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Keywords (separated by '-')	Dredged sediments - Mortars - Sand substitution - Mechanical properties - Thermo-physical properties	





# Mechanical and thermo-physical characterization of mortars made with uncontaminated marine sediments

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## Abstract

In a context of sustainable development, civil engineering must increasingly use recyclable materials to preserve natural resources. Sediments could be a solution in the context of their recovery in several areas. Uncontaminated marine sediments were used to partially substitute sand aggregate in the formulation of mortars. Five mortars were manufactured with different percentages of sand substitution. Physical, chemical, mineralogy, and mechanical characterization were carried out in order to investigate their possible use and impact in cemented-based materials. The mechanical strengths and the elastic modulus of mortars were analyzed. The hydration kinetics of the mortars were studied at different temperatures. The acceleration of the chemical reactions was also assessed through the determination of the apparent activation energy of mortars. Finally, the microstructure of two formulations of mortars was observed by Scanning Electron Microscope (SEM). Results show a drop of the mechanical properties with the increase of the percentage of substitution. This can be attributed to the natural composition of sediments and to the increase of the total porosity of the mixtures. The measured heat released of mortars increase with the increase of sediment content. This result suggests an additional chemical activity linked to the presence of sediments within the matrix.

**Keywords** Dredged sediments · Mortars · Sand substitution · Mechanical properties · Thermo-physical properties

## Introduction

**AQ1** The accumulation of sedimentary particles at the bottom and on the edges of watercourses and shipping lanes as well as in seaports leads to their clutter. For navigable waterways, it slows the flow and prevents activities of transportation. In order to maintain and restore these accesses, it is necessary to dredge or clean up regularly from coast and estuarine lines around the world.

**AQ2** Each year, several millions of tons of materials are dredged throughout the world [1]. In France, around 50 million m<sup>3</sup> of sediments are produced from ports. These materials, which range from rocks to clays, can contain various types and amounts of pollutants and organic matter.

Currently, the French, European and international legislation aims to preserve the environment. Traditional solutions for the management of dredged sediments, such as dumping the sediments at the sea, are constrained. **AQ3**

In this context, due to the shortages of natural resources and the environmental restrictions on the establishment of new quarries as well as the sustainable development approach adopted by several countries, sediment management is increasingly oriented toward valorization processes by respecting technical, environmental, and economic criteria.

The beneficial use of dredged sediments has gained a large acceptance in various domains such as civil engineering, agriculture, and manufacturing.

However, various factors influence the effective use of dredged material, including mineral composition, grain size distribution, and physical and chemical characteristics [2]. Furthermore, there are a number of scientific challenges that must be overcome in order to achieve this beneficial application. Among these, inorganic contaminants, such as heavy metals (copper, chromium, lead, etc.), salts, as well as organic hydrocarbons, such as PAHs (polycyclic aromatic

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56 hydrocarbons), PCBs (polychlorinated biphenyls), and TBT  
57 (Tributyltin), may be present in sediment [3, 4].

58 Various treatments and reuse possibilities are available,  
59 such as brick production [5–8] or to replace a portion of raw  
60 materials in the production of Portland cement clinker [9,  
61 10], sometimes after applying an effective treatment, such  
62 as the Novosol process. Aouad et al. [9] showed comparable  
63 compressive strengths of a commercially produced Portland  
64 cement and a manufactured cement based on sediment.

65 Many studies on the reuse of sediments as a new materi-  
66 al resource in some geotechnical applications as in road  
67 basement construction or backfill were conducted [11–15].  
68 The results showed that the use of additives, such as cement,  
69 lime, fly ash, and slag, for the solidification/stabiliza-  
70 tion of sediments can meet the mechanical characteristics  
71 requirements as well as the environmental impact standards  
72 [15–17]. Depending on the final use, the cost/performance  
73 ratio can be assessed by adjusting the proportions of cement  
74 and admixtures addition.

75 In literature, the reuse of sediments in the formulations  
76 of mortars or concretes were less studied compared to  
77 their reuse in road construction [18–20]. Large amounts of  
78 cement, aggregate, and water are necessary for the manu-  
79 facture of concrete. Many studies have investigated the pos-  
80 sibility of the use of sediments in the cement-based materials  
81 [21, 22]. Due to the mineralogical and chemical constitu-  
82 tion (siliceous, limestone, clayey, etc.) of sediments, their  
83 valorization in such cementitious mixtures is very prom-  
84 ising. It was demonstrated that treated sediment could be  
85 introduced with various proportions into the production of  
86 mortars or concretes. Concrete valorization provides some  
87 flexibility in terms of granular size distribution and the quan-  
88 tities and types of materials that can be substituted (binder  
89 or aggregates).

90 Most of these studies relies on the substitution of cement  
91 by the sediment for the formulations of mortars and con-  
92 cretes [23–27]. However, few studies were conducted on the  
93 replacement of sand by sediments in the cement-based materi-  
94 als [18, 28, 29]. Most studies deal with a simple stabiliza-  
95 tion–solidification process [30–33]. Dredged sediments can  
96 fully or partially substitute the sand aggregate [34, 35]. This  
97 valorization is very interesting in order to solve a current  
98 environmental and economic issue related to the pollution  
99 generated by cement industry, a major emitter of CO<sub>2</sub>, and  
100 the depletion of natural aggregates. The construction indus-  
101 try in France consumes 400 million tons of aggregates per  
102 year where 40% are used for concrete structures.

103 Agostini et al. [28] substituted sand by treated sediment  
104 at replacement percentage of 33%, 66%, and 100%. They  
105 obtained a compression strength enhancement of 33% with up  
106 to 20% of sediment replacement. This result can be explained  
107 by the formation of a denser interfacial transition zone in the  
108 presence of treated sediment despite the high porosity linked

109 to the presence of a fine fraction and its constitution in the  
110 sediments [28, 29]. This porosity can lead to high amount of  
111 absorbed water and high drying shrinkage of mortars.

112 The full substitution of sand by the coarse fraction of  
113 marine sediment was the subject of Couvidat et al. [18]. The  
114 authors confirmed that the use of an 80 µm-sized sediment  
115 offered an interesting valorization potential as a replacement of  
116 sand in mortars for non-structural applications. These studies  
117 focus on the valorization of contaminated and treated marine  
118 sediments.

119 The main objective of this paper is the valorization of  
120 uncontaminated marine sediments, not subjected to any spe-  
121 cific high-cost pre-treatments and the preservation of natural  
122 resources. Sediments are used to replace sand in the manu-  
123 facture of mortars where five formulations were studied. This  
124 research was carried out to demonstrate the feasibility of sub-  
125 stituting sediments by sand aggregate. These sediments were  
126 dredged from the port of Dunkirk, north of France. Physical,  
127 chemical, mineralogy, and mechanical characterizations of the  
128 dredged sediments were assessed in order to ensure a sustain-  
129 able management of sediments and investigate their possible  
130 use and impact in cemented-based materials.

131 This impact was evaluated through the evolution of the  
132 mechanical properties, such as flexural, compressive, and  
133 elastic modulus.

134 In addition, in order to understand the phenomena behind  
135 this substitution, a complete thermo-physical study was con-  
136 ducted. The hydration kinetics was studied by isothermal calo-  
137 rimetry at three different temperatures 20°, 30°, and 40 °C.  
138 The activation energy that describes the acceleration of the  
139 chemical reactions was determined in order to explain the  
140 reactivity issues in the presence of sediments. Therefore, this  
141 study was completed by the determination of the thermal con-  
142 ductivity and the specific heat capacity of mortars. Finally, the  
143 microstructures of a reference mortar and a sediment-based  
144 mortar were given in order to identify the cementitious mineral  
145 phases which participate in the mechanisms mentioned above.

146 This paper describes in Materials and methods, the  
147 materials used for the formulation of mortars and the cor-  
148 responding methods for the characterizations. In the Experi-  
149 ments, the mechanical and thermo-physical experiments  
150 are presented. Results of the different performed tests are  
151 then reported in Results. At the end, an Interpretations and  
152 discussion is dedicated to analyze and discuss the obtained  
153 results, followed by the conclusions of this contribution.

## 154 Materials and methods

### 155 Sediments characterization

156 The used marine sediments were dredged from the Grand  
157 Port Maritime of Dunkirk (GPMD) located in the North of

158 France. This port dredges about 4 million cubic meters of  
 159 sediments per year. The characterization of such materials  
 160 is essential in order to understand their influence and behav-  
 161 ior in cemented matrixes and explain some mechanical and  
 162 physico-chemical properties that can be altered.

163 Physical characterizations of sediments were performed  
 164 and results are summarized in Table 1.

165 The particle size distribution was conducted by laser par-  
 166 ticle size analysis presented in Figure. 1.

167 Chemical analysis was also conducted on the used sedi-  
 168 ments. The proportion of each chemical element was deter-  
 169 mined by X-ray Fluorescence (XRF). The major chemical  
 170 elements are Oxygen (~ 50%), Silicon (~ 18%), and Calcium  
 171 (~ 12%). PAHs value was 5.5 mg/kg and PCBs value was  
 172 0.04 mg/kg, which were below the limited value according  
 173 to AMATR [36].

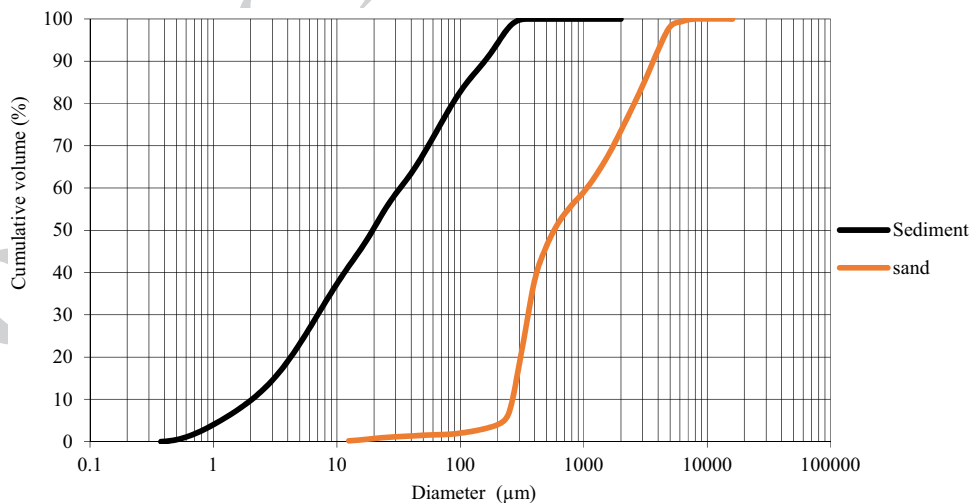
174 In addition, leaching tests of the raw sediments were  
 175 conducted in accordance with the European standard EN  
 176 12457-2 (2002). The proportions of heavy metals, pollutants  
 177 leached and major ions are shown in Table 2. A comparison  
 178 of the obtained values with the inert waste reference thresh-  
 179 olds ISDI (Decree of October 28, 2010 concerning inert  
 180 waste storage facilities) was made. For the used sediments,  
 181 the determined values are below the threshold values with  
 182 the exception of molybdenum (Mo). The studied raw sedi-  
 183 ments were not contaminated with heavy metals, but they  
 184 contained high chlorides and sulfates content.

185 The mineralogical characteristics of the used sediments  
 186 were also investigated using Siemens D5000 diffractometer  
 187 (Fig. 2). The XRD analysis provides the presence of crystal-  
 188 line phases. The major elements detected were quartz (SiO<sub>2</sub>)  
 189 and calcite (CaCO<sub>3</sub>).

**Table 1** Physical characterization of marine sediments

Density (g/cm <sup>3</sup> )	Surface area BET (cm <sup>2</sup> /g)	Organic matter—LOI (450 °C) (%)	Water content (%)	Liquid limit LL (%)	Plastic limit PL (%)	Fine fraction (D < 63 μm) (%)	Clay fraction (D < 2 μm) (%)
2.45	134,472	10.9	23.64	107	46	72.06	7.13

**Fig. 1** Particle size distribution of sediment and sand



**Table 2** Contaminant concentrations in sediments (mg/kg)

Element	Antimony (Sb)	Arsenic (As)	Barium (Ba)	Cadmium (Cd)	Chromium (Cr)	Copper (Cu)	Mercury (Hg)	
Concentration (mg/Kg)	0.066	<0.08	0.370	<0.008	<0.03	0.192	0.004	
ISDI limits	0.1	0.5	20	0.04	0.5	2	0.01	
Element	Lead (Pb)	Molybdenum (Mo)	Nickel (Ni)	Selenium (Se)	Zinc (Zn)	Fluorides	Chlorides	Sulfates
Concentration (mg/Kg)	<0.03	0.618	<0.03	<0.07	<0.06	<2.5	14,900	12,580
ISDI limits	0.5	0.5	0.4	0.1	4	10	800	1000

190 **Formulations**

191 Five mortars were formulated for which the sand being  
 192 replaced by the same volume of sediment at replacement  
 193 percentages of 0%, 5%, 15%, 30%, and 40%, referred to  
 194 MT, MT5, MT15, MT30, and MT40, respectively. The  
 195 reference mortar MT is a standard mortar mixed on a ratio  
 196 basis of 1 part of cement to 3 parts of sand and half part  
 197 of water.

198 The slump of mortars was measured using the Abrams'  
 199 mini-cone (see Table 3). It can be seen that all formula-  
 200 tions have almost the same consistency.

201 Table 3 presents the composition of the various for-  
 202 mulations studied in 1 L of volume. The effective water-  
 203 to-cement (W/C) ratio was kept constant (0.5) for all for-  
 204 mulations. The absorption coefficient of sediments was  
 205 determined according to standard NF EN 1097-6. A value  
 206 of 35% was obtained. The sediments were pre-saturated  
 207 for 2 h before mixing in order to prevent the main issue  
 208 of porous aggregate linked to their high water absorption  
 209 capability. The formulations were prepared according to  
 210 the mixture procedure described in the European stand-  
 211 ard EN 196-1 [37]. For each formulation, three prismatic  
 212 samples of  $4 \times 4 \times 16 \text{ cm}^3$  were prepared and cured until  
 213 28 days. The slump of mortars was measured using the  
 214 Abrams' mini-cone (see Table 3). It can be seen that all  
 215 formulations have almost the same consistency.

216 Portland cement CEM I 52.5R (Calcia Cement from  
 217 Couvrot) was used. The mortar aggregate was CEN sili-  
 218 ceous sand (according to EN 196-1), with rounded grains  
 219 (Class 0/5). This sand has a silica content of at least 98%.  
 220 Its density is  $2.60 \text{ g/cm}^3$  measured by helium pycnometer  
 221 and it has an absorption coefficient equal to 0.7%. The  
 222 particle size distribution of sand is presented in Fig. 1.

**Table 3** Composition of the sediment-based mortars (in 1L)

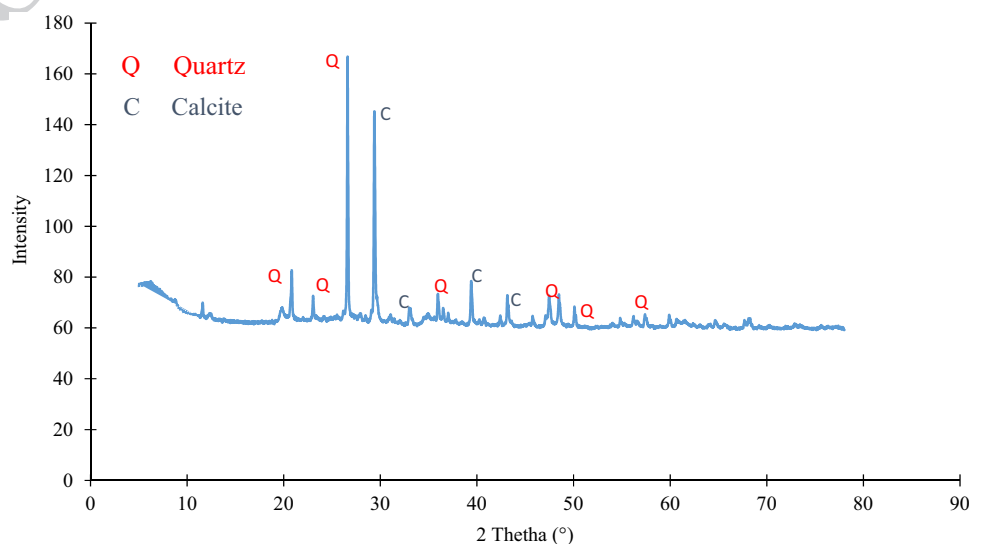
Mortar	MT	MT5	MT15	MT30	MT40
Cement (g)	507	507	507	507	507
Sand (g)	1526	1450	1298	1068	916
Effective Water (g)	253	253	253	253	253
Sediment (g)	0	95	285	570	760
W/C	0.5	0.5	0.5	0.5	0.5
Density ( $\text{g/cm}^3$ )	2.11	2.09	2.07	2.04	1.98
Slump (cm)	0	1.0	1.1	1.2	3.1

**Experiments**

224 The mechanical strengths of the prismatic mortar samples  
 225 were determined in accordance with the European stand-  
 226 ard EN 196-1. Flexural and compressive tests were carried  
 227 out using a mechanical INSTRON testing machine (loading  
 228 capacity of 1500 kN). These tests were performed on the  
 229 reference and sediment-based mortars after being cured in  
 230 water for 7, 14, and 28 days. Results are the averages of  
 231 three samples.

232 A method was developed to determine the elastic modu-  
 233 lus E of the mortars using strain gages. It consists in record-  
 234 ing the deformation of the tested specimen when subjected  
 235 to a compression imposed by the INSTRON testing machine  
 236 with a displacement control. Optimization works were real-  
 237 ized in order to obtain accurate values of the elastic mod-  
 238 ulus. These optimizations concern the length of the used  
 239 strain gages ( $L = 20 \text{ mm}$ ) and their installation on the speci-  
 240 men's surface, the rate of displacement, and the compensa-  
 241 tion of temperature effect.

242 The pore size distribution of mortars was measured  
 243 with a Micrometrics Auto pore IV using Mercury Intrusion

**Fig. 2** XRD analysis on the studied sediment

244 Porosimetry (MIP) technique (Standard NF P 94-410-3).  
 245 Measurements were taken for the reference mortar and for  
 246 the sediment-based mortars. The samples were stored under  
 247 the same conditions as those mechanically tested at 20 °C  
 248 and cured in water for 28 days.

## 249 Heat of hydration

250 Many previous research is limited on the study of the  
 251 mechanical properties of cemented-based materials. In this  
 252 paper, a further study on the thermo-physical properties was  
 253 conducted in order to better understand the influence of sedi-  
 254 ments and the mechanisms that can occur when incorporated  
 255 in mortars.

256 The hydration reaction being thermoactive, an isother-  
 257 mal calorimeter was used at three imposed temperatures  
 258 (20 °C, 30 °C, and 40 °C) that correspond to usual concrete  
 259 applications [38]. For each formulation of mortar, the total  
 260 flux, the heat of hydration, and the activation energy were  
 261 determined.

262 The activation energy of the formulated mortars was  
 263 determined in order to understand the reactivity issues  
 264 when substituting a given proportion of sand by sediment in  
 265 cementitious matrix [39, 40]. The hydration reactions being  
 266 exothermic, at a given time, the amount of heat released is  
 267 the reflection of the overall state of progress of the various  
 268 chemical reactions between cement constituents and mixing  
 269 water. Previous studies have shown that in the case of con-  
 270 crete, the Arrhenius law was the most suitable to describe  
 271 the acceleration of the chemical reactions. It is of the form:

$$272 \quad K(T) = A \exp\left(-\frac{E_a}{RT}\right), \quad (1)$$

273 where  $K(T)$  denotes the kinetic constant at temperature  
 274  $T$ ,  $A$  the constant of proportionality (1/s),  $R$  the perfect gas  
 275 constant = 8.314 J/mol K,  $E_a$  the apparent activation energy  
 276 (J/mol), and  $T$  temperature (°Kelvin).

277 The apparent activation energy is specific to the studied  
 278 composition and must be determined before each maturo-  
 279 metric study. The apparent activation energy must be cal-  
 280 culated for a range of temperatures representative of those  
 281 encountered on the site. For two different thermal histories,  
 282 the apparent activation energy can be determined from the  
 283 progress of the hydration reaction or from the quantities  
 284 related to this progress, such as the compressive strength or  
 285 the heat released by hydration.

286 The method used in this study is the “superposition”  
 287 method. It consists in determining a constant value of the  
 288 apparent activation energy allowing to superimpose as much  
 289 as possible two curves of resistance or heat evolution as a  
 290 function of the equivalent age.  
 291

292 The equivalent age  $t_{eq}$  corresponds to the time during  
 293 which a concrete must be kept at a reference temperature  
 294  $T_{ref}$  (generally 20 °C) to reach the same maturity as under  
 295 real curing conditions. It can be written as

$$296 \quad t_{eq} = \int_0^t \exp\left(-\frac{E_a}{R}\left(\frac{1}{T(\tau)} - \frac{1}{T_{ref}}\right)\right) d\tau, \quad (2)$$

297 where  $T(\tau)$  is the absolute temperature at the moment  
 298  $\tau$  (°Kelvin),  $R$  the perfect gas constant = 8.314 J/mol K,  
 299  $T_{ref}$  the reference temperature,  $E_a$  the apparent activation  
 300 energy (J/mol).

301 The target is finding the value of the apparent activa-  
 302 tion energy that minimizes the differences between the  
 303 different pairs of equivalent ages ( $t_{eq1i}$ ,  $t_{eq2i}$ ) over the tar-  
 304 getted range. This optimization is performed using the least  
 305 squares method. The value of the apparent energy sought  
 306 is the one that meets the following criterion:  
 307

$$308 \quad \min_{E_a} \left( \sum_{X_i \in [X_{inf}, X_{sup}]} |t_{eq1i} - t_{eq2i}|^2 \right), \quad (3)$$

309 where  $t_{1i}$  and  $t_{2i}$  correspond to the actual curing ages for  
 310 the same amount of heat  $Q_i$  (or the same resistance  $R_{ci}$ ),  
 311  $t_{eq1i}$  and  $t_{eq2i}$  are the corresponding equivalent ages.  
 312

## 313 Thermal conductivity and specific heat capacity

314 The thermal conductivity is a measurement of a material’s  
 315 capacity to conduct heat. It is often used in the construc-  
 316 tion industry to assess its thermal performance. It meas-  
 317 ures the amount of heat transmitted due to a unit tem-  
 318 perature gradient through a unit thickness in a direction  
 319 normal to the surface of a unit area in a steady condition.  
 320 Thus, to evaluate the influence of sediments on mortars,  
 321 the thermal conductivity ( $\lambda$ ) of samples was determined  
 322 with the heat flux meter method, based on the EN 12664  
 323 standard [41]. This method involves measuring tempera-  
 324 tures and heat flow on both sample faces at the same time.  
 325 Surface temperatures are measured by T-type thermocou-  
 326 ples and heat flux are measured by flux meters. The tem-  
 327 peratures are applied on both sides of the sample thanks to  
 328 two exchanger plates and controlled by bath thermostats.  
 329 The sample and flux meters are surrounded by an insulat-  
 330 ing boundary to ensure unidirectional flow in the central  
 331 measurement area. To determine  $\lambda$ , a temperature gradient  
 332 is generated ( $\Delta T = 10$  °C) and the thermal conductivity is  
 333 calculated for an almost zero heat flux difference on both  
 334 sample sides. The thermal conductivity is expressed by  
 335 Eq. 4:



$$\lambda = \frac{\sum \phi \times e}{2 \times \Delta T}, \quad (4) \quad C_p = \frac{Q}{\rho \times A \times e \times \Delta T}, \quad (5)$$

with  $\lambda$ : thermal conductivity of the material ( $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ ),  $e$ : sample thickness (m),  $\Sigma\phi$ : sum of both recorded heat flux at the sample surface ( $\text{W} \cdot \text{m}^{-2}$ ), and  $\Delta T$ : temperature gradient ( $^{\circ}\text{C}$ ).

The specific heat capacity is also an important parameter for construction materials. It depends on the density and the nature of the material in study. It characterizes the ability of a material to store heat and represents the amount of heat per mass unit required to raise the temperature by one degree. To determine the thermal capacity of a material, the method is based on the process of heat storage of the material. In the initial state, the system is isothermal: the exchange plates are at temperature ( $T_{\text{init}}$ ). The final system is also in an isothermal state ( $T_{\text{fin}}$ ). The sample stores an amount of energy that represents the system's internal energy variation between these two states ( $Q$ ). The material's specific heat ( $C_p$ ) can then be calculated using Eq. (5). A higher specific heat capacity value means that the material can store more heat and contribute more effectively to building insulation.

with  $C_p$ : specific heat capacity ( $\text{J} \cdot \text{K}^{-1} \cdot \text{kg}^{-1}$ ),  $e$ : sample thickness (m),  $\rho$ : density ( $\text{kg} \cdot \text{m}^{-3}$ ),  $A$ : surface of the material in contact with the exchange plates,  $Q$ : stored energy (J), and  $\Delta T$ : temperature gradient ( $^{\circ}\text{C}$ ).

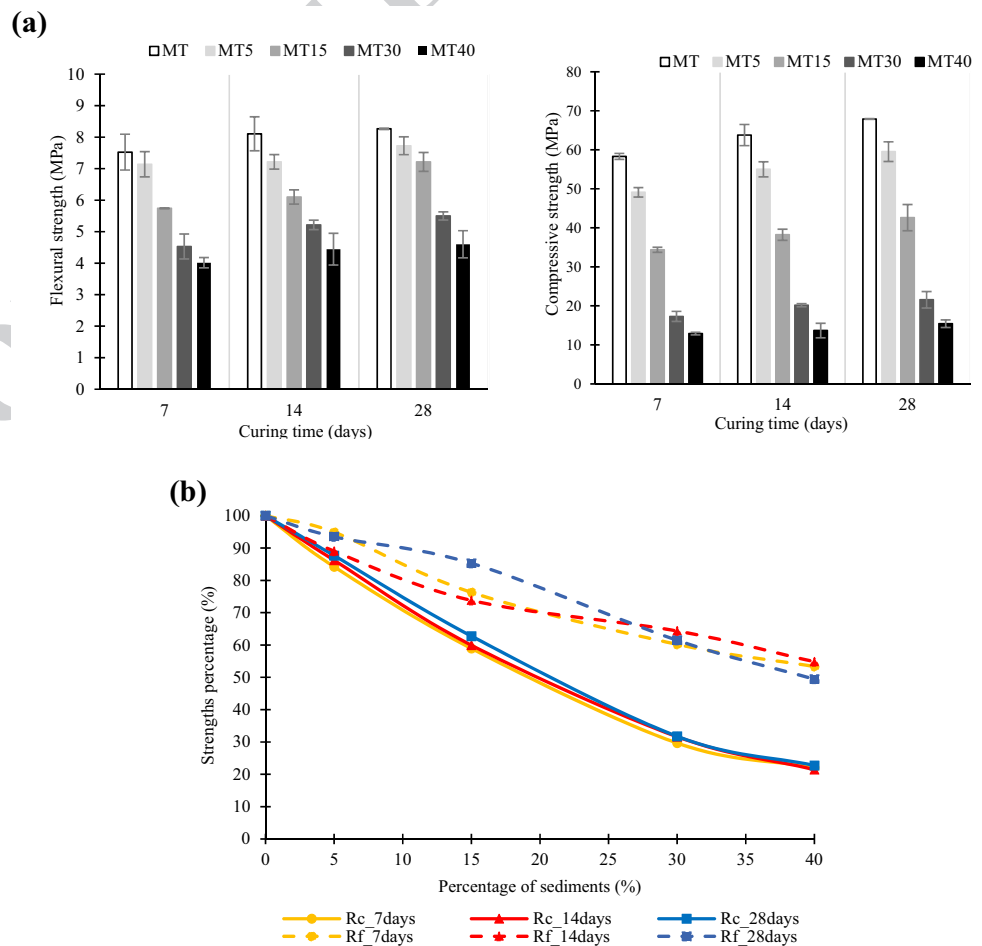
## Results

### Mechanical properties

#### Flexural and compressive strengths

The flexural and compressive strengths of the reference and sediment-based mortars are presented in Fig. 3a. Average values obtained by three measurements for flexural strength and six measurements for compressive strength are shown with the corresponding standard deviation. The mechanical strengths of all the formulated mortars increase with the curing time as expected.

**Fig. 3** **a** Flexural and compressive strengths of mortars at 7, 14, and 28 curing days. **b** Flexural strength (Rf) and compressive strength (Rc) percentage of mortars at 7, 14, and 28 curing days



Both the flexural and compressive strengths of mortars decreased with the proportion increase of sediment for 7, 14, and 28 days of curing in water.

This decrease is linear with the amount of substitution of sand by sediment up to 30% of replacement, independently of the curing time as illustrated in Fig. 3b. Rc and Rf stand for the compressive and flexural strengths, respectively. For MT5, the compressive strength dropped by 13%, while 38% for MT15, 69% for MT30, and 77% for MT40. For the flexural strength, it dropped by 10% for MT5 to 50% for MT40.

### Elastic modulus

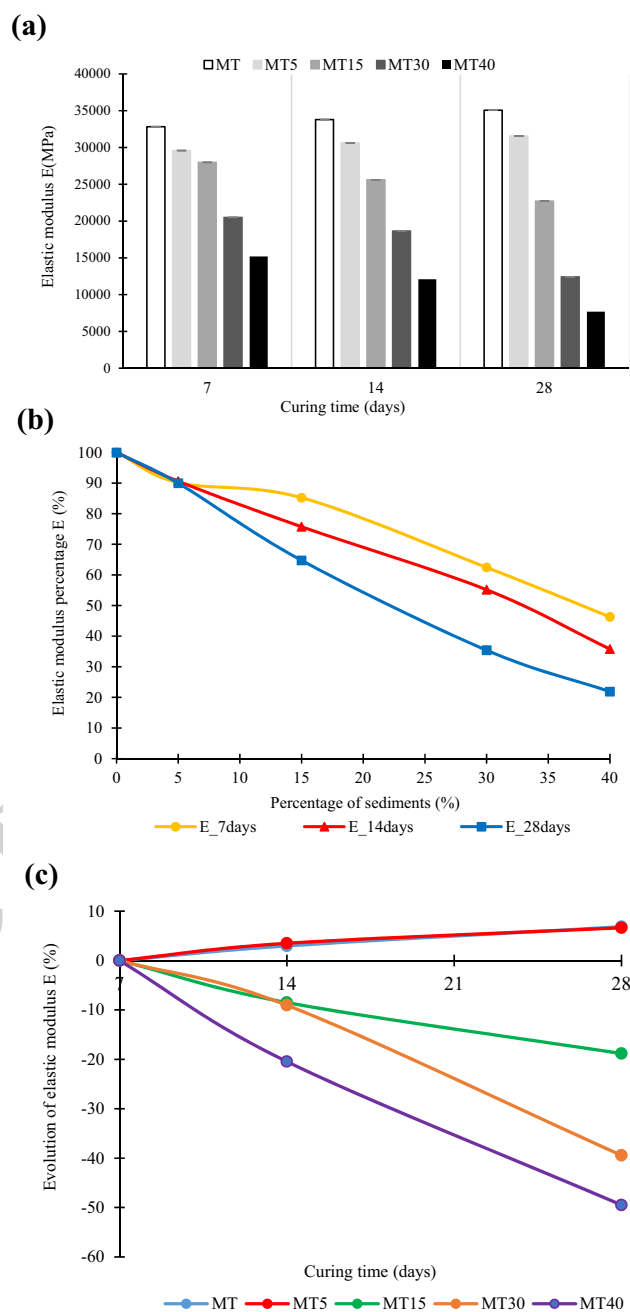
The average elastic modulus of three measurements for each formulation of sediment-based mortars are presented in Fig. 4, for 7, 14, and 28 curing days. The errors on the measurements are less than 5%. The elastic modulus E decreases when the percentage of replacement of sand by sediment increase independently of the curing time. The mortars became more deformable with the increase of substitution percentage. The elastic modulus of the reference mortar is equal to 35000 MPa, while the sediment-based mortar MT40 has a value around 8000 MPa at 28 days.

The percentage of decrease of the elastic modulus at 28 days with respect to the reference mortar MT is similar to the results obtained for the compressive strength. 10% of decrease was noticed for MT5 whereas approximately 80% for MT40 (see Fig. 4b).

In addition, it is remarkable that the elastic modulus of MT and MT5 increase between 7 and 28 curing days contrary to the other formulations. This can be linked to the formation of an internal cracking for high substitution rate of sediments due to the presence of some disruptive elements that will be discussed in Interpretations and discussion.

### Mercury porosity

The porosity of a cement matrix is a fundamental measurement for characterizing the microstructure and determining the material's durability. This test yields a global result ("total porosity"), which is an indicator of the quality of the material [42]. It evaluates the percentage of connected voids. The test results at 28 curing days are given in Table 4. The results confirm that the presence of sediments modifies the pore structure. The average pore diameter is smaller for mortars having high percentage of sediments. However, the total intrusion volume and total pore area are higher for MT40 than for mortars with low percentage of replacement. This result can be refined by presenting the pore size distribution of mortars in Fig. 5. The mortars with high percentage of sediments (MT30 and MT40) have higher proportion of small pores but lower large pores. This highlights the porous nature of the used marine sediments. The total porosity of



**Fig. 4** a Elastic modulus of mortars at 7, 14, and 28 curing days. b Percentage of elastic modulus of mortars at 7, 14, and 28 curing days. c Evolution in time of the elastic modulus (%) of mortars

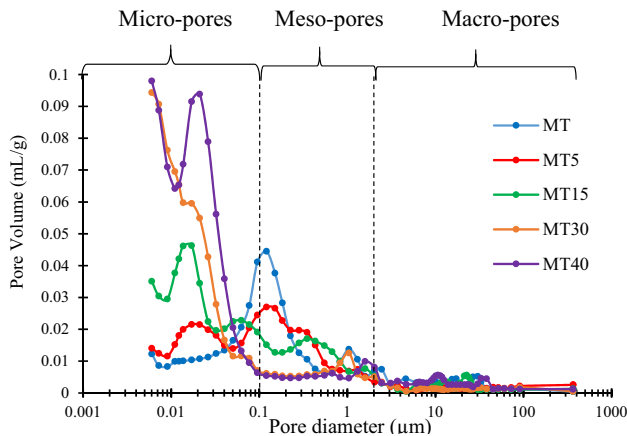
mortars increases with the percentage of sediments. MT presents 9.92%, whereas MT40 is equal to 16.56%. MT is the least porous mortar in this study.

### Heat of hydration measurements

The results of the hydration kinetics observed on the five formulations of mortars are presented in this section. The

**Table 4** Mortars porosity using a mercury porosimetry analysis at 28 curing days

Formulations	MT	MT5	MT15	MT30	MT40
Average pore diameter (4 V/A) ( $\mu\text{m}$ )	0.05372	0.03851	0.02470	0.01510	0.01704
Total intrusion volume (mL/g)	0.0458	0.0446	0.0559	0.0658	0.0841
Total pore area ( $\text{m}^2/\text{g}$ )	3.41	4.63	9.05	17.42	19.75
Porosity (%)	9.92	9.85	11.95	13.45	16.56

**Fig. 5** Pore size distribution of mortars measured by MIP at 28 days

total flux and the heat of hydration  $Q$  (J/g) of the formulated mortars at the three imposed temperatures 20, 30, and 40 °C are presented in Fig. 6a–f, respectively. The heat of hydration  $Q$  (J/g) can be determined by an integration of the heat flux. The start time of the hydration reaction is calculated for each mortar at 20 °C and 30 °C (Table 5). It represents the end of the “latent period” where the flux changes are very small and the beginning of the “acceleration period” where the flux starts to evolve. It is difficult to determine such period for the formulated mortars at 40 °C due to the accelerating effect of temperature on the hydration reactions of cement as it is clearly shown in Fig. 6a–c. Thus, the latent period is very short at 40 °C compared to 20 °C. It can be noticed a delay when the percentage of sediments increase for both temperatures. At 20 °C, the hydration reaction started after 163 min for the reference mortar MT while after 200 min for the sediment-based mortar MT40. Nearly the same delay effect was observed at 30 °C, the reactions begins after 54 min for MT while 96 min for MT40.

One can also notice an increase of the maximum heat flux with the increase of sediment substitution. Same trend was observed for the total heat flux at the imposed temperatures 20, 30, and 40 °C. However, at high temperature (40 °C), there is an acceleration of the hydration reactions of cement. This effect can be highlighted by presenting the results of the activation energy in the upcoming section.

Comparing the heat curves of the formulated mortars, it is apparent that the heat released of mortars increases with

the increase of sediment content. At 20 °C, the heat released for MT is 333 J/g whereas 383 J/g for MT40. At 30 °C, it is 315 J/g for MT and 367 J/g for MT40 while at 40 °C, 315 J/g for MT and 376 J/g for MT40. The reaction is much more accelerated when the temperature increases. These results could suggest an additional chemical activity due to the presence of sediments within the matrix [43].

In order to describe more precisely these observed phenomena, a parameter was determined which refers to the apparent activation energy  $E_a$  described previously in Results. In this paper, a superposition method for determining this parameter for the formulated mortars is presented. This method is based on the evolution curves of the heat released which define the maturity of the hydration reaction of cement.

An apparent activation energy value is calculated for each pair of temperatures (20–30 °C), (30–40 °C), and over the entire temperature range (20–40 °C) for the different formulated mortars. These results are presented in Table 6.

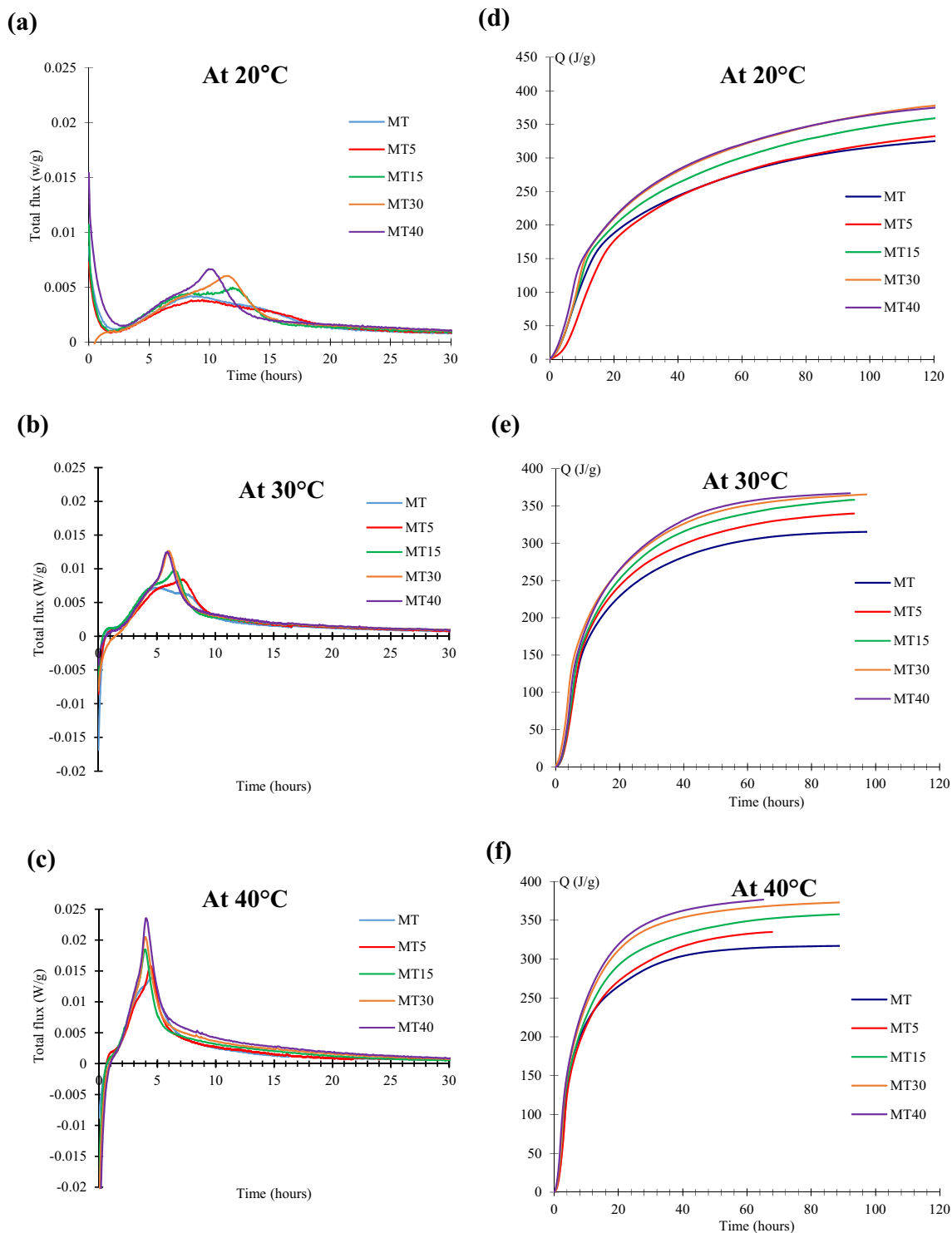
It can be noticed that the apparent activation energy is higher when the substitution percentage of sediments is high. At the range (20–30 °C),  $E_a$  for MT is 31.4 kJ/mol, whereas  $E_a$  for MT40 is equal to 36.8 kJ/mol. In addition, one can observe that the apparent activation energy varies with the temperature. It is obvious that  $E_a$  is higher for higher temperatures.

The evolution of the apparent activation energy is highlighted in Fig. 7. A quasi-linear evolution of  $E_a$  is observed as function of sediment percentage for the three pairs of temperature. This evolution is small up to 5% of substitution. Therefore, the hydration reaction is little affected by this introduction of sediments. However, the evolution of  $E_a$  is much more important for MT15, MT30, and MT40.  $E_a$  increases around 9%, 15%, and 18%, respectively, for each pair of temperature. One can say that the effect of sediments is not modified with the increase of temperature.

### Thermal conductivity and specific heat capacity

Figure 8 presents the evolution of the thermal conductivity and the specific heat capacity of the reference mortar (MT) and sediment-based mortars.

The thermal conductivity of (MT) is  $1.25 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  in conformity with the values obtained in literature [44–46], while it decreases to  $0.66 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  for MT40. This



**Fig. 6** Total flux (a,b,c) and heat of hydration  $Q$ (J/g) (d,e,f) of the formulated mortars at imposed temperatures 20°, 30° and 40 °C

499 decrease is linear with the percentage of sediments incor-  
 500 porated in the formulated mortars. It ranges from 10 to 50%  
 501 for MT5 and MT40, respectively.

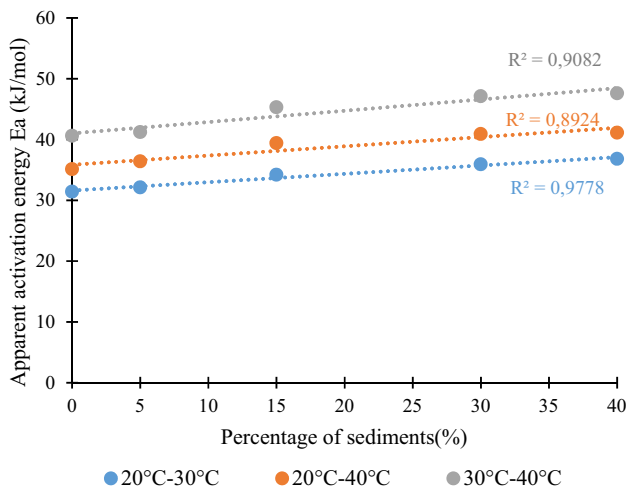
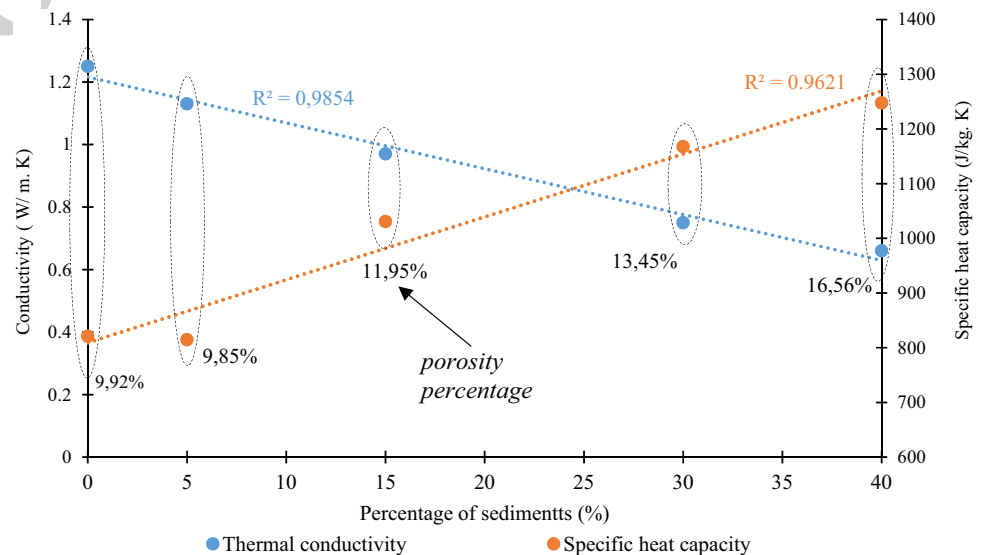
Contrary to the thermal conductivity, the specific heat 502  
 capacity increase in the presence of sediments.  $C_p$  increases 503  
 from 821 J/kg. K for MT to 1247 J/kg. K for MT40. An 504  
 increase of 50% was observed for 40% of sand replacement. 505

**Table 5** Hydration start time of mortars (min)

Temperatures	MT	MT5	MT15	MT30	MT40
20 °C	163	165	170	180	200
30 °C	54	57	78	90	96

**Table 6** Values of the apparent activation energy  $E_a$  (kJ/mol) determined by superposition method

Range of temperatures (°C)	MT	MT5	MT15	MT30	MT40
20–30	31.4	32.1	34.2	35.9	36.8
20–40	35.1	36.4	39.4	40.9	41.1
30–40	40.6	41.2	45.3	47.1	47.6

**Fig. 7** Evolution of the apparent activation energy of mortars**Fig. 8** Evolution of the thermal conductivity and the specific heat capacity of mortars

## Microstructural properties: scanning electron microscopy observations.

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The microstructures of the reference mortar MT and the sediment-based mortar MT30 were observed by Scanning Electron Microscope (SEM) with back-scattered electron diffraction.

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After 28 days curing in water, pieces of 1cm<sup>3</sup> were cut from the core of the mortar samples. All samples were epoxy impregnated and polished according to a standardized process.

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Figure 9 displays the textures and cementitious phases microstructures obtained for the studied mortars MT and MT30.

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From a qualitative point of view, no major differences in terms of cement matrixes can be observed between the reference and the sediment-based mortar in the SEM images. Crystalline portlandite, needle-shaped ettringite and amorphous calcium-silicate-hydrates (C-S-H) can be clearly recognized. Pores of various sizes can also be seen among the solids.

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However, C-S-H gel was found in greater amounts in samples of the reference mortar compared to MT30. It was observed that the C-S-H mineral phases were well formed, and large amounts of hydration products covered the particles forming a dense and homogeneous microstructure. The well formation of C-S-H improved the microstructure by improving the solid-to-solid phase connectivity and minimizing the pore-to-pore connectivity.

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Diverse CH crystallization was observed in the sediment-based mortar MT30, with thin hexagonal and globular-shaped portlandite crystals (alite and belite).

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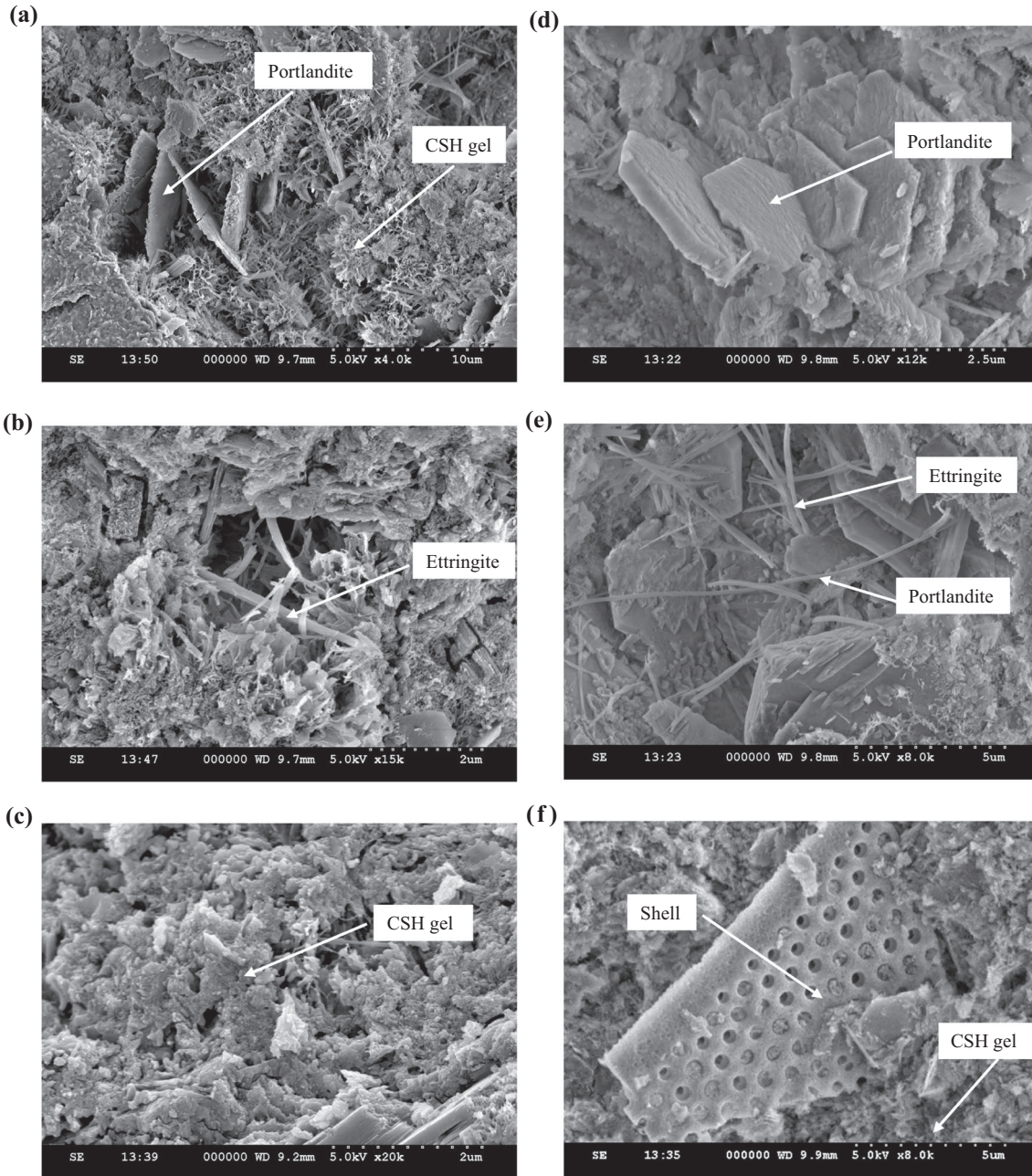


Fig. 9 SEM with back-scattered electron pictures of MT (a, b, c) and MT 30 (d, e, f) samples at 28 days of curing time

537 **Interpretations and discussion**

538 The feasibility of the uncontaminated marine sediments  
 539 used in the formulation of mortars as a replacement of

sand aggregate was assessed.

The mechanical strength is an important characteristic  
 that has to be determined for the purposes of valorization.

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It allows the evaluation of the material's physical integrity as well as the determination of its potential reuses.

The mechanical strength comes mainly from the formation of cemented products. The hydrating processes are affected by introducing the sediments as aggregates.

The used sediments contain 10.9% of organic matter (Table 1). These latter are known to interfere with the hydration process of cement as mentioned in previous works [47, 48]. In particular, it is known that the most acidic fraction prevents the formation of CSH phases by lowering the pH of water.

The marine sediments contain also chloride salts (Table 2). Their high salinity has a negative impact on the mechanical strength development of cement-based materials. The influence of salts was the subject of a previous study conducted by Kamali et al. [13]. The authors have found a low mechanical performance of mortars mixtures made with unwashed sand. They demonstrated that the effect of chloride contained in sediments can be reduced by a washing process.

Besides, traces of metals are contained in the studied sediments (Table 2). Although their concentrations are low, they can influence the quality of the obtained mortars.

The presence of constituents, such as Zinc (Zn), Cadmium (Cd), or Chromium (Cr), may alter the hydration processes and have a negative impact on the mechanical strength by affecting the mortar's average porosity according to Arliguie and Grandet [49]. Zinc presents the largest impact out of all the other pollutants. Its presence causes a significant reduction in the compressive strength by affecting the hydration of  $C_3S$  and  $C_3A$  [55, 56]. Bhatti [52] previously investigated the impact of all trace elements in cement production and consumption. The mechanisms that normally lead to the development of hydrates (CSH) or the pores network can be disturbed [51, 53, 54].

Same trend was observed for the elastic modulus which decreases in the presence of sediments (Fig. 4). It is known that the elastic modulus is highly influenced by the aggregates modulus that compose the cemented materials as well as by their form and texture [55]. The decrease with the sediment substitution can be explained by the means of two factors. Sediments are known to have low resistance compared to natural aggregates like sand. In addition, sediments are high porous by nature. This high internal porosity generates an increase of the mortars porosity as it is given in Table 4 where the sediment-based mortar MT40 has the highest total porosity (16.56%).

As described previously, the hydration in presence of sediments is modified due to the existence of some heavy metals, organic matter, salts, such as chlorides, sulfates, etc. This modification is accentuated with the quantity of such elements which is directly correlated to the substitution rates of sediments in the cement matrix. In the presence

of these elements, we have a dissolution of the portlandite and a progressive decalcification in time of the CSH leading to an increase in their porosity [56, 57]. These phenomena cause internal cracking, which explains the decrease of the elastic modulus as a function of time, particularly for mortars MT30 and MT40 [58–60]. The elastic modulus is more sensitive to these variations compared to the resistances. For the latter, these effects are masked due the continuation of hydration and the increase of the bonds between the CSH. It is noticed that we have almost no evolution of resistances for the mortars with high substitution rates of sediments.

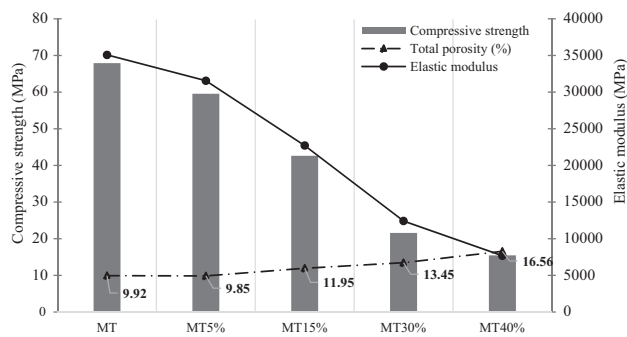
The micro-porosity of the sediments can be observed when comparing the distribution of porosity (Fig. 5). The pore structure can be divided into three main ranges as it was reported in Cheng et al. [61]. The micro-pores which correspond to the pore diameter  $< 0.1 \mu\text{m}$ , the meso-pores for pore diameter ranging between  $0.1 \mu\text{m}$  and  $2 \mu\text{m}$ , and the macro-pores for pore diameter  $> 2 \mu\text{m}$ . The mortars with high percentage of replacement have a porosity distribution ranged in the first domain representing micro-pores. This can be explained by the internal porosity of the used marine sediments.

The meso-pores and macro-pores which come mainly from water loss are low. This can be attributed to the fact that the sediments were pre-saturated before mixing. Thus, the water added for saturation is sufficient to respond to the water demand of sediments due to the presence of high concentrations of organic matter and clays.

There exists a certain link between the mechanical properties discussed. Figure 10 represents the link between the compressive strength, the total porosity, and the elastic modulus of the formulated mortars. A clear correlation is observed. When porosity is high, the mechanical strength and the elastic modulus are low. The mechanical strength decrease is linked to the total porosity [62].

According to Verbeck and Helmuth [63], a change in porosity ratio of 1% can result in a 10% loss in elastic modulus. Same result was obtained in this study where the elastic modulus of MT40 decreases by 80% when its porosity increases by around 8%. The resistance of mortars is primarily a consequence of their porosity. Up to 30% of sand replacement by sediment, the decrease of the mechanical properties is significant. Between 30 and 40%, this effect is reduced. Similar results were obtained by Couvidat et al. [18], who fully replaced sand by sediments. The authors obtained a linear correlation between the increase in total pore area and the decrease in compressive strength.

The hydration kinetics of the formulated mortars were investigated by means of the isothermal calorimeter. The heat released is higher in the presence of the used sediments (Fig. 6). The physical characterization presented in Table 1 shows that 70% of the sediments have a diameter lower than  $63 \mu\text{m}$  and mineralogical characteristics of



**Fig. 10** Compressive strength of mortars linked to the total porosity and elastic modulus at 28 days of curing time

sediments revealed that the major elements are calcite and quartz. Regarding these findings, it seems that sediments are not totally considered as inert admixture. However, they have a certain chemical activity linked to their granulometry and mineralogical composition. They can participate in the exothermic reactions of hydration. It seems also that the quality of the bonds between the hydrates formed in the presence of sediments are altered despite the increase of heat released. This assumption is confirmed by the degraded properties determined previously.

A small delay of the hydration was observed (Table 5) in the presence of sediments. This can be associated to the presence of some heavy metals, such as Zinc (Zn) [49], and also to the presence of marine salts in sediments as shown by Minocha et al. [50].

Apparent activation energy was calculated for the formulated mortars. An increase was observed when the percentage of replacement of sand by sediments increase. This can be explained by the fact that higher energy is necessary to activate the hydration reaction of cement in the presence of the used sediments. This result confirm the decrease of the mechanical strengths observed previously. Bresson [64] have found a correlation between the activation energy of a cement and its actual class at two days. Waller [65] compared values of the activation energy for different types of cement and demonstrated that the activation energy is inversely proportional to the mechanical strength.

The high porosity observed in the sediment-based mortars has also an influence on the thermal conductivity and the specific heat capacity (Fig. 8). The available voids inside the cement-based materials and the sediment's internal porosity have a significant effect on the thermal conductivity due to the low thermal conductivity of air [66]. In addition, the rise of the specific heat capacity when substituting sand by sediment can be explained by the decrease of the density of mortars as shown previously in Table 3 [67, 68].

## Conclusions

This study investigated the potential use of uncontaminated marine sediments to replace sand aggregate in mortars. Five mortars were formulated for which the sand being replaced by the same volume of sediment at replacement percentage of 0%, 5%, 15%, 30%, and 40%.

Based on the different experimental tests carried out, the following conclusions can be drawn:

- The used sediments had important fine particles content ( $70\% < 63 \mu\text{m}$ ). They were classified as uncontaminated sediments containing high amount of salts. They were mainly composed of quartz ( $\text{SiO}_2$ ) and calcite ( $\text{CaCO}_3$ ).
- The mechanical properties of mortars decreased with the increase of sediments percentage. Up to 30% of sand replacement, the decrease is significant. It attained 70% for the sediment-based mortar MT40.
- The total porosity increased as the sediment substitution content increase, but the pore size distribution was shifted toward smaller particles. This highlights the porous nature of the used marine sediments. MT is the least porous mixture (9.92%). The increase in total porosity is linked to the decrease of the mechanical strengths.
- The total flux and heat released measured were higher in the presence of sediments. A delay of the exothermic hydration reactions was also observed. Sediments had a certain chemical activity linked to their granulometry and mineralogical compositions.
- A linear evolution of the apparent activation energy  $E_a$  was observed as function of sediments percentage. An increase of 18% was observed for MT40. Higher energy was necessary to activate the hydration reactions in the presence of sediments which confirm the decrease of the mechanical strengths.
- The thermal conductivity  $\lambda$  of mortars decreased, while the specific heat capacity  $C_p$  increased with the percentage of sand replacement. An evolution of 50% was observed for MT40. This can be attributed to the high porosity and low density of the sediment-based mortars. MT is the mixture having the highest density correlated to the lowest value of  $C_p$  and highest value of  $\lambda$ . The higher the  $C_p$  value, the more the material retains the stored heat and contributes to the insulation of the construction.
- Cementitious mineral phases (CSH, portlandite, ettringite) were identified in the reference (MT) and sediment-based (MT30) mortars. In particular, CSH were well formed and were found in greater amount in MT.

From the mechanical and thermo-physical experimental results of this study, the uncontaminated marine sediments



736 can be used as cementitious materials in mortars. The  
 737 obtained properties confirm the possibility of sediments  
 738 valorization depending on the intended applications. The  
 739 sediment-based mortar MT40 has the lowest compressive  
 740 strength of 15 MPa. This value is higher than the minimum  
 741 strength required for structural applications (8 MPa).

742 One can mention that the origin of sediments (marine or  
 743 fluvial) could influence the properties of recovered materials  
 744 especially concerning the salinity in presence. Supplemen-  
 745 tary experiments would be needed to assess the durability  
 746 of mortars.  
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## 748 Declarations

749 **Conflict of interest** The authors declare that they have no known compet-  
 750 ing financial interests or personal relationships that could have ap-  
 751 peared to influence the work reported in this paper.

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