC) and 1427.41 N (SD6OPC), due to the addition of 2 %, 4 % and 6 % OPC binder. Moreover, the addition of 2 %, 4 % and 6 % CSA 430 binder to the sediments increases the peak tensile force to 453.01 N (SD2CSA), 431 432 869.69 N (SD4CSA) and 934.21 N (SD6CSA) in relation to the raw sediments after 433 28 d curing. For SD4OPC (Fig. 10c) and SD4CSA (Fig. 10d), it seems that the peak tensile force increase significantly with curing time increase from 3 d to 28 d. The 434 peak tensile force increases to 482.39 N (3 d), 558.00 N (7 d) and 1200.82 N (28 d) 435 for SD4OPC and to 651.08 N (3 d), 786.05 N (7 d) and 914.69 N (28 d) for SD4CSA. 436 437



Fig. 10. Splitting tensile force-displacement curves of solidified sediments (a)
Sediments with OPC cured 28 d; (b) Sediments with CSA cured 28 d; (c) SD4OPC
cured for 3 d, 7 d, and 28 d; (d) SD4CSA cured for 3 d, 7 d, and 28 d.

Fig. 11 shows the variation of the splitting tensile strength ( $q_{it}$ ) and the tensile strength ratio ( $\eta_{it}$ , defined as the ratio splitting tensile strength of solidified sediment q<sub>itn</sub> to the splitting tensile strength of raw sediment q<sub>it0</sub> as shown on Equation (5)) with the amount of binder treatment and curing time.

$$450 \qquad \eta_{it} = \frac{q_{itn}}{q_{ito}} \tag{5}$$

It can be observed that the OPC inclusion contributes to the enhancement of the 451 splitting tensile strength of solidified sediments. In the same curing time of 28 d, the 452 splitting tensile strength q<sub>it</sub> increases from 0.050 MPa (SD) to 0.077 MPa (SD2OPC), 453 0.162 MPa (SD4OPC) and 0.192 MPa (SD6OPC) with the increase of OPC content 454 from 0 % to 6 %, the  $\eta_{it}$  correspondingly increases to 1.54 (SD2OPC), 3.24 (SD4OPC) 455 and 3.84 (SD6OPC) as compared with that of SD (1.00). Moreover, the increase of 456 curing time can effectively improve the  $q_{it}$  and  $\eta_{it}$  of the solidified sediments with 457 OPC. The splitting tensile strength of SD4OPC specimen increases to 0.109 MPa (7 d) 458 459 and 0.162 MPa (28 d) as compared with that at 3 d (0.104 MPa), meanwhile, the corresponding  $\eta_{it}$  changes to 2.18 and 3.24 as compared with that of SD4OPC 460 specimen at 3 d (2.08). 461

462



**Fig. 11.** (a) Splitting tensile strength and (b) tensile strength ratio of solidified

466

467

468 Fig. 12 shows the splitting tensile strength values and tensile strength ratio of 25

sediments with OPC.

469 CSA-solidified sediments specimens cured for 3 d, 7 d and 28 d. As expected, the
470 CSA has an obviously positive effect on the tensile strength during all times. This is
471 because the CSA with water systems produced a large number of hydration products,
472 which are formed filled pores to give a higher strength.

Especially for the specimens with 2, 4 and 6 % CSA at 28 d, which had the higher 473 tensile strength of 0.057 MPa, 0.114 MPa and 0.119 MPa than that of SD (0.050 MPa), 474 and the n<sub>it</sub> correspondingly is 1.14 (SD2OPC), 2.28 (SD4OPC), 2.38 (SD6OPC) and 475 1.00 (SD). This indicated that the addition of CSA could significantly improve the 476 tensile strength of the solidified sediments. As the reaction proceeds, there was an 477 increase in tensile strength for SD4CSA specimens after cured, and the tensile 478 strength achieved the most about 0.082 MPa, 0.100 MPa and 0.114 MPa at 3 d, 7 d 479 480 and 28 d, respectively, while the n<sub>it</sub> correspondingly is 1.64 (3 d), 2.00 (7 d) and 2.28 (28 d). According to the research of Zhang et al. [62], the ettringite is the main 481 hydration product and plays a significant role in improving the strength of CSA 482 treated specimens, and ettringite continues to increase with curing time and CSA 483 content. Thus, this is the reason that the splitting tensile strength of CSA-solidified 484 sediment specimens increases with the increasing of curing time and CSA content. 485

486



490 Fig. 12. (a) Splitting tensile strength and (b) tensile strength ratio of solidified491 sediments with CSA.

#### 493 **3.6.** Relationship of mechanic parameters q<sub>c</sub>, q<sub>it</sub> and E

In this study, a regression analysis was performed on the relationship between the compressive and tensile splitting strengths values, as shown in Fig 13 (a). Based on this figure, a simple linear model has been used for describing the relationship between the compressive and tensile splitting strengths of solidified sediments. Equations (6)-(7) have been derived for describing the relationship between the compressive and splitting strength of OPC solidified sediments and CSA solidified sediments in this study.

501  $q_c = 9.5q_{it}$  (6)

502 
$$q_c = 12.2q_{it}$$
 (7)

These equations are plotted in Fig. 13 (a) with the experimental data. It can be seen that the regression lines from equations (6)-(7) showed a relatively good relationship between the compressive and splitting strength. The coefficient of determination ( $\mathbb{R}^2$ ), which indicates how much of the total variation in the dependent variable can be accounted for by the regression equation, was obtained as 0.99 and 0.98 for equations (6)-(7) in this study, respectively. In particular, it is found that a unified equation (8) with  $R^2 = 0.97$  can satisfactorily describe the correlation between  $q_c$  and  $q_{it}$  on all solidified sediment specimens. Therefore, the derived linear equations may successfully be used to represent the relationship between the compressive and splitting strength of solidified sediments.

513 
$$q_c = 10.4q_{it}$$
 (8)

Fig 13 (b)-(c) show the variation of compressive strength  $q_c$  versus elastic modulus E 514 and splitting tensile strength q<sub>it</sub> versus elastic modulus E, respectively. It is observed 515 that both qc and qit have a trend to increase with the increasing E according to the 516 517 obtained test data points. In this study, the simple exponential model was used for describing the relationship between the strength and elastic modulus. The model 518 results between qc and E of OPC- and CSA-solidified sediments are reflected in 519 equation (9) and (10) with the coefficient of determination  $R^2 = 0.93$  and 0.98, 520 respectively. The unified equation (11) with  $R^2 = 0.92$  can explain how compressive 521 strength is related to the elastic modulus of solidified sediment specimens. 522

523 
$$q_c = 0.471e^{0.931E}$$
 (9)

524 
$$q_c = 0.407 e^{1.313E}$$
 (10)

525 
$$q_c = 0.454e^{1.040E}$$
 (11)

Equation (12) and (13) with  $R^2 = 0.94$  and 0.95, respectively, are derived from the regression analysis to express quantitatively the correlation of  $q_{it}$  and E of OPC- and 528 CSA-solidified sediments. A unified equation can be expressed by equation (14) with 529  $R^2 = 0.94$ , which can be acceptable to describe approximately the changing trend of 530 splitting strength with elastic modulus on the designed materials.

531 
$$q_{it} = 0.046e^{0.997E}$$
 (12)

532 
$$q_{it} = 0.043e^{0.990E}$$
 (13)

533 
$$q_{it} = 0.044e^{1.011E}$$
 (14)

In general, equation (8), (11) and (14) demonstrates the relationships among the 534 compressive strength, tensile strength and elastic modulus, this is to say, these 535 536 equations provide an alternative approach to predict the approximate value of other two parameters if one parameter is given. By example, this leads to help to predict the 537 tensile strength and the elastic modulus from the compression strength. This later test 538 539 is easier to perform, cheaper and quicker to carry out in comparison to the tensile strength and the elastic modulus measurements. Moreover, in the stage of designing a 540 new formulation, this procedure could save time, reduce the number of samples and 541 hence save the materials. Even if the ratio of 10 between the compressive strength 542 over the tensile strength is commonly encountered for OPC treated materials, the 543 relation between the tensile strength or the compressive strength with the young 544 modulus has to be specific to a given material (in our case to treated sediments). 545



**Fig. 13.** Relationship of (a) compression and splitting tensile strength; (b)

compression strength and elastic modulus; (c) splitting tensile strength and elastic

modulus.

#### 557 **3.7 Evaluation of solidified sediments as road materials**

Fig. 14 assess the suitability of solidified dredged sediments as road materials according to European standard NF EN 14227-15 [63], using the mechanical characteristics of the direct tensile strength ( $q_t$ ) and elastic modulus (E) at 360 d of the solidified dredged sediments.

It is noted that the direct tensile strength qt shall be derived from qit using the Equations (15) [56] [63]. The estimated values at 360 d for solidified sediments with cement should be calculated from the measured values at the curing period of 28 d, according to Equations (16) and (17) [8] [59]. In the absence of specific data, the coefficients 0.6 and 0.65 are assumed to remain valid for solidified sediments. The use of this extrapolation was confirmed in the literature. The results from Wang et al. [8] and Achour et al. [59] confirmed this extrapolation.

569 
$$q_t = 0.8q_{it}$$
 (15).

570 
$$\frac{q_t - 28d}{q_t - 360d} = 0.6$$
 (16)

571 
$$\frac{E-28d}{E-360d} = 0.65$$
 (17)

However, a great improvement of material classification can be observed, compared with raw sediments. This proves the beneficial effects of OPC and CSA treatment to increase the mechanical properties of raw sediments. It should be noted that the minimum class required for potential use as a sub-base material in class T2. Hence, this means that no samples in this study can be used as a sub-base or base course material for a large class of traffic intensities. The samples of SD4OPC and SD6OPC, which belong to the class T1 might be possibly reused as roadbed filling materials for roads of low traffic intensity. This conclusion is consistent with the previous study [8]. If the need arises to enhance the material classification to Class T2 for high traffic intensity, the effective way may be to increase the amount of binder and/or to explore the combined effect of OPC and CSA, finally to combine the present sediments with coarser materials to improve its mechanical properties. This work will be carried out in a next stage.





**Fig. 14.** Classification of solidified sediments with OPC and CSA.

588

586

#### 589 **3.8 X-ray diffraction analysis**

590 X-ray diffraction (XRD) analysis was used to determine the mineralogical 591 composition of raw and OPC/CSA-solidified sediments at 7d, and the representative 592 results are given in Fig. 15. For raw sediments, quartz (SiO<sub>2</sub>) and calcite (CaCO<sub>3</sub>) 593 were observed as the main phases, the result also indicated some minor phases such as 594 gypsum (CaSO<sub>4</sub>), kaolinite, muscovite, microcline, and albite. The main hydration

phases observed of OPC-solidified sediments (SD6OPC) are portlandite (CaOH<sub>2</sub>) and a little of ettringite (Ca<sub>6</sub>Al<sub>2</sub>S<sub>3</sub>O<sub>50</sub>H<sub>64</sub>), however, calcium silicate, calcium aluminate and aluminosilicate phases were not detected. XRD results reveal that the ettringite (Ca<sub>6</sub>Al<sub>2</sub>S<sub>3</sub>O<sub>50</sub>H<sub>64</sub>) is the main hydration product in CSA-solidified sediments (SD6CSA). The XRD results show the different main hydration product for OPC and CSA-solidified sediments, lead to the different mechanical strength of solidified sediments.





Fig. 15. XRD traces of raw sediments, OPC-solidified sediments and CSA-solidifiedsediments specimens.

606

603

#### 607 **3.9 Micropore structure analysis**

The micropore structure of the solidified sediments was investigated and the results is shown in Fig. 16 and Table 4. It's easily observed that the four typical samples showed approximately the same pattern with apparent diameters of pores in the range of 0.006-400  $\mu$ m, and all the pore size distributions model are bimodal. Indeed, the main pores of all the samples distributed in the range of 0.01–1.0  $\mu$ m and 1.0–40  $\mu$ m.

According to previous research [64], the pore structure can be divided into four major 613 ranges:  $<0.01 \mu m$ , 0.01-1  $\mu m$ , 1-40  $\mu m$  and  $>40 \mu m$ , as presented in Table 4. With the 614 curing time increase from 3 d to 7 d, the pores volume percentage of larger pores in 615 the range of 1-40  $\mu$ m and >40  $\mu$ m shows a decreasing trend, while the pores volume 616 percentage of the smaller pores in the range of <0.01µm and 0.01-1 µm shows an 617 increasing trend. This is due probably to the progress of hydration of binders, which 618 causes that the larger pores (1-40  $\mu$ m and >40  $\mu$ m) transform to smaller pores 619 (< $0.01\mu$ m and  $0.01-1\mu$ m), which induces that the pores of the solidified sediments 620 became finer and denser. 621



623

624



**Fig. 16.** Micropore structure analysis of (a) OPC-solidified sediments and (b) and

628

CSA-solidified sediments.

629

**Table 4** Pore size distribution of the solidified sediments

Doro sizo (um)	Pore size distribution (%)					
Pole size (µIII)	SD6CSA-3d	SD6CSA-7d	SD6OPC-3d	SD6OPC-7d		
>40	2.80	2.72	4.72	3.18		
1-40	34.51	33.88	33.12	32.30		
0.01-1	60.78	61.82	59.53	61.06		
<0.01	1.91	1.57	2.63	3.45		
Total	100	100	100	100		

631

#### 632 **4. Conclusion**

In this study, a series of experiments were performed to clarify the feasibility of recycling dredged marine sediments with novel eco-binder CSA cement as road construction materials. The results are compared with the OPC treated sediments. Based on the obtained results, the following conclusions can be drawn:

(1) Compared with the raw sediments, the compaction performance of the solidified
sediments is significantly improved, which reflects the behavior improvement of
solidified sediment in a short time that curing for less than 1 hour. The increase in the
I-CBR index value is almost linear with the increase of the binder content (at least in
the range of binder content studied in this work). In terms of maximum dry density
and the optimum moisture of solidified sediment:

643

a. For OPC treatment, the maximum dry density is decreased

644 accompanied by increasing optimal water content.

b. For CSA treatment, after the first decrease of the treatment with 2 %
CSA, an increase in maximum dry density is observed for higher
treatment content. In the same way, after an increase in the optimal
water content of the treatment with 2 % CSA, a decrease is observed
for higher treatment content.

(2) The incorporation of binder in raw sediments is effective for the improvement in
mechanic behavior of solidified sediment in the long term that curing for several days
or even longer periods, includes compressive strength, tensile strength and elastic
modulus, and these mechanical parameters increase with binder content and curing
time.

(3) A relative evaluation of the effectiveness of treatment with OPC and CSA has
revealed that CSA increases relatively the mechanical properties of solidified
sediments in the early stage (during the first three days). Whereas OPC shows a
marked increase between 3 d and 28 d.

(4) The simple model can satisfactorily describe, respectively, the relationships among the compressive strength, tensile strength and elastic modulus; these equations provide an alternative approach to predict the approximate value of two parameters if the one parameter is given. Which leads to reduce the number and time of the experiments. It is to note that the relation between the strength and the elastic modulus is material dependent.

665 (5) The microstructure induced by OPC treatment and CSA treatment seems to be

666 comparable. The pore size distribution induced is of type bimodal.

667 (6) The evaluation of solidified sediment classification confirms that valorization668 sediment in road materials is entirely possible.

669

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