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1 **From dredged sediment to supplementary cementitious material:**
2 **characterization, treatment and reuse**

3
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1 **From dredged sediment to supplementary cementitious material:**
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8 **ABSTRACT**

9 Nowadays, the preservation of natural resources, the reuse, and the recycling of waste and by-products
10 in the cement industry are gaining increasing attention in a sustainable development project. In
11 this perspective, many studies focus on finding solutions in order to propose environmentally
12 friendly materials. Nowadays the sediments represent a way to preserve the natural resources
13 by their reuse as a secondary raw material in multiple applications (concrete, roads,
14 landscaped mound, etc...). However, they commonly contain contaminants, organic matter
15 (5% to 30%), high water content (50% to 200%), and relatively small particle size ($D_{max} \leq$
16 300 microns). Therefore to improve the mechanical properties of this material as well as its
17 physiochemical ones, specific methods of characterization and appropriate treatment
18 techniques (calcination, chemical treatment, etc...) are required. This article presents a
19 bibliographical review of the efficient use of sediments in cementitious matrix. It details
20 experimental tests that must be performed to ensure the durability of sediment-based structure
21 and assess their environmental impact under prescribed conditions.

22
23 **KEYWORDS:** Sediment - Characterization - Calcination - Pozzolanicity - Durability.
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40 1. Introduction

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

48 Considerable volumes of material are dredged annually in the world. Annually 400 million tons of
49 natural granular material are consumed in France in the construction sector compared to 40 billion
50 tons in the world. In France, more than 56 million m³ of sediment are dredged per year, against 200
51 million m³ in Western Europe (CEDA¹ - 2008), 100 million m³ in China and 300 million m³ in the
52 USA (Oh et al., 2011). Nowadays, for sustainable development reasons, the reuse of materials is
53 strongly encouraged. There are actually three levers for sustainable construction which are: reduction,
54 recycling, and reuse of raw materials (Marie & Quiasrawi, 2012). Dredged sediments seem to be
55 potentially useful as secondary raw material in various civil engineering sectors. These include road
56 engineering by their use in foundation layer (Siham et al., 2008a), manufacturing of building bricks
57 (Cappuyns et al., 2015) and their use as alternative materials by partial substitution to cement or sand
58 in concrete or mortars (Belas et al., 2011; Oh et al., 2011). Moreover, sediments have been used in
59 some countries in important infrastructures: Rotterdam port, the Palm island in Dubai, National
60 Theatre of London, or the artificial island of Chek Lap Kok where the Hong Kong airport is located
61 (Limeira & Etxeberria, 2011). Due to the increase of global cement production in the world that will
62 reach 5 billion tons by 2020, the CO₂ emissions from the cement industry will therefore increase
63 significantly. This CO₂ emission represents 6% of the total anthropogenic greenhouse gases (Gartner
64 & Hirao, 2015; Scrivener et al., 2018; Zea-Garcia et al., 2019). In fact, the production of ordinary
65 Portland cement (OPC) generates 842 kg CO₂/t of clinker. The use of sediment as supplementary
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).

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

¹ CEDA = Central Dredging Association

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.
86 Therefore to classify and evaluate the impact of these particles on cement matrix behavior and
87 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
90 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**

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

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

118 Due to the contaminants with various origins and nature, it is interesting to identify chemical elements
119 using analytical techniques such as inductively coupled plasma (ICP). The liquid sample is vaporized
120 at very high temperatures in the presence of argon gas-induced plasma and causes atomization,
121 ionization, and thermal excitation. These emissions are identified and assessed either by Optical
122 Emission Spectrometry (OES), by measuring the emitted wavelengths; or by mass spectrometry (MS),
123 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

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

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

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

150 Table 1: Characterization methods recommended for sediment studies

	Parameters	Characterization methods	Standards
Physical characterization	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
	Water content	Water content test	NF P94-050
	Clay content	Methylene blue value test	NF P94-068
	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 characterization	Chemical elements	XRF	---
	Oxide elements	XRF	---
	Carbon content	TGA	---
	Mineral and organic pollutants	Leaching test	NF EN 12457-2
Mineralogical Analysis and Microscopy	Identification of minerals	XRD	---
	Quantitative determination of mineral	XRD Rietveld	---
	Morphology	SEM	---
Investigation methods for material and mortars	Resistance	Mortar formulation	NF P 196-1, NF P18-400
	Strength	Compression and flexural test	NF P 196-1, NF P18-400
	Pozzolanicity	Pozzolanic activity tests : (Chapelle, Frattini, Conductimetry)	NF EN 196-5 NF EN 196-6 ASTM C619-A
	Durability	Freeze and thaw, carbonatation, sulfate attack, chloride ion penetration, alkali-	NF P18-454 XP P18-458 NF P18-459 ASTM C1202-97

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

3. Impact of sediments contaminants in cement matrix

The morphology and composition of the organic material present in the sediment confer the typical properties of the latter. When sediments are used in the cement matrix, a treatment to improve sediment properties could be required. It was shown that the presence of organic matter is not without consequences on the physical and chemical properties. For (Bennett et al., 1985) and (Tremblay et al., 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 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).

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^{2+} ions consumption necessary for portlandite crystals formation, and a decrease in the organic molecules adsorption efficiency when superplasticizer is used.

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 transformation and more particularly by ion exchange, precipitation, substitution, etc...

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

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

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:

- *Lead (Pb)* may precipitate on the hydrates surface (Park, 2000a) and may induce a decrease in the compressive strength. This decrease is not significant for (Minocha et al., 2003).
- *Chromium (Cr)* may be absorbed by the hydration products such as CSH (Park, 2000b) and may also take place in pore formation mechanisms.
- *Cadmium (Cd)* can replace the Ca^{2+} ion and leads to the formation of $\text{CdCa}(\text{OH})_4$ ions (Fig. 1).
- *Nitrate (Ni)* leads to a decrease in hydration efficiency by more than 50% while reducing the total porosity (Malviya & Chaudhary, 2006a).
- *Mercury (Hg)* has no significant effect on the total pore volume (Malviya & Chaudhary, 2006a, 2006b).
- *Copper (Cu)* can induce a drop in compressive strength when its concentration is above 8% but under the latter concentration, it may have a beneficial effect (Minocha et al., 2003).

191 • *Zinc (Zn)* has the greatest impact out of all the other minerals pollutants. Its presence leads strongly
192 to lower the compressive strength and a decrease in material density. For (Arliquoie & Grandet, 1990),
193 Zn inhibits the hydration of C₃S and C₃A with a more pronounced effect on C₃S. This phenomenon is
194 due to the formation of an amorphous impermeable film around anhydrous grains named zinc
195 hydroxide (Zn(OH)₂) (Eq. 1 and Fig. 1).



196
197 However, the presence of Ca²⁺ and OH⁻ ions allows the destruction of the seal by forming a calcium
198 hydroxyzincate layer and therefore the hydration reactions continue (Eq. 2).



200
201 • Marine salts such as sodium hydroxide (NaOH) or sodium sulfates with concentrations below 2%
202 cause an increase in short term resistance but this effect is reversed when concentrations exceed 8%
203 (Minocha et al., 2003).

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

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

210
211
212 Figure 2: Solidification/stabilization of pollutants around a cement grain (Yousuf et al., 1995).

213
214
215
216 Figure 3: Inhibition mechanism of cement hydration by pollutants (Hills and Pollard, 1997b)

218 4. The use of sediments as SCMs

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

222 4.1 Use of sediment without treatment

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

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

241
 242
 243
 244 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 style="list-style-type: none"> • Evaluation of the performance and the durability of concrete blocks incorporating 12.5% and 20% of dredged sediments. • Sediment addition should be limited to 12.5% of the concrete mix to ensure the integrity of mechanical properties
(Zhao et al., 2018)	Dredged marine sediment from the Dunkirk Harbor	<ul style="list-style-type: none"> • Study of the use of raw sediment in partial substitution of cement in the manufacture of mortars and concretes • It was demonstrated that concrete C30/37 could be designed with 20% cement replaced by sediment without the use of admixture.
(Limeira et al., 2012, 2013)	Dredged marine sand (DMS) from the Port of Sant Carles de la Ràpita (Tarragona, Spain)	<ul style="list-style-type: none"> • 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) • Strength resistance improvement in mortars compared to the control mix was identified.
(Ozer-Erdogan et al., 2016)	Samples were dredged from İstanbul Ambarlı Port, Mersin Erdemli Fishery Harbor, İzmir PETKİM Container Port, and Samsun Port	<ul style="list-style-type: none"> • Sediments are used as a fine aggregate in the ready mixed concrete • Incorporation of untreated sediments in concrete decreases the mechanical performances due to the presence of sulfates and chlorides ions
(Maherzi et al., 2020)	River sediment from Aire sur la Lys (North of France)	<ul style="list-style-type: none"> • River sediment is used and valorized • Use of sediment in substitution of natural sand in resin mortars has been shown interesting • Mechanical performance and physicochemical properties of products was satisfying
(Xu et al., 2014b)	Urban river sediments were produced by a dredging project in Qinhuai River (China)	<ul style="list-style-type: none"> • The feasibility of the use of urban river sediments as a primary raw material to produce high-insulation brick is established • Bricks with 50% urban river sediments fired at 1,050 °C exhibited more beneficial characteristics: conductivity and compressive strength improvement.
(Liu et al., 2016)	Yellow River sediment comes from Huayuankou	<ul style="list-style-type: none"> • Microstructure and performance of stabilized earth bricks prepared from the Yellow River sediment is studied • Compressive strength and softening coefficient of stabilized earth bricks is further improved by a polymer bonding agent
(Junakova et al., 2015; Junakova & Junak, 2017b)	Sediments dredged from the Klusov and Ruzin reservoirs situated in Eastern Slovakia	<ul style="list-style-type: none"> • Focus on the possibility of reusing sediment from two Slovak reservoirs as an alternative raw material in concrete production • Mixtures containing 40% of binder replacement by the sediment satisfied the strength requirements
(Tang et al., 2011)	Fine sediment deposits dredged from the Shihmen	<ul style="list-style-type: none"> • The investigation revealed the sediments contain all the necessary elements to enable the bloating and calcining

	Reservoir in Taiwan.	<ul style="list-style-type: none"> processes within the commercial kiln Sediment-based materials designed have presented compressive strengths comparable to normal density concretes and were 29%–35% lighter.
(Ennahal et al., 2020)	Marine sediment from Dunkirk Grand Marine Harbor (France)	<ul style="list-style-type: none"> 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)	<ul style="list-style-type: none"> 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 style="list-style-type: none"> The sediment was used as a raw material for the production of fired bricks 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 The compressive strength is improved when natural clay is mixed with sediment and met the requirements for severe weathering (SW) building bricks
(Komnitsas, 2016)	Marine sediment from the ports of Souda in China, Crete and patras in NW Peloponnese, Greece	<ul style="list-style-type: none"> Marine sediments were valorized with construction and demolition waste through alkali activation 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)
(Wei et al., 2008)	Harbor sediment from Taichung Harbor in Taiwan	<ul style="list-style-type: none"> 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.

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™, bioreactor, Rotamix™ 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,

270 2018). Flash calcination is an innovative and low energy consumption method named flash
271 applied to treat efficiently and activate materials. The process of flash calcination is applied to
272 the treatment of dredged sediments to activate some of the sediment phases (including clay
273 phases). The main properties that determine sediment potentiality of reuse are mineral
274 composition, pollution content, and clays.

275 The determination of adequate calcination conditions (in term of time and temperature of
276 calcination) need some optimization studies as described by (Benzerzour et al., 2017). All the
277 modifications induced by the treatment process target to improve sediment performances and
278 produce an activated material.

279
280 In a recent study conducted (Wang et al., 2017), dredged sediment from Kwun Tong Typhoon Shelter
281 in Hong Kong was used as a cement substitution with two types of cement: OPC and MOC
282 (magnesium oxide cement). The results show that using binary cement followed by 1-day CO₂ curing
283 for recycling contaminated sediment could present a novel and green stabilization/solidification
284 technology. The contaminated sediment from Hong Kong was also used by the same authors (Wang et
285 al., 2015, 2017, 2018a, 2018b) as a fill material, partition blocks and paving blocks by using it as an
286 aggregate.

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

288 **4.2.1 Heat treatment by calcination**

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

304
305
306 Figure 4: a) Methodology of calcination and b) calcination lab furnace.

307
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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314 Table 3: Calcination treatment method applied for sediment in previous works in literature

Authors	Sediment origin	Sediment treatment
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(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 µm 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 ₃ PO ₄ then thermal treatment calcination at 650°C for 2 hours
(Agostini et al., 2007)	Marine sediments dredged in the harbor of Dunkirk	Novosol™ 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™ 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 × 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 H ₃ PO ₄ (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 ⁻¹

315 **4.2.2 Flash calcination method**

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

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

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333
334 Figure 5: a) Flash calciner chamber; b) Semi-mobile flash tower; c) Pilot installation (San Nicolas et al., 2013)

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

347 **4.3 Chemical Treatment**

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

Protocol	Effects	Reference
Use 500ml of a concentrated solution between 15 and 30% of H ₂ O 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 ₂ O 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 ₄ P ₂ O ₇ with 22g of NaHCO ₃ 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 CO ₂ .	Strength enhancement Carbon footprint reduction	(Wang et al., 2015)

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These methods have the advantage of being less expensive (10€/L H₂O₂) than the heat treatment, but they do not seem to involve pozzolanic activity of sediments. In addition, the organic fraction is not

373 completely removed using this method (10% to 95%) (Eusterhues et al., 2003; Mikutta et al., 2005;
 374 Theng et al., 1999).

375 5. Chemical activity quantification

376 The quantification of the chemical activity is important in order to test the reactivity of the mineral
 377 addition such sediments in our case. Therefore, the following will describe the different methods used
 378 in the literature to calculate the activity index and coefficient of the addition. Some of the methods
 379 described in the following are based on index calculation referring to a control mortar or alkaline
 380 concentration. For the others, they are based on chemical tests aiming to quantify the lime
 381 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
 385 substitute in mortar or concrete. This value is the result of the ratio between the compressive strength
 386 of standard reference mortar (NF EN 196-1) and the compressive strength of the mortar with 25 w.t. %
 387 of cement substitution by the pozzolanic supplementary cementitious material (De Larrard, 2000).
 388 In 1935, (Bolomey, 1935) established that the strength of mortar can be estimated at 28 days as (Eq.
 389 3):

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

$$R_o = \left(\frac{K_{Bolomey}}{2} \right) \left[\left(\frac{\Delta}{2350} \right)^2 * \frac{C_o}{E} \right]^{3/2} \quad (3)$$

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

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

393 In this study, we will exclusively use the above formula (Eq.4).

394 For substituted mortar, the expression is given in Eq.5:

$$R_o = \left(\frac{K_{Bolomey}}{2} \right) \left[\left(\frac{\Delta}{2350} \right)^2 * \left(\frac{C + \chi_A A}{E} \right) \right]^{3/2} \quad (5)$$

395

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

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

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

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

398 The mechanical activity coefficient χ_A deduced from Eq.8 given below

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

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 determining the amount of lime consumed by a pozzolan after 8 and 14 days (Fig. 6). Then it evaluates the pozzolanic reactions of cements and pozzolanic additions properties. The method consists of an evaluation of hydroxide ion concentration (OH^-), and calcium ions Ca^{2+} expressed as quicklime or calcium oxide (CaO) in a mixture containing 80% cement and 20% pozzolan. The mixture is kept in a thermostatically controlled room at 40°C. This test has been applied in previous studies where the activity of calcined clay pozzolan was established (Gastaldini et al., 2015; Tironi et al., 2013). The following figure is derived from the studies of (Amar et al., 2017) where a detailed description is given.

Figure 6: Frattini's test principle

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:

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

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

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 $\text{Na}_2\text{O}_{\text{equiv}}$ content can be obtained by quantitative XRF analysis.

$\text{Na}_2\text{O}_{\text{equiv}}$ is the equivalent sodium oxide content (Eq.10). The $K_p(28)$ (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).

$$\text{Na}_2\text{O}_{\text{equiv}} = \text{NaO} + 0.658\text{K}_2\text{O} \quad (10)$$

$$K_p(28) = 2.8 - 0.7\text{Na}_2\text{O}_{\text{equiv}} \quad (11)$$

435 **5.5 Method of the conductivity measuring**

436 There seems to be a good correlation between the decrease in conductivity and the formation of
 437 hydrates in a pozzolanic reaction. This test is based on (Luxán et al., 1989) work, which established a
 438 correlation between the pozzolanic properties of one hundred different SCMs and their electrical
 439 conductivity (Delair, 2008). From the first moments of the reaction between a pozzolan and lime, the
 440 authors note a significant decrease in conductivity due to the early binding of calcium by amorphous
 441 silica. The installation diagram is represented in Fig. 7. Testing cells containing pozzolan and lime are
 442 monitored using a conductimeter and an acquisition system. This method is previously presented by
 443 (Amar et al., 2017) where a detailed description is given.

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447 Figure 7: Principe for conductivity measurements

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449 The method of measuring the conductivity consists of:

- 450 1. Add into a beaker 100 ml of saturated $Ca(OH)_2$ solution already thermostatically controlled
- 451 in a water bath at 40 ± 1 ° C;
- 452 2. Measure the initial conductivity denoted C_1
- 453 3. Add 5 g of the powder sample (the material must be dry and fine)
- 454 4. Keep the solution in agitation and measure regularly the conductivity
- 455 5. Note the value of the conductivity after 120 seconds. This value is C_2
- 456 6. Continue until 300 to 400 minutes and typically until stabilization.

457 The value $\Delta_C = C_1 - C_2$ gives the classification of the product according to its level of pozzolanic
 458 properties.

- 459 • (A) $\Delta_C \leq 400 \text{ mS/cm}$: \Rightarrow not a pozzolanic product
- 460 • (B) $400 \text{ mS/cm} \leq \Delta_C \leq 1200 \text{ mS/cm}$: \Rightarrow variable pozzolanic properties
- 461 • (C) $\Delta_C \geq 1200 \text{ mS/cm}$: \Rightarrow High pozzolanic properties

462 **5.6 Accelerated Chapelle method**

463 This test reproduces the pozzolanic reaction between a reactive powder and lime as applied for
 464 metakaolin pozzolanic activity test (NF EN 18 543 standard). The method consists to react one gram
 465 (1gr) of powder with one gram (1 gr) of quicklime (CaO) or portlandite ($Ca(OH)_2$), in 200 ml of
 466 boiling water for 16 hours. Therefore the fixed lime upon the reaction with the powder is estimated
 467 (Mechling, 2000). The experimental system is shown in Fig. 8. The following figure is derived from
 468 the studies of (Amar et al., 2017) where a description is given.

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472 Figure 8: Experimental installation of Chapelle test

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474 After the test, the solution is cooled to a constant temperature at $60^\circ\text{C} \pm 1^\circ\text{C}$ and then the excess of lime
 475 is solubilized with 60g of sucrose diluted in 800 ml of water. By chemical dosage, the mass of lime
 476 consumed can be deduced knowing that the lime initial mass is 1g and the total volume of the solution

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

- 479 • The calcined sediments can be considered pozzolanic regarding the Frattini test results (Amar
 480 et al., 2017; Faure et al., 2017; Snellings & Scrivener, 2016)
- 481 • The Chapelle test inferred that calcined sediment materials can be considered chemically
 482 reactive with an intermediate pozzolanic level (< 66%) (Amar et al., 2017; Faure et al., 2017).
- 483 • From, the electrical conductivity measurements monitored on lime/pozzolan suspension, the
 484 paper also concludes that flash calcined sediment is the most pozzolanic material compared to
 485 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

490 **6. Durability and environmental impact**

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

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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 Dunkirk	(Achour et al., 2019) (Amar et al., 2018)
External sulfate attack	Marine sediments dredged in the harbor of Dunkirk	(Achour et al., 2019) (Amar et al., 2018)
Alkali-aggregate reaction	Marine sediments dredged in the harbor of Dunkirk Reservoir sediment was obtained from the Shih-man Reservoir in Taoyuan County	(Achour et al., 2019) (Amar et al., 2018) (Chiou & Chen, 2013)
Porosity investigation	Marine sediments dredged in the harbor of Dunkirk	(Amar et al., 2018)
Chloride permeability	Marine sediments dredged in the harbor of Dunkirk Dredged silt from reservoirs in southern Taiwan Fine sediment excavated from reservoirs	(Amar et al., 2018) (Wang, 2009) (Hwang & Hung, 2005)
Resistance to the chemical aggressions	sediments collected from different docks of the port of Oran 3% of chloridric acid (H ₂ SO ₄) 3% of chloridric acid (HCL) 5% of chloridric acid (HCL) 5% of chloridric acid (H ₂ SO ₄)	(Aoual-Benslafa et al., 2015) (Benkaddour et al., 2009) (Chiou & Chen, 2013)
Volume of permeable pores	Dredged marine sand extracted from the Port of	(Limeira et al., 2010)

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

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503 6.1 Alkali reaction

504 The alkali reaction is a deleterious reaction that affects the durability of structures. It represents the
505 reaction between hydroxyl ions provided by the hydrated cement paste and the reactive siliceous
506 phases coming from some reactive aggregates. As a result, an expansive calcium silicate gel is formed
507 in the cement matrix (Cyr & Pouhet, 2015; Kleib et al., 2018; Lombardi et al., 1997). According to the
508 study completed by (Fatt & Beng, 2007), this reaction is the combination of three factors: the reactive
509 silica in aggregates; alkali interstitial solution (K^+ and Na^+) of critical thresholds and external water or
510 moisture. The ASTM C 1260 standard recommends a critical limit of 0.6% Na_2O equivalent (Prince &
511 Perami, 1993). The use of sediment as SCMs can induce alkali-silica reaction due to the presence of
512 amorphous silica. However, in aggregates or mineral addition SCMs such as dredged sediments, two
513 phenomena may take place due to its sand-lime nature: alkali-silica reaction or the alkali carbonates
514 reaction (Murlidhar et al., 2016a). The alkali test is used to estimate the potential expansion of
515 aggregates with alkali. The principle is to estimate the evolution of the geometric dimension of mortar
516 4x4x16 cm samples and also mass variations (XP P18-594, ASTM C 1260, standard, (Amar et al.,
517 2018; Murlidhar et al., 2016b).

518 Mortars with different cement/aggregate C / G ratios (0.5 - 1.25 - 2.5) must be prepared for the test.
519 The preparation and mixing of mortars can be performed following NF EN 196-1, taking care to add
520 to the mixing solution 4.0% $Na_2O_{equivalent}$ relative to the cement mass in order to obtain the required
521 alkalinity. The specimens are initially placed in an environment with 90% RH ($20 \pm 1^\circ C$) then removed
522 from the water after $48 h \pm 30 min$ to determine the initial length L_{0i} . The samples are exposed for 5
523 hours in an autoclaved cell at a temperature of $127^\circ C$ and with a relative pressure of 0.15 MPa. The
524 final length L_{1i} is measured after $(18 \pm 1) h$. The material tested is qualified as non-reactive (NR) when
525 the expansion is less than 0.15%, potentially reactive (PR), or PRP. The deformations are then
526 expressed as presented in Eq.12:

$$\varepsilon_i = \frac{L_{1i} - L_{0i}}{L_{0i}} \quad (12)$$

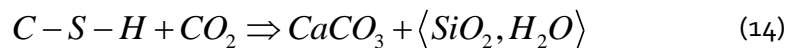
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It should be noted that there are also two different methods: a test on micro mortar and a test based on
chemical kinetics (not applicable if $Al_2O_3 > 5\%$). (Achour et al., 2019) has studied the alkali-aggregate
reaction for two concretes containing 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.

6.2 Accelerated carbonation

Exposed to specific humidity and regarding temperature conditions, concrete can undergo the carbonation process. According to (Rozière, 2007), carbonation is the phenomenon by which carbon dioxide (CO₂) penetrates the concrete and dissolves in the cementitious pore solution and then reacts both with portlandite Ca(OH)₂ which approximately 20% to 30% of the hardened paste (Houst et al., 1989) and calcium silicate hydrates (CSH) (Chaussadent, 1999; Chaussadent et al., 2000). Some characteristics of CO₂ such as its molecular diameter of 0.426 nm and a diffusion coefficient that equals $D = 1.60 \cdot 10^{-5} \text{ m}^2/\text{s}$ at 1 bar, 20°C, allow it to penetrate easily and stay into the cementitious matrix.

The chemical reactions of portlandite and CSH by CO₂ are described in Eq. 13 and Eq. 14 below:



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; Rozière, 2007):

$$x = K\sqrt{t} \quad (15)$$

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% CO₂ and 50% air (Castel et al., 2001; Chaussadent et al., 2000).

Figure 9: Accelerated carbonation test

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

574 **6.3 Porosity accessible to water**

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

$$P_w = \frac{M_{air} - M_{dry}}{M_{air} - M_w} \quad (16)$$

584
 585 Where:

- 586 • P_w = porosity
- 587 • M_{air} is the sample weight in air
- 588 • M_w is the sample weight under hydrostatic weighing
- 589 • 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:**

593 Marine sediments contain chloride salts (calcium chloride, potassium chloride, sodium chloride) that
 594 could involve negative effects when used as a construction material. The presence of salt induces
 595 problems on mechanical performances and can accelerate the corrosion of steel in reinforced concrete.
 596 In fact, for each cement application, a chloride limit is defined according to the European standard EN
 597 206-1 (Table 6).

598
 599 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
 601 The exposure of cement matrix to chloride ions has multiple origins and different consequences. These
 602 chlorides may have two origins according to (Rozière, 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.

605 The experimental installation is shown in Fig. 10. A sample of sawed mortar or concrete is placed
606 between two testing cells containing NaCl and NaOH solutions. A voltage difference allows chloride
607 ions to migrate between cells and then the acquisition system measures the current intensity and the
608 charge passed (coulombs).

609

610

Figure 10: Chloride migration test (Amar, 2017)

611

612 The penetration and spread of chloride ions are induced by the diffusion resulting from a concentration
613 gradient and the capillary absorption that is the fact of the pore connection status and humidity
614 conditions (Basheer et al., 2001). The first phenomenon (diffusion) leads to the measurement of a
615 diffusion coefficient D by migration into an electric field. Existing protocols agree on measuring a
616 coefficient $Deff$ (m^2/s) corresponding to chloride ions which is the flow through the sample in the
617 permanent regime or $Dapp$ (m^2/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,
619 the XP18461 standard provides the determination of the diffusion coefficient by accelerated test in
620 stationary regime by determining $Deff$ (m^2/s). The latter corresponds to ions chloride depth penetration
621 (Hornain, 2007). The mechanisms involved can be quite complex but are often related to the reactions
622 of C3A and C4AF with chloride ions to form an expansive salt named Friedel salt or calcium
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
627 that the chloride permeability of mortars with 10% of calcined dredged sediments in substitution of
628 Portland cement is almost similar to the mortar without substitution. Whereas with a higher cement
629 substitution, mortars performances decrease. (Snellings et al., 2017; Van Bunderen et al., 2019) also
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.

634 It has been shown by (Siham et al., 2008b) that using marine sediment as SCMs without washing
635 decreases the mechanical performances due to the presence of salt. In the study of (Dang et al., 2013),
636 two steps washing process was applied to sediment to reduce the chloride content. The procedure
637 consists to first dilute the sediment with water (ratio sediment to water = 3:1) and second to decanting
638 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
643 also gives an estimation of the effect of certain gases such as carbon dioxide that can induce a
644 depassivation of the matrix, leading to the steel bars corrosion (Basheer et al., 2001). According to
645 (Hermann, 2000), the study of gas permeability is relevant for assessing the carbonation vulnerability
646 and the quality control of concrete, because its gas permeability is higher than water's. The estimation
647 of porosity and is generally between 10^{-14} to 10^{-19} m^2 for ordinary hardened concrete and mortar
648 (Hermann, 2000).

649 The test described by experimental standard XP P18-463 and the AFPC-AFREM methods consists of
650 the exposure of a prepared sample and conserved under constant pressure using low viscous gas (O₂ or
651 N₂). The measuring device can be similar to Cembureau permeameter (Bakhshi et al., 2006; Hornain,
652 2007). The permeability can be assessed at 7 days and 28 days with the following formula derived
653 from Hagen-Poiseuille permeability law (Eq. 17):
654

$$k_x = \frac{2.P_1.Q.L.\mu}{A(P_0^2 - P_1^2)} \quad (17)$$

655
656 Where:

657 k_x = permeability (m^2) ; A = cross section (m^2)

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

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

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

664 **6.6 Freeze-Thaw test**

665 The damages caused by freezing and thawing can probably be considered as one of the most
666 aggressive actions, the most harmful and detrimental to the concrete (Aïtcin, 2003; Liu & Hansen,
667 2016). Concrete is a porous material, consisting of three-phase: voids, aggregates, and water. Below
668 water freezing temperature (about -3°C), water contained in the pores freezes. In this case, it can
669 increase in the volume of about 10% and then create adverse internal hydraulic pressures.

670 According to (Bjegovic et al., 1987), internal pressures can reach 1 to 4 MPa, which exceeds the
671 tensile limit of ordinary concrete. By a cyclic process depending on the cooling rate, the number of
672 cycles, and external temperatures, the damage of the concrete can be reached causing cracking,
673 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 ± 2°C to +9 ± 3°C in a thermally controlled room and for a
679 specific time period.

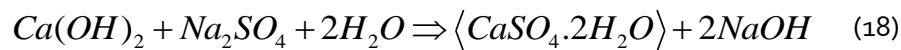
680 According to Faguerlund (cited by (Basheer et al., 2001)), there is a water content threshold beyond
681 which the impact on a cement matrix subjected to freeze-thaw cycles is irrevocable. For (Forster et al.,
682 1994), the impact of the freeze-thaw on a cementitious matrix depends on the geomorphological 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
686 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

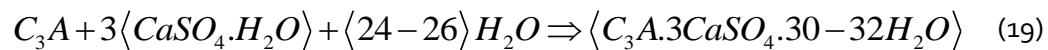
689 organic matter content, the optimization of the granular skeleton, the limitation of the risk of
690 segregation, etc.

691 **6.7 The sulfate attacks**

692 Sulfate attack can be a real and serious threat to concrete durability subjected to aggressive
693 environments (El-Hachem et al., 2012). The mechanism of concrete deterioration concrete, in this
694 case, depends on several factors: temperature, cation associated nature (Na^+ , Mg^{2+} , ...), sulfate ion
695 concentration and also the predominant roles of tricalcium aluminate (C_3A) (around 8% for (Aïtcin,
696 2003)) and the portlandite ($\text{Ca}(\text{OH})_2$) (Neville, 2004; Rozière et al., 2009). Two types of sulfate attack
697 exist the internal and external sulfate attack. According to the NF EN206-1 standard, the environments
698 rich in gypsum, sulfate products, or marine environment, are considered chemically aggressive and are
699 classified as XA or XS exposure. The chemical action of SO_4^{2-} sulphate ions leads to the formation of
700 gypsum, ettringite and thaumasite (Collepari, 1999; El-Hachem et al., 2012). These phases are
701 formed through the following mechanisms (Eq.18 and Eq.19):
702



703



704

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

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

711 The thaumasite ($\text{CaCO}_3 \cdot \text{CaSO}_4 \cdot \text{CaSiO}_3 \cdot 15\text{H}_2\text{O}$) is a reaction produced by the sulfate ions, the CSH,
712 and carbonates. Its formation is enhanced by the low temperature ($< 15^\circ \text{C}$), high relative humidity, and
713 the presence of materials rich in calcite (CaCO_3) (Hermann, 2001). This is also the case for sediments
714 that are often likely sand-silt or clay-limestone or both geological natures. It is also useful to
715 remember the preponderance of parameters such as permeability and portlandite content. The decrease
716 in the amount of portlandite (by pozzolanic reaction for instance) reduces the vulnerability, however, it
717 may induce the degradation of CSH by MgSO_4 solutions.

718 Multiple external sulfate attack tests exist. For instance, the French standard NF 18-837 and US
719 regulations including the ASTM C1012 and ASTM C452 can be applied. The existing protocols
720 consist to immerse the samples in a 5% solution of $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ with immersion/drying cycles
721 (60°C or 105°C). The protocol described in Fig. 11 uses pH regulator to have an accelerated test
722 always representative. Mortar samples $7 \times 7 \times 28 \text{cm}$ are prepared and immersed in a sodium sulfate
723 solution with a $\text{pH} = 7.0 \pm 0.2$ controlled by adding a sulfuric acid solution (H_2SO_4) at 0.02 mol/L. The
724 conduct of the test includes the measurement of mass loss, dynamic modulus, the amount of acid
725 consumed.
726

726

727

728

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

729

730 (Safer et al., 2018) demonstrated that adding calcined dredged sediment up to 20% in concrete induce
731 a comparable resistance to H₂SO₄ attack to that of concrete without sediment. Whereas, the study of
732 (Amar et al., 2018) shows that adding 10% of calcined dredged sediment has equal performances in
733 the external sulfate attack test as the mortar of reference without sediment. Whereas (Achour et al.,
734 2019) showed that the samples with 12.5 % of sediment (used as aggregate) have a better resistance
735 against sulfate attack comparing to the ones containing 20%. For the authors, this behavior is related to
736 the highest porosity in the latter.

737 **6.8 pH constant leaching test**

738 The contact between the concrete surface and fluids leads to several phenomena among which
739 leaching mechanisms. For (Guillon, 2004), the leaching of a cementitious matrix corresponds to the
740 gradual dissolution of the main components and is recurrent in the case of contact with hydraulic
741 structures such as dams, concrete blocks, marine structures, piping systems, tanks, etc. Due to a
742 gradient concentration, the ions diffuse out of the concrete which is at acid pH in the case of
743 lixiviation in water, inducing diverse consequences (Guillon, 2004). The concrete leaching
744 phenomenon is the result of Ca²⁺ and OH⁻ ions transfer that lowers the alkalinity facilitating the
745 chemical compounds penetration of such as sulfates, carbonates, etc. Therefore, the degradation
746 mechanism passes through the pH drop, losses in elastic and mechanical properties (Nguyen et al.,
747 2006), the increase of the structures porosity, the dissolution of the portlandite and
748 monosulfoaluminates and decalcification of ettringite and CSH (Guillon, 2004; Rozière & Loukili,
749 2011).

750
751 Figure 12 : Experimental installation for constant pH leaching test (Rozière et al., 2009)
752

753 Test samples are made from 11x22cm cylinder blocs for concrete or 7x7x28cm in case of mortar. The
754 leaching analysis of this monolith involves immersing for 60 days a sample in a solution regulated at
755 pH=5.0 with a nitric acid solution(HNO₃) at 0.5 mol/L (Rozière & Loukili, 2011) (Fig. 12). However,
756 the degradation test with pure water seems more representative from a phenomenological point of
757 view (Porteneuve et al., 2001).

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

762
763
764
765

766 **7. Conclusion**

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

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

779 The use in civil engineering sectors of sediment can be forbidden regarding the degree of pollution.
780 We were able to see that most of the existing durability tests for common mortar or concrete can be
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
784 (control).

785

786

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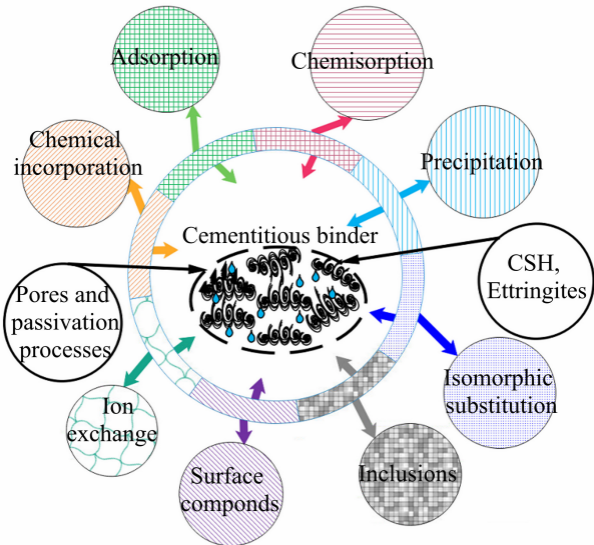
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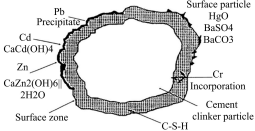
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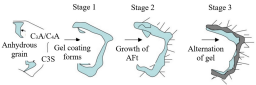
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Sediment sampling

Initial characterizations

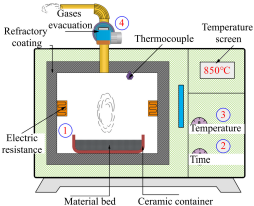
Pre-treatment
Steaming
Crushing

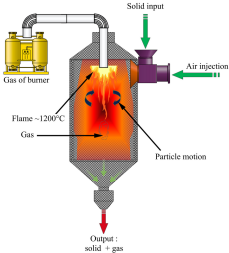
Treatment
Calcination
Cooling

Post-treatment
Characterizations
Leaching tests

Thermal activation

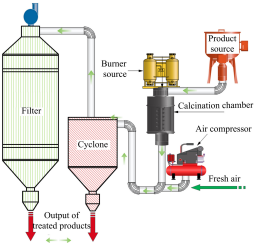






b)





1



Sampling: 100 ml H₂O +
50% cement + 20% polystyrene

2



Incubating at 40°C

3



Analysis after 8 days
and 14 days.

4



Porosity

