

# From dredged sediment to supplementary cementitious material: characterization, treatment, and reuse

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1	From dredged sediment to supplementary cementitious material:
2	characterization, treatment and reuse
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8	ABSTRACT
9	Nowadays, the preservation of natural resources, the reuse, and the recycling of waste and by-products

in the cement industry are gaining increasing attention in a sustainable development project. In this perspective, many studies focus on finding solutions in order to propose environmentally friendly materials. Nowadays the sediments represent a way to preserve the natural resources by their reuse as a secondary raw material in multiple applications (concrete, roads, landscaped mound, etc...). However, they commonly contain contaminants, organic matter (5% to 30%), high water content (50% to 200%), and relatively small particle size (Dmax  $\leq$ 300 microns). Therefore to improve the mechanical properties of this material as well as its physiochemical ones, specific methods of characterization and appropriate treatment techniques (calcination, chemical treatment, etc...) are required. This article presents a bibliographical review of the efficient use of sediments in cementitious matrix. It details experimental tests that must be performed to ensure the durability of sediment-based structure and assess their environmental impact under prescribed conditions. 

**KEYWORDS:** Sediment - Characterization - Calcination - Pozzolanicity - Durability.

## 40 **1. Introduction**

Sediments are materials coming from dredging marine operation, continental watercourse, and can also be defined as fine material from wear of continents. Sediments are formed by deposits, particles introduced into the ecosystem, and precipitates formed by biochemical processes in aquatic environments (Capilla et al., 2006). Dredging activities inside ports and harbors, also their maintenance and expansion, generate huge volumes of dredged soils called sediments that are usually associated with high water content, high compressibility due to organic matter, and the presence of salt and contaminants (He et al., 2020).

Considerable volumes of material are dredged annually in the world. Annually 400 million tons of 48 natural granular material are consumed in France in the construction sector compared to 40 billion 49 tons in the world. In France, more than 56 million  $m^3$  of sediment are dredged per year, against 200 50 million m<sup>3</sup> in Western Europe (CEDA<sup>1</sup> - 2008), 100 million m<sup>3</sup> in China and 300 million m<sup>3</sup> in the 51 USA (Oh et al., 2011). Nowadays, for sustainable development reasons, the reuse of materials is 52 strongly encouraged. There are actually three levers for sustainable construction which are: reduction, 53 recycling, and reuse of raw materials (Marie & Quiasrawi, 2012). Dredged sediments seem to be 54 potentially useful as secondary raw material in various civil engineering sectors. These include road 55 56 engineering by their use in foundation layer (Siham et al., 2008a), manufacturing of building bricks (Cappuyns et al., 2015) and their use as alternative materials by partial substitution to cement or sand 57 58 in concrete or mortars (Belas et al., 2011; Oh et al., 2011). Moreover, sediments have been used in some countries in important infrastructures: Rotterdam port, the Palm island in Dubai, National 59 Theatre of London, or the artificial island of Chek Lap Kok where the Hong Kong airport is located 60 (Limeira & Etxeberria, 2011). Due to the increase of global cement production in the world that will 61 reach 5 billion tons by 2020, the CO<sub>2</sub> emissions from the cement industry will therefore increase 62 significantly. This CO<sub>2</sub> emission represents 6% of the total anthropogenic greenhouse gases (Gartner 63 & Hirao, 2015; Scrivener et al., 2018; Zea-Garcia et al., 2019). In fact, the production of ordinary 64 Portland cement (OPC) generates 842 kg CO<sub>2</sub>/t of clinker. The use of sediment as supplementary 65

66 cementitious materials (SCMs) represents one alternative to reduce cement production (Scrivener et 67 al., 2018; Snellings et al., 2017; Van Bunderen et al., 2019). However, because of their specific 68 properties and their frequent heterogeneity, they may require appropriate treatment that will probably 69 improve some of the physicochemical and mechanical properties (Tribout et al., 2011).

The use of sediment in the cement matrix leads to the modification of some properties of cementitious 70 material (strength, durability, etc.) (Anger et al., 2014; Belas et al., 2011; Benkaddour et al., 2009; 71 Rodríguez et al., 2013). This modification could be linked to the composition, high water demand, the 72 physicochemical nature of the sediments (contaminants), the impact of the organic fraction, and some 73 clay properties such as absorption. In fact, sediments are characterized by the presence of mineral fine 74 particles with a few small rock parts mixed with vegetal or animal debris. Therefore their water 75 demand is relatively high. The compactness and the heterogeneous nucleation during hydrate 76 formation is impacted by the fine fraction (Cyr et al., 2005; Lawrence et al., 2005). Moreover, 77 previous studies have shown the presence of a pozzolanic effect when sediment is used as a mineral 78 addition in cementitious matrix (Scrivener et al., 2017, 2018). Besides, due to their origin, there is the 79

80 presence of mineral contaminants such as heavy metals (Pb, Cr, Cu, etc.) and organic contaminants

81 (PAHs, PCBs, etc.) or even the organic fraction which enhance the development of humic clayey

82 complex (Cabane, 2004). As a result, different treatment methods have been put in place to improve

83 the performance of sediment-substituted cementitious materials. Those methods will be discussed

84 further in this paper.

85 Furthermore, it is necessary to assess the sustainability and environmental impact of the mix design.

Therefore to classify and evaluate the impact of these particles on cement matrix behavior and abilities, a series of tests and methods can be realized (XRF analysis, leaching test, etc.).

88 This paper presents a review of the valorization of sediment as supplementary cementitious material in 89 cement-based materials. The methods and standards applied to characterize the sediment are

presented, as well as the different treatment methods conducted on sediments to be used as SCMs. The

91 performances, the behavior, and the durability of the cementitious matrix that contain sediments will

92 be presented in this article.

# 93 2. Sediment based materials characterizations and testing

The study of material characteristics should be focused on the measurement and the determination of 94 physical and chemical parameters (see Table 1). In the case of sediments, the proposed tests are based 95 on standards and experimental methods. The determination of these parameters leads to consider the 96 use of sediments in cementitious matrix. The formulation of mortars or concretes containing sediments 97 98 includes the use of different types of materials: cement, sand, water, sediments, and possibly mineral additions or admixture. A suitable cement may be a common Portland cement type CEM I 52.5N for 99 instance. If specific applications are required, the use of other types of cement could also be 100 considered. 101

Concerning the physical characterization, the density of the materials can be achieved using a helium 102 pycnometer that determines the specific gravity (g/cm<sup>3</sup>). This test can be conducted under NF EN 103 1097-7 standard. Also, the specific surface area (BET) based on the Brunauer-Emmett-Teller method 104 is measured according to the standard NF EN ISO 18757 appreciating the finesse of materials. For 105 106 fine particles ( $d_{80}$ <80µm) as sediment, a laser particle size should be used. For the formulation of standard mortar, it is necessary to have clean sand for which most coarse aggregate is less than 2mm 107  $(D_{max} \le 2mm)$ . Standardized sand ISO 679: 2009 compliant to NF EN 196-1 can be suitable. The 108 evaluation of the organic fraction can be performed by the loss on ignition test according to the 109 standard XP P94-047 that consists on calcination at 450°C or 550°C and the measuring of the mass 110 loss. Similarly, the total organic carbon (TOC) can be determined according to NF EN 15169, 111 particularly adapted to sludge and sediment material. The absorption test with methylene blue value 112 (BM) for the evaluation of the clay content must comply with the NF P 94-068 standard or equivalent. 113 Atterberg limits (liquid limit wL and wP plastic limit) are determined regarding the NF P94-051 114 standard that is performed on the fraction  $\leq 400 \ \mu m$  by the Casagrande apparatus and by the rolled 115 thread method. Atterberg limits are an important indicator for several properties of clayey soils such: 116 plasticity, sensitivity, consistency, and shrinkage/swelling potential (Bel Hadj et al., 2014). 117

Due to the contaminants with various origins and nature, it is interesting to identify chemical elements using analytical techniques such as inductively coupled plasma (ICP). The liquid sample is vaporized at very high temperatures in the presence of argon gas-induced plasma and causes atomization, ionization, and thermal excitation. These emissions are identified and assessed either by Optical Emission Spectrometry (OES), by measuring the emitted wavelengths; or by mass spectrometry (MS), by measurement of the mass of the released ionic particles (Dang et al., 2012).

124 It may be appropriate to conduct a mineralogical characterization of sediments mainly through an X-125 ray diffraction analysis (XRD). The analysis consists of measuring the intensity and diffraction angles 126 and gives the internal atomic structure. This test identifies and qualifies the mineralogical nature of

crystalline phases present in the materials. Several authors have already used this technique in the case 127 of sediments study (Rodríguez et al., 2013; Siham et al., 2008a; Xu et al., 2014a). This can be 128 supplemented by an X-ray fluorescence analysis (XRF). The XRF analysis, like the X-ray diffraction, 129 uses the principle of atomic excitation emission. Characteristic energy responses from particles allow 130 highlighting the chemical elements which are present in the sample. It is very often to carry scanning 131 electron microscopy (SEM) in addition to the above-cited analyses for visual observation and 132 characterization of some properties (texture, nano and microscale constituents, etc.). SEM analysis 133 134 consists of observation at a very high definition and very high depth. It provides high magnified view up to 500,000 times which allows analyzing the majority micron constituents of hardened cement such 135 as CSH (<100 Å), the strips of clayey particles (<10Å), the ettringite crystals (<50  $\mu$ m) and portlandite 136 crystals Ca(OH)<sub>2</sub> (<10 µm) (Fernandez et al., 2011; Maso, 2004; Siham et al., 2008a; Xu et al., 137 2014a). 138

The formulated mortars should be characterized to determine their major physicochemical properties and mechanical strength. Bending and compressive strength tests can be performed using prismatic test sample 4x4x16 cm (NF P 196-1 standard), on cylindrical specimens 11x22 cm (NF P18-400 standard) or 16x32 cm (NF P18-400 standard). The size of the specimen is generally a function of the coarser aggregate (Dmax). Mercury Intrusion Porosimetry (MIP) is extensively used to characterize the pore structure in porous material as a result of its simplicity, quickness, and wide measuring range of pore diameter (Riahi & Nazari, 2011).

Finally, the durability can be evaluated by realizing a carbonation test, sulfate attacks, penetration of chloride ions test, air porosity, and alkali-aggregate reaction. Environmental testing methods embedding leaching tests can be also performed to determine the leached chemical component and their proportion (heavy metals, hydrocarbons, PAHs, PCBs, chlorides, etc.).

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Table 1: Characterization methods recommended for sediment studies

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Parameters Characterization methods			Standards	
	Granulometry	Laser particle size distribution	NF ISO 13320-1	
	Density	Helium pycnometer	NF EN 1097-7	
	Specific surface area	Brunauer-Emmett-Teller	NF EN ISO 18757	
Dhysical	Water content	Water content test	NF P94-050	
Physical	Clay content	Methylene blue value test	NF P94-068	
characterization	Plastic limit	Casagrande apparatus	NF P94-051	
	Liquid limit	Rolled thread method	NF P94-051	
	Mass Loss	Loss on ignition	NF EN 15169 NF EN 12879	
	Chemical elements	XRF		
Chemical	Oxide elements	XRF		
characterization	Carbon content	TGA		
	Mineral and organic pollutants	Leaching test	NF EN 12457-2	
Minanalagiaal	Identification of minerals	XRD		
Analysis and Microscopy	Quantitative determination of mineral	XRD Rietveld		
	Morphology	SEM		
	Resistance	Mortar formulation	NF P 196-1, NF P18-400	
Investigation	Strength	Compression and flexural test	NF P 196-1. NF P18-400	
methods for	U		NF EN 196-5	
material and	PozzolanIcity Pozzolanic activity tests	Pozzolanic activity tests : (Chapelle,	NF EN 196-6	
mortars	2	Frattini, Conductimetry)	ASTM C619-A	
	Dunchiliter	Freeze and thaw, carbonatation, sulfate	NF P18-454 XP P18-458	
	Durability	attack, chloride ion penetration, alkali-	NF P18-459 ASTM C1202-97	

Heat of hydrationLangavant calorimetryNF EN 196-9PorosityMercury Intrusion testISO 15901-1			aggregate reaction, gas permeability	XP P18-463 NF P18-425 NF P18-837 ASTM C452
Porosity Mercury Intrusion test ISO 15901-1	Heat	of hydration	Langavant calorimetry	NF EN 196-9
	Poro	sity	Mercury Intrusion test	ISO 15901-1

#### **3. Impact of sediments contaminants in cement matrix** 151

The morphology and composition of the organic material present in the sediment confer the typical 152 properties of the latter. When sediments are used in the cement matrix, a treatment to improve 153 sediment properties could be required. It was shown that the presence of organic matter is not without 154 consequences on the physical and chemical properties. For (Bennett et al., 1985) and (Tremblay et al., 155 156 2002), the presence of organic fraction can have a real impact on the Atterberg limits and it would also reduce the density of material (Abriak et al., 2014). The presence of organic matter may also influence 157 158 the hydration process. An effect on the setting and hardening due to the formation of a particular and protective film around the cement grains was observed (Paria & Yuet, 2006; Sora et al., 2002). 159

160 Besides, the presence of organic matter can decrease the compressive strength of mortars. Sediments can also induce a depassivation effect, a pH drop due to the Ca<sup>2+</sup> ions consumption necessary for 161 portlandite crystals formation, and a decrease in the organic molecules adsorption efficiency when 162 163 superplasticizer is used.

164 Interactions between the cement particles and mineral contaminants were identified (Fig. 1). According to (Yousuf et al., 1995) these interactions take place through exchange mechanisms of 165 transformation and more particularly by ion exchange, precipitation, substitution, etc... 166

The effect of metal contaminants has been studied by (Minocha et al., 2003; Park, 2000a). It has been 167 shown that the presence of inorganic contaminants in cementitious matrix has generally a detrimental 168 effect on the mechanical strength and in its durability properties as well (Minocha et al., 2003). 169 Furthermore, the presence of sea salts like NaCl and mineral pollutants leads, regardless of its 170 concentration or nature, to the modification of the bulk density of the cement matrix based on Portland 171 cement (CEM).

172 Some pollutants can negatively impact the compressive strength acting on the material porosity and on 173 the formation of CSH gel according to (Malviya & Chaudhary, 2006a). It have shown that in this case 174 the micropores (~1 $\mu$ m) decrease and were replaced by larger pores ( $\geq 10 \mu$ m).

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Figure 1: Interaction of pollutants with the cement particles (based on (Yousuf et al., 1995))

- In the following the effect of some elements on the cement-based material properties is presented: 179
- Lead (Pb) may precipitate on the hydrates surface (Park, 2000a) and may induce a decrease in the 180 compressive strength. This decrease is not significant for (Minocha et al., 2003). 181
- Chromium (Cr) may be absorbed by the hydration products such as CSH (Park, 2000b) and may also 182 take place in pore formation mechanisms. 183
- *Cadmium* (*Cd*) can replace the  $Ca^{2+}$  ion and leads to the formation of CdCa(OH)<sub>4</sub> ions (Fig. 1). 184
- Nitrate (Ni) leads to a decrease in hydration efficiency by more than 50% while reducing the total 185 porosity (Malviya & Chaudhary, 2006a). 186
- Mercury (Hg) has no significant effect on the total pore volume (Malviya & Chaudhary, 2006a, 187 188 2006b).
- Copper (Cu) can induce a drop in compressive strength when its concentration is above 8% but 189 under the latter concentration, it may have a beneficial effect (Minocha et al., 2003). 190

• *Zinc* (*Zn*) has the greatest impact out of all the other minerals pollutants. Its presence leads strongly to lower the compressive strength and a decrease in material density. For (Arliguie & Grandet, 1990), Zn inhibits the hydration of  $C_3S$  and  $C_3A$  with a more pronounced effect on  $C_3S$ . This phenomenon is due to the formation of an amorphous impermeable film around anhydrous grains named zinc hydroxide (Zn(OH)<sub>2</sub>) (Eq. 1 and Fig. 1).

$$Zn^{2^{-}} + 2(OH)^{-} \Longrightarrow Zn(OH)_{2}$$
<sup>(1)</sup>

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However, the presence of  $Ca^{2+}$  and  $OH^{-}$  ions allows the destruction of the seal by forming a calcium hydroxyzincate layer and therefore the hydration reactions continue (Eq. 2).

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$$Zn(OH)_4^{2^-} + Ca^{2^+} + 2H_2O \Longrightarrow \left\langle CaZn(OH)_4, 2H_2O \right\rangle$$
(2)

Marine salts such as sodium hydroxide (NaOH) or sodium sulfates with concentrations below 2%
 cause an increase in short term resistance but this effect is reversed when concentrations exceed 8%
 (Minocha et al., 2003).

Generally, the formation of metal hydroxides inhibits cement hydration (Fig. 2, Fig. 3), thus impacting
the correct formation of tobermorite gel (CSH) by precipitation-complexation process (Hills &
Pollard, 1997a; Malviya & Chaudhary, 2006b).

The sulfate and chloride can have an acceleration effect on hydration processes. However, trough exposure to saline water,  $Cl^-$  ions penetrate the structures and subsequently lead to corrosion in reinforced structures by a reducing passivity layer (Barberon et al., 2005; Basheer et al., 2001).

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Figure 2: Solidification/stabilization of pollutants around a cement grain (Yousuf et al., 1995).

Figure 3: Inhibition mechanism of cement hydration by pollutants (Hills and Pollard, 1997b)

## **4.** The use of sediments as SCMs

The literature discusses the use of sediments as SCMs with and without treatment. Here in this section the two methods, with and without treatment, are presented.

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#### **4.1 Use of sediment without treatment**

The literature shows that sediment can be valorized as a construction material without being treated 223 (Table 2). (Achour et al., 2019) used the marine sediments from the port of Dunkirk without any 224 treatment to manufacture concrete accropode blocks to protect the harbor coastline. The results 225 showed that the substitution by 12.5% of fine marine sediment would be recommended to maintain 226 good mechanical properties under external sulfate attack and frost action. The authors used also the 227 same sediment to show their ability to be used in road construction projects (Achour et al., 2014). 228 (Zhao et al., 2018) used the uncontaminated dredged marine sediment of Dunkirk harbor without 229 230 treatment as well. The sediment was only dried and ground to substitute partially the cement (by 10, 20 and 30%) in the mortar and concrete manufacturing. The results show that the substitution with 231

20% of sediment improves the mechanical properties comparing to a CEM II/A -LL 32.5 containing 232 20% of limestone substitution. (Limeire et al., 2012) valorized the dredged marine sand (DMS) from 233 the Port of Sant Carles de la Ràpita (Tarragona, Spain) as a construction material for pastes, mortar, 234 and concrete production. It has been concluded from this study that the mechanical properties of 235 concretes made with dredged marine sand in substitution of fine sand (0/2 mm) were similar to those 236 of the reference concrete. In the same way, the untreated sediment from four Turkish harbors was used 237 by (Ozer-Erdogan et al., 2016) as a fine aggregate in the ready mixed concrete. It has been found that 238 the incorporation of untreated sediments in concrete decreases the mechanical performances due to the 239 presence of sulfates and chlorides ions. 240 241

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Table 2: Use of sediment without treatment in previous works in literature

Authors	Material origin/nature	Study purpose	
(Achour et al., 2019)	Dredged marine sediment from the Dunkirk Harbor	<ul> <li>Evaluation of the performance and the durability of concrete blocks incorporating 12.5% and 20% of dredged sediments.</li> <li>Sediment addition should be limited to 12.5% of the concrete mix to ensure the integrity of mechanical properties</li> </ul>	
(Zhao et al., 2018)	Dredged marine sediment from the Dunkirk Harbor	<ul> <li>Study of the use of raw sediment in partial substitution of cement in the manufacture of mortars and concretes</li> <li>It was demonstrated that concrete C30/37 could be designed with 20% cement replaced by sediment without the use of admixture.</li> </ul>	
(Limeira et al., 2012, 2013)	Dredged marine sand (DMS) from the Port of Sant Carles de la Ràpita (Tarragona, Spain)	<ul> <li>The study aimed to assess DMS as an addition (its influence on cohesion and fluidity) or as a fine granular corrector (its influence on compressive strength)</li> <li>Strength resistance improvement in mortars compared to the control mix was identified.</li> </ul>	
(Ozer-Erdogan et al., 2016)	Samples were dredged from İstanbul Ambarlı Port, Mersin Erdemli Fishery Harbor, İzmir PETKİM Container Port, and Samsun Port	<ul> <li>Sediments are used as a fine aggregate in the ready mixed concrete</li> <li>Incorporation of untreated sediments in concrete decreases the mechanical performances due to the presence of sulfates and chlorides ions</li> </ul>	
(Maherzi et al., 2020)	River sediment from Aire sur la Lys (North of France)	<ul> <li>River sediment is used and valorized</li> <li>Use of sediment in substitution of natural sand in resin mortars has been shown interesting</li> <li>Mechanical performance and physicochemical properties of products was satisfying</li> </ul>	
(Xu et al., 2014b)	Urban river sediments were produced by a dredging project in Qinhuai River (China)	<ul> <li>The feasibility of the use of urban river sediments as a primary raw material to produce high-insulation brick is established</li> <li>Bricks with 50% urban river sediments fired at 1,050 °C exhibited more beneficial characteristics: conductivity and compressive strength improvement.</li> </ul>	
(Liu et al., 2016)	Yellow River sediment comes from Huayuankou	<ul> <li>Microstructure and performance of stabilized earth bricks prepared from the Yellow River sediment is studied</li> <li>Compressive strength and softening coefficient of stabilized earth bricks is further improved by a polymer bonding agent</li> </ul>	
( Junakova et al., 2015; Junakova & Junak, 2017b)	Sediments dredged from the Klusov and Ruzin reservoirs situated in Eastern Slovakia	<ul> <li>Focus on the possibility of reusing sediment from two Slovak reservoirs as an alternative raw material in concrete production</li> <li>Mixtures containing 40% of binder replacement by the sediment satisfied the strength requirements</li> </ul>	
(Tang et al., 2011)	Fine sediment deposits dredged from the Shihmen	• The investigation revealed the sediments contain all the necessary elements to enable the bloating and calcining	

	Reservoir in Taiwan.	<ul> <li>processes within the commercial kiln</li> <li>Sediment-based materials designed have presented compressive strengths comparable to normal density concretes and were 29%–35% lighter.</li> </ul>
(Ennahal et al., 2020)	Marine sediment from Dunkirk Grand Marine Harbor (France)	• Use of sediment as lightweight aggregates as a partial replacement for natural sand in mortar formulations
(Mymrin et al., 2017)	Sediment dredged from Brazilian seaports (seaport of Paranaguá in Paraná state)	• Use of dredged sediments of the Atlantic Ocean (up to 60 wt %) to develop a construction material such as bricks and blocks.
(Mezencevova et al., 2012)	Sediments dredged from the Savannah Harbor (USA)	<ul> <li>The sediment was used as a raw material for the production of fired bricks</li> <li>The compressive strength of bricks made from 100% of dredged sediment and fired at 1,000°C is in compliance with ASTM brick criteria for the lowest grade (NW) building bricks</li> <li>The compressive strength is improved when natural clay is mixed with sediment and met the requirements for severe weathering (SW) building bricks</li> </ul>
(Komnitsas, 2016)	Marine sediment from the ports of Souda in China, Crete and patras in NW Peloponnese, Greece	<ul> <li>Marine sediments were valorized with construction and demolition waste through alkali activation</li> <li>Sediment from the port of Patras can be alkali-activated for the production of building materials while the one from Souda port acquire a lower strength (5 MPa)</li> </ul>
(Wei et al., 2008)	Harbor sediment from Taichung Harbor in Taiwan	• Preparation of lightweight aggregate from sediment, hazardous steel fly ash, and oyster shell

## **4.2 Treatment techniques applied for sediments**

The implementation of policies for the protection of the environment is strongly related to the development of efficient treatment processes. These methods aim to improve specific properties such as physicochemical characteristics like water demand, the ability to react in the presence of lime (pozzolanic character), the organic matter or pollutants, etc. These processing techniques differ in their implementation means, efficiency, interests, targets, limits, and cost.

251 Several treatment techniques can be applied to sediments:

- a) Physical treatments (flotation, vacuum extraction,...) use the physical properties such as gravity or the interparticle attraction The mechanical treatments (filter press, vacuum filter,...).
  Physicochemical treatments (phosphating, acidification, dechlorination leaching,...) use chemical agents or electrical power to attract and separate the polluted particles. Physical and Physicochemical methods use mechanical processes and chemical products mainly to modify material properties
- b) Chemical treatments: by adding a chemical oxidant to promote an elimination reaction.
- The solidification-stabilization method consists to add a hydraulic binder such as cement, lime, pozzolans, or clay. This process can be applied at low temperature (110°C - 150°C) by using organic binders.
- c) Biological treatments (bioremediation, phytoremediation, Sesatec<sup>™</sup>, bioreactor, Rotamix<sup>™</sup>
  process) which consist to use living organisms such as bacteria, fungi, algae, enzymes, to
  precipitate the biodegradation of organic pollutants such as polycyclic aromatic hydrocarbons
  (PAH) or oils. These methods can be particularly adapted for removing organic pollutants.
- d) Heat treatment (thermal desorption, incineration, vitrification, pyrolysis, oxidation) under high
   temperature 600°C to 1200°C), these processes lead to the total or partial disintegration of the
   organic material (Lafhaj et al., 2012). Recently, an innovative and less energy consumption
   method named flash calcination process was also applied to sediments (Scrivener et al., 2017,

2018). Flash calcination is an innovative and low energy consumption method named flash
applied to treat efficiently and activate materials. The process of flash calcination is applied to
the treatment of dredged sediments to activate some of the sediment phases (including clay
phases). The main properties that determine sediment potentiality of reuse are mineral
composition, pollution content, and clays.

- The determination of adequate calcination conditions (in term of time and temperature of calcination) need some optimization studies as described by (Benzerzour et al., 2017). All the modifications induced by the treatment process target to improve sediment performances and produce an activated material.
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In a recent study conducted (Wang et al., 2017), dredged sediment from Kwun Tong Typhoon Shelter in Hong Kong was used as a cement substitution with two types of cement: OPC and MOC (magnesium oxide cement). The results show that using binary cement followed by 1-day  $CO_2$  curing for recycling contaminated sediment could present a novel and green stabilization/solidification technology. The contaminated sediment from Hong Kong was also used by the same authors (Wang et al., 2015, 2017, 2018a, 2018b) as a fill material, partition blocks and paving blocks by using it as an aggregate.

- 286 aggregate.
- 287 In the following, three treatment methods commonly applied for sediments will be detailed.

## 288 4.2.1 Heat treatment by calcination

The calcination of granular material is a thermal method used for sediment treatment. It consists to 289 calcine the material at high temperature  $(600^{\circ}\text{C} - 1200^{\circ}\text{C})$  to oxidize the organic fractions completely, 290 to eliminate some mineral pollutants and organic contaminants, to evaporate the entire water present in 291 the material and to decalcified the calcite at 675° C (Ramaroson, 2008). According to (Benkaddour et 292 al., 2009) and (Anger et al., 2014), sieving and crushing of sediment could be beneficial because it 293 allows the increase of the fineness of the product (increasing the specific surface) and therefore its 294 reactivity increases as well (Faure et al., 2017). (Limeira et al., 2010) suggest that the heat treatment 295 could initiate the potential pozzolanic activity of sediment. Their results are also consistent with those 296 of (Gutteridge & Dalziel, 1990) who have already established the enhancement of cement hydration 297 reactions in the presence of reactive or inert powders. It also seems appropriate to conduct 298 characterization studies in the pre-processing and post-processing phase, to allow assessing the impact 299 300 of sediment presence into cement matrix. The methodology used is described in Fig. 4. This treatment process leads to the sintering of the sediment (Bernache-assollant & Bonnet, 2005a, 2005b). It is 301 important to note that due to the oxidation, emission of combustion gas and volatile products is 302 induced and must be subjected to chemical analysis (Achard et al., 2013; Ramaroson, 2008). 303

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#### Figure 4: a) Methodology of calcination and b) calcination lab furnace.

308 The temperature T (°C), calcination time (hours) and the fineness after grinding, are the calcination 309 parameters that must be optimized according to the physicochemical characteristics of the material 310 Table 3 describes the treatment methods applied in some studies with different origin of sediments.

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Table 3: Calcination treatment method applied for sediment in previous works in literature

 314
 Table 3: Calcination treatment method applied for sediment in previous works in literature

 Authors
 Sediment origin
 Sediment treatment

(Safhi et al. 2019a, 2019b)	Dredged marine sediment from the Dunkirk-Harbor	Sieving raw sediments through 120 µm and calcination at 850°C for 1 hour
(Benzerzour et al., 2017)	Dredged marine sediment from the Dunkirk-Harbor	Physical treatment (grinding and separation) then calcination at 650, 750 and 850°C for 1h and 3 hours
(Amar et al., 2017, 2018)	Dredged marine sediment from the Dunkirk-Harbor	Sieving raw sediments through 120 µm and calcination at 850°C for 1 hour
(Dang et al., 2013)	Sediment from the northern coast of Brittany	Washing process to reduce the salinity, decantation, drying at 40°C, sieving at 200 $\mu$ m then calcination at 650 or 850 °C for 5 hours
(Laoufi et al., 2016)	Mud dredged from the Algerian dam of Fergoug	Calcination at different temperatures, i.e. 750, 850 and 950°C, for 3 hours
(Dia et al., 2014, 2019)	Dredged sediment from Dunkirk Harbor (north of France)	Phosphatation with $H_3PO_4$ then thermal treatment calcination at 650°C for 2 hours
(Agostini et al., 2007)	Marine sediments dredged in the harbor of Dunkirk	Novosol <sup>™</sup> treatment then calcination at a temperature of 650°C
(Slimanou et al., 2019)	Dredging sediment waste resulting from the dredging works of Bejaia port	Calcination at 750°C for 4 hours
(Ramaroson, 2009, 2012)	Sediments of Dampremy and sediments of Vermont	Novosol <sup>™</sup> treatment The heating rate of 10 °C/min was imposed from room temperature to the designated isotherm temperatures for the experiments (from 100 to 1,000 °C) for 3 hours
(Lafhaj et al., 2007, 2008)	Dredged sediment in the channels of Lille and sediment from the river La Marque in the North of France	The calcination phase consists of calcining the phosphated sediments at 650° C in a rotary kiln
(Ez-zaki et al., 2016a, 2016b)	Marine sediments dredged from the trap Lyvet on the Rance river and the Bay of Mont Saint Michel in the north of Brittany, France	Washing, Grinding-sieving at 200 µm and calcination at 650°C/850°C for 5 hours
(Du & Pang, 2018)	Marine clay	Calcination at 400, 600, 800 and 1,000 °C for 1 hour
(Rozière et al., 2015)	Raw sediments were collected from Dampremy–Charleroi region (Belgium)	Raw sediments were subjected to a Preliminary treatment using the Novosol process which consists of two major phases: phosphatation and calcination
(Wei et al., 2014)	Taichung harbor sediment	Mixes of sediments and slag pellets (about 2.5 cm $\times$ 1.75 cm) are realized and fired at 950–1,100 °C for 18 min after a de-volatile pre-treatment at 500 °C for 2 min.
(Tribout et al., 2011)	Polluted fluvial sediment from Belgium canal at Dampremy	Novosol process: treatment by mixing raw sediments with phosphoric acid H3PO4 (3–3.5%) then calcination of the phosphated sediments at 700°C in a rotary kiln.
(Ferone et al., 2013)	Two sediments from reservoir located in Southern Italy (Occhito, Carlantino, Foggia and Sabetta, Morigerati, Salerno)	Thermal treatment at 650 °C and 750 °C at 1 and 2h
(Ferone et al., 2015)	Two sediments from reservoir located in Southern Italy (Occhito, Carlantino, Foggia and Sabetta, Morigerati, Salerno)	Thermal treatment at 400 °C and 750 °C at 1 and 2h using a Nabertherm furnace with a heating rate of 10 °C/min
(Kribi et al., 2012)	Sediments from the Farciennes site in Belgium	Treatment with phosphoric acid $(3, 5, and 7\%)$ then calcination for 3 hours at 300, 500, 700, 900 and 1,100 °C with a heating rate of 10°C min <sup>-1</sup>

#### 315 4.2.2 Flash calcination method

Flash calcining is a heat treatment technique for the rapid exposure of finely crushed materials in the 316 presence of air, under high temperatures, using a technique which was initiated and developed by 317 Salvador in 1992 (Salvador, 2000). The flash calcination technique was originally applied to 318 chemically activate some clays such as Kaolinite and to confer pozzolanic properties. When applying 319 this technique to Kaolin clays, a dehydroxylation process following the dehydration was noted (San 320 Nicolas et al., 2013; Snellings et al., 2017; Teklay et al., 2015). This fits with the elimination of a 321 hydroxyl bond (-OH) that occurs between 450°C and 750°C (Sperinck et al., 2011). Direct calcination 322 can help to achieve such results, however, energy costs and  $CO_2$  footprint, in particular, are often 323 exorbitant. Also, due to the instantaneousness character of the flash process, the flash calcination 324 causes the emergence of glassy phases, a destructuration of the matter, an amorphous state, and 325 therefore a potential reactivity of the product (Bich et al., 2009). Two parameters are however 326 predominant: the exposure time (in the order of a few tenths  $(1/10)^{\text{th}}$  of seconds), and the temperature, 327 which is around 1200°C inside the calciner unit (Nicolas et al., 2013). The energy consumption can be 328 estimated to 2 GJ/t according to (Nicolas et al., 2012, 2013). 329

This process is applied to the treatment of dredged sediments to activate some of the sediment phases (including clay phases). The calciner unit, flash tower, and pilot principle are shown in Fig 5.

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- 334 335

Figure 5: a) Flash calciner chamber; b) Semi-mobile flash tower; c) Pilot installation (San Nicolas et al., 2013)

This technique was applied by (Van Bunderen et al., 2019) to calcine sediment from the port of 336 Antwerp in Belgium to substitute the Portland cement. The Flash calcination took place in a gas 337 suspension calciner at 865 °C. After the material cooling, no crushing or grinding are applied to the 338 calcined material. (Snellings et al., 2016) used also the flash calcination in the same sediment studied 339 by (Van Bunderen et al., 2019). Three calcination temperatures were tested: 820, 865, and 905 °C to 340 investigate the influence of the latter on the properties and the pozzolanic reactivity of sediment. This 341 study reveals that the increase in the calcination temperature decreases the BET specific surface area. 342 (Amar et al., 2017) compared the properties and the pozzolanic activity of cementitious materials 343 substituted by calcined sediment either by direct calcination or Flash calcination. The sediment used is 344 coming from Dunkirk harbor in France. It has been concluded from this study that the flash treated 345 sediment is more pozzolanic material than the direct calcined one. 346

#### 347 **4.3 Chemical Treatment**

348 Chemical treatment consists of eliminating organic fraction, to remove some ions, and to trap some heavy metals through chemical reactions. Various chemical products can be used for this treatment, 349 such as sodium hypochlorite (NaOCl) (Anderson, 1963) and the proxidisulfate disodium ( $Na_4P_2O_7$ ) 350 (Eusterhues et al., 2003), but the most widely used is hydrogen peroxide  $(H_2O_2)$  (Mikutta et al., 2005) 351 after its introduction in the soil analysis by (Robinson, 1922). Moreover,  $H_2O_2$  is the oxidizer 352 recommended by the Belgian standard NBN 589-207 §3. The use of these products targets the 353 oxidation of the organic fraction but may have additional consequences especially by changing the 354 mineral structure of sediments (Mikutta et al., 2005). Some previous studies have already suggested 355 some protocols for efficient removal of the organic fraction (Leifeld & Kögel-Knabner, 2001; Schmidt 356 357 & Noack, 2000; Schultz et al., 1999; Sheldrick & Wang, 1993; Theng et al., 1999) as detailed in Table 358 4.

Table 4: Chemical	treatment	methods	applied	for sec	liments.

Protocol	Effects	Reference
Use 500ml of a concentrated solution between 15 and 30% of $H_2O_2$ . <u>Temperature</u> : under room temperature and then at 70°C. <u>Duration</u> : 24-72 hours	83-94% removal of organic material (OM). Impact on the granular skeleton	(Leifeld & Kögel- Knabner, 2001)
Use 50 ml of a concentrated solution of $H_2O_2(6\%)$ repeated 5 times. <u>Temperature</u> : 80 ° C.	66-97% removal of OM. Impact on the specific surface	(Theng et al., 1999)
Use 250 ml of a concentrated solution at 6% NaOCl repeated 6 times 6 hours. <u>Temperature</u> : 25 ° C; pH=8	49-81% removal of OM. Isolation of the stable organic matter	(Mikutta, et al., 2005)
Use 20g of Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub> with 22g of NaHCO <sub>3</sub> buffer solution <u>Temperature</u> : 80°C; <u>Duration</u> : 48 hours	16-99% removal of OM. Isolation of stable organic matter	(Eusterhues et al., 2003)
The sediments were leached and dewatered and then dried at 40 °C. After drying, a chemical treatment was realized by adding 3% of phosphoric acid by mass of sediment. At the end, the sediments were matured and dried for 3 days.	The heavy metals were trapped after treatment.	(Aoual-Benslafa et al., 2015)
Sediments are milled for 3 minutes in laboratory vibrating mill together with granulated NaOH in a ratio of 2/1 (sediment/NaOH).	Mechanical activation	(Junakova & Junak, 2017a)
Sediment pretreated by thermal degradation (at 300 or 400 °C for 1 h in a muffle furnace) and alkali extraction (1-h solubilization by NaOH at pH 12 and the solid-to-solution ratio of 100 g L–1), then sediment bocks were cured in the presence of CO2.	Strength enhancement Carbon footprint reduction	(Wang et al., 2015)

370

371 These methods have the advantage of being less expensive (10 $\notin$ /L H<sub>2</sub>O<sub>2</sub>) than the heat treatment, but

they do not seem to involve pozzolanic activity of sediments. In addition, the organic fraction is not

completely removed using this method (10% to 95%) (Eusterhues et al., 2003; Mikutta et al., 2005;

374 Theng et al., 1999).

## **5.** Chemical activity quantification

The quantification of the chemical activity is important in order to test the reactivity of the mineral addition such sediments in our case. Therefore, the following will describe the different methods used in the literature to calculate the activity index and coefficient of the addition. Some of the methods described in the following are based on index calculation referring to a control mortar or alkaline concentration. For the others, they are based on chemical tests aiming to quantify the lime consumption capacity of sediments into prepared formulations.

382

#### 383 5.1 Bolomey's method

384 The calculation of the activity index  $i = R_p / R_0$  estimates the effectiveness of an addition as a cement

substitute in mortar or concrete. This value is the result of the ratio between the compressive strength
 of standard reference mortar (NF EN 196-1) and the compressive strength of the mortar with 25 w.t. %
 of cement substitution by the pozzolanic supplementary cementitious material (De Larrard, 2000).

In 1935, (Bolomey, 1935) established that the strength of mortar can be estimated at 28 days as (Eq. 38)
3):

The following equations are derived from the studies of (Amar et al., 2017) where a detailed description is given.

$$\mathbf{R}_{o} = \left(\frac{\mathbf{K}_{Bolomey}}{2}\right) \left[ \left(\frac{\Delta}{2350}\right)^{2} * \frac{\mathbf{C}_{o}}{\mathbf{E}} \right]^{3/2}$$
(3)

392 The most commonly used formula (Eq.4) is a simplification of Eq.3 used in construction sites.

$$R_0 = K_{Bolomey} \left[ \frac{C_0}{E} - 0.5 \right]$$
(4)

21

- 393 In this study, we will exclusively use the above formula (Eq.4).
- 394 For substituted mortar, the expression is given in Eq.5:

$$\mathbf{R}_{o} = \left(\frac{\mathbf{K}_{Bolomey}}{2}\right) \left[ \left(\frac{\Delta}{2350}\right)^{2} * \left(\frac{C + \chi_{A}A}{E}\right) \right]^{2}$$
(5)

395

396 The activity index i is determined by (Eq.6):

$$i = R_P / R_0 = \frac{\left(\frac{K_{Bolomey}}{2}\right) \left[ \left(\frac{\Delta}{2350}\right)^2 * \left(\frac{C + \chi_A A}{E}\right) \right]^{\frac{3}{2}}}{\left(\frac{K_{Bolomey}}{2}\right) \left[ \left(\frac{\Delta}{2350}\right)^2 * \left(\frac{C_0}{E}\right) \right]^{\frac{3}{2}}} = \left(\frac{\frac{C + \chi_A A}{E}}{\frac{C_0}{E}}\right)^{\frac{3}{2}}$$
(6)

And then  $C = (1-p)C_0$  and  $A = pC_0$  have the result below (Eq.7):

$$i = R_P / R_0 = \left(1 - p \left(1 - \chi_A\right)\right)^{\frac{3}{2}}$$
(7)

398 The mechanical activity coefficient  $\chi_A$  deduced from Eq.8 given below

$$\chi_A = 1 + \frac{i^{2/3} - 1}{p} \tag{8}$$

\_

#### 399 **5.2 Frattini's method (NF EN 196-5)**

This method of quantification is inspired by the standard NF EN 196-5. This method is used for 400 determining the amount of lime consumed by a pozzolan after 8 and 14 days (Fig. 6). Then it evaluates 401 the pozzolanic reactions of cements and pozzolanic additions properties. The method consists of an 402 evaluation of hydroxide ion concentration (OH<sup>-</sup>), and calcium ions Ca<sup>2+</sup> expressed as quicklime or 403 calcium oxide (CaO) in a mixture containing 80% cement and 20% pozzolan. The mixture is kept in a 404 thermostatically controlled room at  $40^{\circ}$ C. This test has been applied in previous studies where the 405 activity of calcined clay pozzolan was established (Gastaldini et al., 2015; Tironi et al., 2013). The 406 following figure is derived from the studies of (Amar et al., 2017) where a detailed description is 407 given. 408

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Figure 6: Frattini's test principle

#### 413 **5.3 Kinetic Method (2000)**

This method is proposed by (De Larrard, 2000) for pozzolanic additions. It consists to make two mortar formulations. The first one is a standardized mortar according to ISO (NF 196-1 standard) and the second one is a mortar in which 25 w.t. % of cement is substituted by the addition. It is appropriate to ensure fairness in terms of consistency, by adding a certain amount of superplasticizer if needed. The proposed formula (Eq. 9) corresponds to an activity coefficient taking into account the activity index and the curing time:

$$\chi_{A} = -3 * \ln \left[ 1.91 - \frac{2.2}{\left( i(t) \left[ 0.0522 + d(t) \right] - d(t) \right)^{-0.351} - 1} \right]$$
(9)

420

421

422 Where i(t) = activity index: the ratio between the compressive strength of the reference mortar and the 423 substituted one; d(t) is the age of concrete (days).

#### 424 **5.4 Physicochemical analysis method**

It is possible to find a linear type of semi-empirical relationship between the activity coefficient and the alkaline content. In the case of silica fume, it was shown that their activity does not depend on the cement type, but so far on the alkaline content (De Larrard & Le Roy, 1992). The Na<sub>2</sub>O<sub>equi</sub> content can be obtained by quantitative XRF analysis.

Na<sub>2</sub>O<sub>equiv</sub> is the equivalent sodium oxide content (Eq.10). The Kp(28) (Eq.11) value is estimated on
mortars substituted by addition (10 w.t. % in the case of the study done by (De Larrard & Le Roy,
1992).

432

$$Na_2 O_{equiv} = NaO + 0.658K_2O \tag{10}$$

433

$$K_P(28) = 2.8 - 0.7 Na_2 O_{equiv}$$
 (11)

434

#### 5.5 Method of the conductivity measuring 435 There seems to be a good correlation between the decrease in conductivity and the formation of 436 hydrates in a pozzolanic reaction. This test is based on (Luxán et al., 1989) work, which established a 437 correlation between the pozzolanic properties of one hundred different SCMs and their electrical 438 conductivity (Delair, 2008). From the first moments of the reaction between a pozzolan and lime, the 439 authors note a significant decrease in conductivity due to the early binding of calcium by amorphous 440 silica. The installation diagram is represented in Fig. 7. Testing cells containing pozzolan and lime are 441 monitored using a conductimeter and an acquisition system. This method is previously presented by 442 (Amar et al., 2017) where a detailed description is given. 443 444 445 446 Figure 7: Principe for conductivity measurements 447 448 The method of measuring the conductivity consists of: 449 1. Add into a beaker 100 ml of saturated $Ca(OH)_2$ solution already thermostatically controlled 450 in a water bath at $40 \pm 1$ ° C; 451 2. Measure the initial conductivity denoted $C_1$ 452 3. Add 5 g of the powder sample (the material must be dry and fine) 453 4. Keep the solution in agitation and measure regularly the conductivity 454 5. Note the value of the conductivity after 120 seconds. This value is $C_2$ 455 6. Continue until 300 to 400 minutes and typically until stabilization. 456 The value $\Delta_C = C_1 - C_2$ gives the classification of the product according to its level of pozzolanic 457 458 properties. • (A) $\Delta_C \leq 400 \text{ mS/cm}$ : $\Rightarrow$ not a pozzolanic product 459 • (B) 400 mS/cm $\leq \Delta_C \leq 1200$ mS/cm : $\Rightarrow$ variable pozzolanic properties 460 461 • (C) $\Delta_C \ge 1200 mS / cm$ : $\Longrightarrow$ High pozzolanic properties 5.6 Accelerated Chapelle method 462 This test reproduces the pozzolanic reaction between a reactive powder and lime as applied for 463 metakaolin pozzolanic activity test (NF EN 18 543 standard). The method consists to react one gram 464 (1gr) of powder with one gram (1 gr) of quicklime (CaO) or portlandite (Ca(OH)<sub>2</sub>), in 200 ml of 465 boiling water for 16 hours. Therefore the fixed lime upon the reaction with the powder is estimated 466 467 (Mechling, 2000). The experimental system is shown in Fig. 8. The following figure is derived from the studies of (Amar et al., 2017) where a description is given. 468 469 470 471 Figure 8: Experimental installation of Chapelle test 472 473 After the test, the solution is cooled to a constant temperature at $60^{\circ}C \pm 1^{\circ}C$ and then the excess of lime 474 is solubilized with 60g of sucrose diluted in 800 ml of water. By chemical dosage, the mass of lime 475 consumed can be deduced knowing that the lime initial mass is 1g and the total volume of the solution 476

is 1 liter. This measure is then done by complexometry (eg EDTA + use of a metallochrome indicator)
(Mechling, 2000), or any other suitable method such as Inductively Coupled Plasma analysis (ICP ).

- The calcined sediments can be considered pozzolanic regarding the Frattini test results (Amar et al., 2017; Faure et al., 2017; Snellings & Scrivener, 2016)
- 481 The Chapelle test inferred that calcined sediment materials can be considered chemically reactive with an intermediate pozzolanic level (< 66%) (Amar et al., 2017; Faure et al., 2017).</li>
- From, the electrical conductivity measurements monitored on lime/pozzolan suspension, the paper also concludes that flash calcined sediment is the most pozzolanic material compared to metakaolin and limestone (Amar et al., 2017).
- 486 The mechanical strength investigations indicate an activity coefficient for the flash calcined 487 sediment of  $\chi$ =0.88 that is higher than the limestone filler value (Amar et al., 2017; Faure et 488 al., 2017).
- 489

# **6. Durability and environmental impact**

Concrete and mortar are materials subject to different types of attacks. This requires to ensure the 491 satisfaction of conditions both in sustainability and in its immediate environmental impact. 492 Accordingly, multiple durability tests exist and are detailed in the following of this paper. The concept 493 of equivalent performance introduced by EN 206-1/CN opens up the possibility of qualifying a 494 concrete with a particular composition and behaving at least as well as a reference concrete whose 495 level of risk has been deemed acceptable, in a given environment. This concept is used in multiple 496 cases of SCMs performance validation. Table 5 describes some durability studies made on sediment 497 based materials. 498

499 500

Table 5: Durability studies on sediment based material from literature

Durability study	Sediment origin	Authors		
Freeze and thaw test	Marine sediments dredged in the harbor of	(Achour et al., 2019)		
	Dunkirk	(Amar et al., 2018)		
External sulfate attack	Marine sediments dredged in the harbor of	(Achour et al., 2019)		
	Dunkirk	(Amar et al., 2018)		
Alkali-aggregate reaction	Marine sediments dredged in the harbor of	(Achour et al., 2019)		
	Dunkirk	(Amar et al., 2018)		
	Reservoir sediment was obtained from the	(Chiou & Chen, 2013)		
	Shih-man Reservoir in Taoyuan County			
Porosity investigation	Marine sediments dredged in the harbor of	(Amar et al., 2018)		
	Dunkirk			
Chloride permeability	Marine sediments dredged in the harbor of	(Amar et al., 2018)		
	Dunkirk	(Wang, 2009)		
	Dredged silt from reservoirs in southern	(Hwang & Hung, 2005)		
	Taiwan			
	Fine sediment excavated from reservoirs			
Resistance to the chemical	sediments collected from different docks of the	(Aoual-Benslafa et al., 2015)		
aggressions	port of Oran	(Benkaddour et al., 2009)		
3% of chloridric acid (H2SO4)	Sediment from Fergoug dam (Algeria)	(Chiou & Chen, 2013)		
3% of chloridric acid (HCL)	Reservoir sediment was obtained from the			
5% of chloridric acid (HCL)	Shih-man Reservoir in Taoyuan County			
5% of chloridric acid (H2SO4)				
Volume of permeable pores	Dredged marine sand extracted from the Port of	(Limeira et al., 2010)		

Sant Carles de la Ràpita (Tarragona, Spain)	
Dredged marine sand extracted from the Port of	(Limeira et al., 2011)
Barcelona	
Dredged marine sand extracted from the Port of	(Limeira et al., 2011)
Barcelona	
Dredged silt from reservoirs in southern	(Wang, 2009)
Taiwan	(Hwang & Hung, 2005)
Fine sediment excavated from reservoirs	
	Sant Carles de la Ràpita (Tarragona, Spain)         Dredged marine sand extracted from the Port of Barcelona         Dredged marine sand extracted from the Port of Barcelona         Dredged silt from reservoirs in southern Taiwan         Fine sediment excavated from reservoirs

501 502

## 503 6.1 Alkali reaction

The alkali reaction is a deleterious reaction that affects the durability of structures. It represents the 504 reaction between hydroxyl ions provided by the hydrated cement paste and the reactive siliceous 505 phases coming from some reactive aggregates. As a result, an expansive calcium silicate gel is formed 506 in the cement matrix (Cyr & Pouhet, 2015; Kleib et al., 2018; Lombardi et al., 1997). According to the 507 508 study completed by (Fatt & Beng, 2007), this reaction is the combination of three factors: the reactive silica in aggregates; alkali interstitial solution ( $K^+$  and  $Na^+$ ) of critical thresholds and external water or 509 moisture. The ASTM C 1260 standard recommends a critical limit of 0.6% Na<sub>2</sub>O equivalent (Prince & 510 Perami, 1993). The use of sediment as SCMs can induce alkali-silica reaction due to the presence of 511 amorphous silica. However, in aggregates or mineral addition SCMs such as dredged sediments, two 512 phenomena may take place due to its sand-lime nature: alkali-silica reaction or the alkali carbonates 513 reaction (Murlidhar et al., 2016a). The alkali test is used to estimate the potential expansion of 514 aggregates with alkali. The principle is to estimate the evolution of the geometric dimension of mortar 515 516 4x4x16 cm samples and also mass variations (XP P18-594, ASTM C 1260, standard, (Amar et al., 517 2018; Murlidhar et al., 2016b).

Mortars with different cement/aggregate C / G ratios (0.5 - 1.25 - 2.5) must be prepared for the test. 518 The preparation and mixing of mortars can be performed following NF EN 196-1, taking care to add 519 to the mixing solution 4.0% Na<sub>2</sub>O<sub>equivalent</sub> relative to the cement mass in order to obtain the required 520 alkalinity. The specimens are initially placed in an environment with 90% RH ( $20\pm1^{\circ}$  C) then removed 521 from the water after 48 h  $\pm$  30 min to determine the initial length L<sub>0i</sub>. The samples are exposed for 5 522 hours in an autoclaved cell at a temperature of 127 ° C and with a relative pressure of 0.15 MPa. The 523 final length  $L_{1i}$  is measured after (18 ± 1) h. The material tested is qualified as non-reactive (NR) when 524 the expansion is less than 0.15%, potentially reactive (PR), or PRP. The deformations are then 525 526 expressed as presented in Eq.12:

$$\varepsilon_i = \frac{L_{1i} - L_{0i}}{L_{0i}} \tag{12}$$

527

It should be noted that there are also two different methods: a test on micro mortar and a test based on chemical kinetics (not applicable if  $Al_2O_3 > 5\%$ ). (Achour et al., 2019) has studied the alkali-aggregate reaction for two concretes containing 12.5 and 20% of marine sediment as aggregates in the accropode blocks. The test of the alkali-aggregate reaction was performed according to the CSA A23.2-14A standard. The results showed that increasing the amount of sediment increases the expansion due to the alkali-aggregate reaction. (Amar et al., 2018) showed in his study, that the use of calcined marine sediment as SCMs does not affect the alkali-aggregate reaction.

#### 535 **6.2** Accelerated carbonation

Exposed to specific humidity and regarding temperature conditions, concrete can undergo the 536 carbonation process. According to (Roziére, 2007), carbonation is the phenomenon by which carbon 537 dioxide  $(CO_2)$  penetrates the concrete and dissolves in the cementitious pore solution and then reacts 538 both with portlandite Ca(OH)<sub>2</sub> which approximately 20% to 30% of the hardened paste (Houst et al., 539 1989) and calcium silicate hydrates (CSH) (Chaussadent, 1999; Chaussadent et al., 2000). Some 540 characteristics of CO<sub>2</sub> such as its molecular diameter of 0.426 nm and a diffusion coefficient that 541 equals  $D = 1.60.10^{-5}$  m<sup>2</sup>/s at 1 bar, 20°C, allow it to penetrate easily and stay into the cementitious 542 matrix. 543

544

545 The chemical reactions of portlandite and CSH by  $CO_2$  are described in Eq. 13 and Eq. 14 below:

$$Ca(OH)_2 + CO_2 + H_2O \Longrightarrow CaCO_3 + 2H_2O \tag{13}$$

$$C - S - H + CO_2 \Longrightarrow CaCO_3 + \langle SiO_2, H_2O \rangle \tag{14}$$

546 Carbonation induces in the concretes several effects:

Negative effects are due to the depassivation of the matrix, the pH decreases from 13 to 9. The neutralizing of basic compounds and a decrease in volumes induce shrinkage (Houst et al., 1989). The above effects can cause the rusting of 0.05 mm/year in the case of reinforced pieces (Arup, 1983). It can also induce the transformation of CSH from an amorphous gel structure to the porous structure (RILEM, 2016.).

Beneficial effects have been reported and correspond to the decrease of the total pore volume
of the order of 12% according to (Chaussadent, 1999) and cause an improvement in
mechanical strength. For (Houst et al., 1989), carbonation of non-reinforced concrete involves
a global improvement of the mechanical properties: improvement of compressive strength up
to 30%, increasing the elastic modulus, reduction in the gas and liquid permeability.

The assessment of carbonation effects is made through the determination of carbonation depth after an accelerated carbonation test. However, when concrete is exposed to rain, the effect of carbonation can be reduced (Basheer et al., 2001). Several prediction models exist, such Van Balen and Van Gemert, Papadakis. All these models agree on the fact that the carbonation depth can be expressed by a mathematical relation in the form of Eq. 15 (Chaussadent, 1999; Roziére, 2007):

562

$$x = K\sqrt{t} \tag{15}$$

563

564 Where K is the carbonation coefficient and t = time

The test refers to the standard XP P18-458 (Hornain, 2007) and the principle is shown in Fig. 9. In France, the test most common test used consists to circulate within a carbonation box, a gas mixture containing 50%  $CO_2$  and 50% air (Castel et al., 2001; Chaussadent et al., 2000).

- 568
- 569 570

Figure 9: Accelerated carbonation test

571 At the end, it is necessary to use a color indicator (color shift at pH of 9) like phenolphthalein to 572 distinguish the carbonation front.

#### 574 **6.3 Porosity accessible to water**

This test evaluates the percentage of connected voids from the surface to the mass of concrete. From a 575 ratio W/C = 0.6, about 30% of capillary pores have a dimensions  $\phi = 10^{-3}$  to  $10^{-4}$  mm. Therefore a 576 porous network exists that facilitates the transport of fluids such as water. For ordinary concrete, 577 porosity represents 11 to 16% of the total volume (Mazars, 1984). The presence of this porosity is 578 beneficial for freeze-thaw phenomena especially due to the presence of interstitial water. The test of 579 580 porosity accessible to water refers to the NF EN 14459 and consists to prepare cylindrical concrete samples 11x22 cm. These specimens are placed in a vacuum desiccator connected to water supply 581 valves and silica gel for the humidity control. The calculation of the porosity accessible to water is 582 583 given by Eq. 16:

$$P_{W} = \frac{Mair - Mdry}{Mair - Mw}$$
(16)

584

585 Where:

586 • Pw = porosity

 $M_{air}$  is the sample weight in air

- ${}_{588}$   $M_w$  is the sample weight under hydrostatic weighing
- $M_{dry}$  is the dry mass obtained after drying at 105°C to constant weight.

590 In the case of mortar, 4x4x16cm bars can be prepared according to NF EN 196-1 as performed by 591 (Amar et al., 2018; Achour et al., 2019; Limeira et al., 2011).

## 592 **6.4 Migration of chloride ions:**

Marine sediments contain chloride salts (calcium chloride, potassium chloride, sodium chloride) that
could involve negative effects when used as a construction material. The presence of salt induces
problems on mechanical performances and can accelerate the corrosion of steel in reinforced concrete.
In fact, for each cement application, a chloride limit is defined according to the European standard EN
206-1 (Table 6).

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Table 6: Chloride content limits depending on the application (Dang et al., 2013).

Use of concrete	Level chloride	Maximum content of Cl— relative to the mass of cement and additions took into account
No reinforcement, no embedded metal parts	Cl 1.0	1.0%
Containing steel reinforcement or embedded metal and made with cement type CEM III	Cl 0.65	0.65%
Containing steel reinforcement or embedded metal parts	Cl 0.4	0.4%
Containing pre-stressed steel	Cl 0.2	0.2%

600

The exposure of cement matrix to chloride ions has multiple origins and different consequences. These
 chlorides may have two origins according to (Roziére, 2007): seawater and melting salts. Magnesium

- 603 chloride  $MgCl_2$  and calcium chloride  $CaCl_2$  are the two most aggressive salts present in seawater with 604 a pH of 7.8 to 8.3.
- The experimental installation is shown in Fig. 10. A sample of sawed mortar or concrete is placed between two texting cells containing NaCl and NaOH solutions. A voltage difference allows chloride ions to migrate between cells and then the acquisition system measures the current intensity and the charge passed (coulombs).
- 609
- 610 611

#### Figure 10: Chloride migration test (Amar, 2017)

The penetration and spread of chloride ions are induced by the diffusion resulting from a concentration 612 gradient and the capillary absorption that is the fact of the pore connection status and humidity 613 conditions (Basheer et al., 2001). The first phenomenon (diffusion) leads to the measurement of a 614 diffusion coefficient D by migration into an electric field. Existing protocols agree on measuring a 615 coefficient Deff  $(m^2/s)$  corresponding to chloride ions which is the flow through the sample in the 616 617 permanent regime or Dapp (m<sup>2</sup>/s). The second phenomenon corresponds to the absorption 618 measurements as defined in the standard EN 13369. Because the regular tests can last several months, the XP18461 standard provides the determination of the diffusion coefficient by accelerated test in 619 stationary regime by determining Deff (m<sup>2</sup>/s). The latter corresponds to ions chloride depth penetration 620 (Hornain, 2007). The mechanisms involved can be quite complex but are often related to the reactions 621 of C3A and C4AF with chloride ions to form an expansive salt named Friedel salt or calcium 622 623 carboaluminate (Basheer et al., 2001).

624 Among the effects of chloride ions diffusion, it should be considered the inhibition effects that induce 625 rust. This capacity is increased proportionally to chloride concentration but depends on the concrete 626 properties and the exposure (Bjegovic et al., 1987). The study realized by (Amar et al., 2018) shows that the chloride permeability of mortars with 10% of calcined dredged sediments in substitution of 627 Portland cement is almost similar to the mortar without substitution. Whereas with a higher cement 628 substitution, mortars performances decrease. (Snellings et al., 2017; Van Bunderen et al., 2019) also 629 630 proved that adding 30% of calcined sediment in concrete increased the resistance to chloride ingress 631 compared to Portland cement without sediments, consistent with a refined pore structure as a result of 632 the pozzolanic reactions; however, the scaling resistance was reduced when evaluated in presence of 633 de-icing salts.

It has been shown by (Siham et al., 2008b) that using marine sediment as SCMs without washing decreases the mechanical performances due to the presence of salt. In the study of (Dang et al., 2013), two steps washing process was applied to sediment to reduce the chloride content. The procedure consists to first dilute the sediment with water (ratio sediment to water = 3:1) and second to decanting the mixture. The results show that after this washing process the chloride amount in the raw sediment 639 decreases 5 times comparing to the non-washed one.

#### 640 6.5 Gas permeability

641 Concrete is a porous material, therefore, it can be subjected to liquid or gaseous fluids transfer. The 642 study of this property is appropriate because it gives an appreciation of the porous network structure. It also gives an estimation of the effect of certain gases such as carbon dioxide that can induce a 643 depassivation of the matrix, leading to the steel bars corrosion (Basheer et al., 2001). According to 644 (Hermann, 2000), the study of gas permeability is relevant for assessing the carbonation vulnerability 645 and the quality control of concrete, because its gas permeability is higher than water's. The estimation 646 of porosity and is generally between 10<sup>-14</sup> to 10<sup>-19</sup> m<sup>2</sup> for ordinary hardened concrete and mortar 647 648 (Hermann, 2000).

The test described by experimental standard XP P18-463 and the AFPC-AFREM methods consists of the exposure of a prepared sample and conserved under constant pressure using low viscous gas ( $O_2$  or  $N_2$ ). The measuring device can be similar to Cembureau permeameter (Bakhshi et al., 2006; Hornain, 2007). The permeability can be assessed at 7 days and 28 days with the following formula derived from Hagen-Poiseuille permeability law (Eq. 17):

654

$$k_{x} = \frac{2.P_{1}.Q.L.\mu}{A(P_{0}^{2} - P_{1}^{2})}$$
(17)

655

656 Where:

657  $k_x$  =permeability  $(m^2)$ ;  $A = cross section <math>(m^2)$ 

 $P_0$  = pressure upstream (*Pa*);  $P_1$  = pressure out stream (*Pa*).

658 Q = Gas flow  $(m^3/s)$ ; L=sample length (m);  $\mu$ =dynamic viscosity of the gas  $(N/m^2)$ .

Since water parameter is a major factor in sediment based concretes, the measurement of gas permeability must be taken rigorously. In fact, a mortar with high water saturation may indicate a gas permeability lower than when it is dry (Hermann, 2000). It should be considered finally that the cement type and W/C ratio have an impact on the permeability that increases proportionally with these two factors.

#### 664 6.6 Freeze-Thaw test

The damages caused by freezing and thawing can probably be considered as one of the most aggressive actions, the most harmful and detrimental to the concrete (Aïtcin, 2003; Liu & Hansen, 2016). Concrete is a porous material, consisting of three-phase: voids, aggregates, and water. Below water freezing temperature (about  $-3^{\circ}$ C), water contained in the pores freezes. In this case, it can increase in the volume of about 10% and then create adverse internal hydraulic pressures.

According to (Bjegovic et al., 1987), internal pressures can reach 1 to 4 MPa, which exceeds the
tensile limit of ordinary concrete. By a cyclic process depending on the cooling rate, the number of
cycles, and external temperatures, the damage of the concrete can be reached causing cracking,
peeling, delamination, etc.

674 Referred to the ASTM C666 standard, when a material has a 60% durability factor after 300 cycles of

675 freeze-thaw, it can be considered as resistant to frost. The test under standard NF P18-424 in France
676 indicates the preparation and storage of samples 10x10x40cm according to the NF EN 12390-1

677 standard. The test is a measure of the initial length and the dynamic modulus. The samples are 678 subjected to temperature cycles from  $-18 \pm 2^{\circ}$ C to  $+9 \pm 3^{\circ}$ C in a thermally controlled room and for a 679 specific time period.

According to Faguerlund (cited by (Basheer et al., 2001)), there is a water content threshold beyond which the impact on a cement matrix subjected to freeze-thaw cycles is irrevocable. For (Forster et al.,

1994), the impact of the freeze-thaw on a cementitious matrix depends on the geomorphological origin

683 of the aggregates, the shape and arrangement of pores, the cooling rate, the repetition of freeze-thaw

684 cycles, and the pores saturation. Consider also that the presence of dissolved salts may also lower the 685 freezing point of water. This is particularly the case when melting salts are used, thus accentuating the

685 freezing point of686 phenomenon.

687 Several solutions exist to limit the damage related to freeze and thaw effects particularly in the 688 formulation of sediment mortars: limiting mixing water in connection with petrographic nature and 689 organic matter content, the optimization of the granular skeleton, the limitation of the risk of 690 segregation, etc.

#### 691 **6.7 The sulfate attacks**

692 Sulfate attack can be a real and serious threat to concrete durability subjected to aggressive environments (El-Hachem et al., 2012). The mechanism of concrete deterioration concrete, in this 693 case, depends on several factors: temperature, cation associated nature (Na<sup>+</sup>, Mg<sup>2+</sup>,...), sulfate ion 694 concentration and also the predominant roles of tricalcium aluminate (C<sub>3</sub>A) (around 8% for (Aïtcin, 695 2003)) and the portlandite (Ca  $(OH)_2$ ) (Neville, 2004; Rozière et al., 2009). Two types of sulfate attack 696 exist the internal and external sulfate attack. According to the NF EN206-1 standard, the environments 697 rich in gypsum, sulfate products, or marine environment, are considered chemically aggressive and are 698 classified as XA or XS exposure. The chemical action of SO<sup>4-</sup> sulphate ions leads to the formation of 699 gypsum, ettringite and thaumasite (Collepardi, 1999; El-Hachem et al., 2012). These phases are 700 formed through the following mechanisms (Eq.18 and Eq.19): 701

702

 $Ca(OH)_{2} + Na_{2}SO_{4} + 2H_{2}O \Longrightarrow \langle CaSO_{4}.2H_{2}O \rangle + 2NaOH \quad (18)$ 

703

$$C_{3}A + 3 \langle CaSO_{4}.H_{2}O \rangle + \langle 24 - 26 \rangle H_{2}O \Longrightarrow \langle C_{3}A.3CaSO_{4}.30 - 32H_{2}O \rangle \quad (19)$$

704

For solutions with a poor concentration in calcium hydroxide, gypsum formation can also result from ettringite dissolution, when the pH is  $11.5 \cong 12$  (Santhanam et al., 2001). (Mehta, 1975) shows that gypsum formation can cause flaking and a loss of mass.

The formation of expansive products such as secondary and delayed ettringite often leads to disorders and cracks in the cement matrix (Santhanam et al., 2003), unlike the primary one which is a setting controller. This damage is often observed in the heat-treated structures (>  $60 \circ C$  (LCPC)).

The thaumasite (CaCO<sub>3</sub>.CaSO<sub>4</sub>.CaSiO<sub>3</sub>.15H<sub>2</sub>O) is a reaction produced by the sulfate ions, the CSH, and carbonates. Its formation is enhanced by the low temperature ( $<15^{\circ}$ C), high relative humidity, and the presence of materials rich in calcite (CaCO<sub>3</sub>)(Hermann, 2001). This is also the case for sediments that are often likely sand-silt or clay-limestone or both geological natures. It is also useful to remember the preponderance of parameters such as permeability and portlandite content. The decrease in the amount of portlandite (by pozzolanic reaction for instance) reduces the vulnerability, however, it may induce the degradation of CSH by MgSO<sub>4</sub> solutions.

Multiple external sulfate attack tests exist. For instance, the French standard NF 18-837 and US 718 regulations including the ASTM C1012 and ASTM C452 can be applied. The existing protocols 719 consist to immerse the samples in a 5% solution of Na<sub>2</sub>SO<sub>4</sub>.10H<sub>2</sub>O with immersion/drving cycles 720 (60°C or 105°C). The protocol described in Fig. 11 uses pH regulator to have an accelerated test 721 always representative. Mortar samples 7x7x28cm are prepared and immersed in a sodium sulfate 722 solution with a pH =  $7.0\pm0.2$  controlled by adding a sulfuric acid solution (H<sub>2</sub>SO<sub>4</sub>) at 0.02 mol/L. The 723 conduct of the test includes the measurement of mass loss, dynamic modulus, the amount of acid 724 consumed. 725

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Figure 11 : Sulfate attack test (E. Rozière, R. El Hachem, 2015)

(Safer et al., 2018) demonstrated that adding calcined dredged sediment up to 20% in concrete induce a comparable resistance to  $H_2SO_4$  attack to that of concrete without sediment. Whereas, the study of (Amar et al., 2018) shows that adding 10% of calcined dredged sediment has equal performances in the external sulfate attack test as the mortar of reference without sediment. Whereas (Achour et al., 2019) showed that the samples with 12.5 % of sediment (used as aggregate) have a better resistance against sulfate attack comparing to the ones containing 20%. For the authors, this behavior is related to the highest porosity in the latter.

## 737 6.8 pH constant leaching test

The contact between the concrete surface and fluids leads to several phenomena among which 738 leaching mechanisms. For (Guillon, 2004), the leaching of a cementitious matrix corresponds to the 739 gradual dissolution of the main components and is recurrent in the case of contact with hydraulic 740 structures such as dams, concrete blocks, marine structures, piping systems, tanks, etc. Due to a 741 gradient concentration, the ions diffuse out of the concrete which is at acid pH in the case of 742 lixiviation in water, inducing diverse consequences (Guillon, 2004). The concrete leaching 743 phenomenon is the result of  $Ca^{2+}$  and  $OH^{-}$  ions transfer that lowers the alkalinity facilitating the 744 chemical compounds penetration of such as sulfates, carbonates, etc. Therefore, the degradation 745 mechanism passes through the pH drop, losses in elastic and mechanical properties (Nguyen et al., 746 2006), the increase of the structures porosity, the dissolution of the portlandite and 747 monosulfoaluminates and decalcification of ettringite and CSH (Guillon, 2004; Rozière & Loukili, 748 2011). 749

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- 751 752

Figure 12 : Experimental installation for constant pH leaching test (Rozière et al., 2009)

Test samples are made from 11x22cm cylinder blocs for concrete or 7x7x28cm in case of mortar. The leaching analysis of this monolith involves immersing for 60 days a sample in a solution regulated at pH=5.0 with a nitric acid solution(HNO<sub>3</sub>) at 0.5 mol/L (Rozière & Loukili, 2011) (Fig. 12). However, the degradation test with pure water seems more representative from a phenomenological point of view (Porteneuve et al., 2001).

Recently in the studies of (Wang et al., 2018), it has been demonstrated that using a dry-mix/press
method, the contaminated sediment can be used as a construction material for partition blocks, paving
blocks, and fill material. Also, the leaching concentrations of toxic elements were acceptable regarding
the on-site reuse.

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#### 7. Conclusion 766

A detailed literature review about the beneficial use of sediment in a cement matrix is proposed in this 767 768 paper. A presentation of the usual characterization techniques is first proposed. This includes a description of the physicochemical characterization methods commonly used and the standards which 769 they refer to. 770

Hydration of cement is a combination of multiple complex chemical reactions which leads to hydrates 771 formation such as the CSH and the portlandite that represent both up to 90% of the hardened cement 772 matrix. Sediments, when they are used in a cement matrix, can interact with these elements (delaying 773 effect, changes in the effective amount of water, etc.). However, with optimized treatment methods, 774 some beneficial properties for cement hydration can be enabled or improved: the pozzolanic activity 775 (by heat treatment) or removal of the organic fraction (chemical treatment), heterogeneous nucleation 776 effect (grinding). This way, the emphasis is laid on the different tests that must be realized for optimal 777 use on sediment into a cementitious matrix. 778

The use in civil engineering sectors of sediment can be forbidden regarding the degree of pollution. 779

We were able to see that most of the existing durability tests for common mortar or concrete can be 780

781 transposed in the case of sediment-based concretes and mortars. Due to the introduction of the 782 performance approach by the NF EN206-1/CN, the use of sediments as supplementary cementitious

783 material may be considered provided that they justify similar performances to an equivalent concrete (control).

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