

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

Amani Abdallah, Hassina Kada, Abdelwaheb Amrouche

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Corresponding Author	FamilyName	Abdelwaheb					
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	Given Name	Amrouche					
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	Division	Junia, ULR 4515 – LGCgE, Laboratoire de Génie Civil Et Géo-Environnement					
	Organization	Univ. Artois, Univ. Lille, Institut Mines-Télécom					
	Address	Technoparc Futura, F-62400, Béthune, France					
	Phone						
	Fax						
	Email	abdelwaheb.amrouche@univ-artois.fr					
	URL						
	ORCID	http://orcid.org/0000-0003-2173-409X					
A							
Author	FamilyName Particle	Abdallah					
		Amoni					
	Given Name	Amani					
	Suffix						
	Division	Junia, ULR 4515 – LGCgE, Laboratoire de Génie Civil Et Géo-Environnement					
	Organization	Univ. Artois, Univ. Lille, Institut Mines-Télécom					
	Address	Technoparc Futura, F-62400, Béthune, France					
	Phone						
	Fax						
	Email						
	URL						
	ORCID						
Author	FamilyName	Kada					
<i>i</i> unor	Particle	11404					
		Wester.					
	Given Name	Hassina					
	Suffix						
	Division	Junia, ULR 4515 – LGCgE, Laboratoire de Génie Civil Et Géo-Environnement					
	Organization	Univ. Artois, Univ. Lille, Institut Mines-Télécom					
	Address	Technoparc Futura, F-62400, Béthune, France					
	Phone						
	Fax						
	Email						
	URL						
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	sediment content. This result suggests an additional chemical activity linked to the presence of sediments within the matrix.						
Keywords (separated by '-		Mortars - Sand substitution - Mechanical properties - Thermo-physical properties					

Footnote Information

ORIGINAL ARTICLE



Mechanical and thermo-physical characterization of mortars made 2 with uncontaminated marine sediments 3

4 Amani Abdallah¹ · Hassina Kada¹ · Amrouche Abdelwaheb¹

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7 Abstract

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8 In a context of sustainable development, civil engineering must increasingly use recyclable materials to preserve natural 9 resources. Sediments could be a solution in the context of their recovery in several areas. Uncontaminated marine sediments 10 were used to partially substitute sand aggregate in the formulation of mortars. Five mortars were manufactured with different 11 percentages of sand substitution. Physical, chemical, mineralogy, and mechanical characterization were carried out in order 12 to investigate their possible use and impact in cemented-based materials. The mechanical strengths and the elastic modulus 13 of mortars were analyzed. The hydration kinetics of the mortars were studied at different temperatures. The acceleration of 14 the chemical reactions was also assessed through the determination of the apparent activation energy of mortars. Finally, 15 the microstructure of two formulations of mortars was observed by Scanning Electron Microscope (SEM). Results show a 16 drop of the mechanical properties with the increase of the percentage of substitution. This can be attributed to the natural 17 composition of sediments and to the increase of the total porosity of the mixtures. The measured heat released of mortars 18 increase with the increase of sediment content. This result suggests an additional chemical activity linked to the presence 19 of sediments within the matrix.

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

21 Introduction

The accumulation of sedimentary particles at the bottom and AQ1 23 on the edges of watercourses and shipping lanes as well as 24 in seaports leads to their clutter. For navigable waterways, 25 it slows the flow and prevents activities of transportation. In 26 order to maintain and restore these accesses, it is necessary 27 to dredge or clean up regularly from coast and estuarine lines 28 around the world.

AQ2 Each year, several millions of tons of materials are 30 dredged throughout the world [1]. In France, around 50 mil-31 lion m³ of sediments are produced from ports. These mate-32 rials, which range from rocks to clays, can contain various 33 types and amounts of pollutants and organic matter. 34

 \bowtie Amrouche Abdelwaheb A1

abdelwaheb.amrouche@univ-artois.fr A2

Junia, ULR 4515 - LGCgE, Laboratoire de Génie Civil A3 Et Géo-Environnement, Univ. Artois, Univ. Lille, Institut Α4 A5 Mines-Télécom, Technoparc Futura, F-62400 Béthune, France A6

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. AO3

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 th dredged material, including mineral comp distribution, and physical and chemical c Furthermore, there are a number of sci that must be overcome in order to achie application. Among these, inorganic cont heavy metals (copper, chromium, lead, e as organic hydrocarbons, such as PAHs (p

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

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

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

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

Most of these studies relies on the substitution of cement 90 by the sediment for the formulations of mortars and con-91 cretes [23–27]. However, few studies were conducted on the 92 replacement of sand by sediments in the cement-based mate-93 rials [18, 28, 29]. Most studies deal with a simple stabiliza-94 tion-solidification process [30-33]. Dredged sediments can 95 fully or partially substitute the sand aggregate [34, 35]. This 96 valorization is very interesting in order to solve a current 97 98 environmental and economic issue related to the pollution generated by cement industry, a major emitter of CO₂, and 99 the depletion of natural aggregates. The construction indus-100 101 try in France consumes 400 million tons of aggregates per year where 40% are used for concrete structures. 102

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

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

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

The main objective of this paper is the valorization of uncontaminated marine sediments, not subjected to any specific high-cost pre-treatments and the preservation of natural resources. Sediments are used to replace sand in the manufacture of mortars where five formulations were studied. This research was carried out to demonstrate the feasibility of substituting sediments by sand aggregate. These sediments were dredged from the port of Dunkirk, north of France. Physical, chemical, mineralogy, and mechanical characterizations of the dredged sediments were assessed in order to ensure a sustainable management of sediments and investigate their possible use and impact in cemented-based materials.

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

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

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

Materials and methods

Sediments characterization

The used marine sediments were dredged from the Grand 156 Port Maritime of Dunkirk (GPMD) located in the North of 157 France. This port dredges about 4 million cubic meters of
sediments per year. The characterization of such materials
is essential in order to understand their influence and behavior in cemented matrixes and explain some mechanical and
physico-chemical properties that can be altered.

Physical characterizations of sediments were performedand results are summarized in Table 1.

The particle size distribution was conducted by laser particle 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].

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

The mineralogical characteristics of the used sediments were also investigated using Siemens D5000 diffractometer (Fig. 2). The XRD analysis provides the presence of crystalline phases. The major elements detected were quartz (SiO₂) and calcite (CaCO₃).

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Table 1	Physical	characterization	of marine	sediments
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Density (g/cm ³)	Surface area BET (cm ² /g)	Organic mat- ter—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 si of sediment and s		100 90 90 0 0 0 0 0 0 0 0 0 0 0 0 0 0			1000		Sediment sand
	Y			Diamete	r (μm)		

	Table 2	Contaminant	concentrations	in	sediments	(mg/kg))
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Element	Antimony (Sb)	Arsenic (As) Barium (Ba) Cadmium (Cd)	Chromium	(Cr) Co	opper (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)	Molybde- num (Mo)	Nickel (Ni)	Selenium (Se)	Zinc (Zn)	Fluorides	Chlorie	des 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

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190 Formulations

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

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

Table 3 presents the composition of the various for-201 mulations studied in 1 L of volume. The effective water-202 to-cement (W/C) ratio was kept constant (0.5) for all for-203 mulations. The absorption coefficient of sediments was 204 determined according to standard NF EN 1097-6. A value 205 of 35% was obtained. The sediments were pre-saturated 206 207 for 2 h before mixing in order to prevent the main issue of porous aggregate linked to their high water absorption 208 capability. The formulations were prepared according to 209 the mixture procedure described in the European stand-210 ard EN 196-1 [37]. For each formulation, three prismatic 211 samples of $4 \times 4 \times 16$ cm³ were prepared and cured until 212 28 days. The slump of mortars was measured using the 213 Abrams' mini-cone (see Table 3). It can be seen that all 214 formulations have almost the same consistency. 215

Portland cement CEM I 52.5R (Calcia Cement from Couvrot) was used. The mortar aggregate was CEN siliceous sand (according to EN 196-1), with rounded grains (Class 0/5). This sand has a silica content of at least 98%. Its density is 2.60 g/cm³ measured by helium pycnometer and it has an absorption coefficient equal to 0.7%. The particle size distribution of sand is presented in Fig. 1.
 Table 3 Composition of the sediment-based mortars (in 1L)

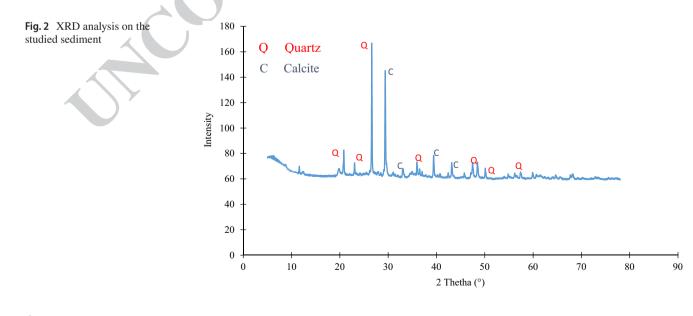
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Mortar	MT	MT5	MT15	MT30	MT40	
Cement (g)	507	507	507	507	507	
Sand (g)	1526	1450	1298	1068	916	
Effective Water (g)	253	253	253	253	253	
Sediment (g)	0	95	285	570	760	
W/C	0.5	0.5	0.5	0.5	0.5	
Density (g/cm ³)	2.11	2.09	2.07	2.04	1.98	
Slump (cm)	0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

Experiments

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

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

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



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

Heat of hydration 249

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

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

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

$$K(T) = A \exp\left(-\frac{E_{a}}{RT}\right),$$
(1)

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 A:04 metric study. The apparent activation energy must be calculated 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

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

$$t_{\rm eq} = \int_{0}^{t} \exp\left(-\frac{E_{\rm a}}{R}\left(\frac{1}{T(\tau)} - \frac{1}{T_{\rm ref}}\right)\right) \mathrm{d}\tau, \tag{2}$$

where $T(\tau)$ is the absolute temperature at the moment τ (°Kelvin), R the perfect gas constant = 8.314 J/mol K, 299 $T_{\rm ref}$ the reference temperature, $E_{\rm a}$ 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_{eali}, t_{ea2i}) over the tar-304 geted 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

$$\min_{E_{a}} \left(\sum_{X_{i} \in [X_{inf} - X_{sup}]} \left| t_{eq \ 1i} - t_{eq \ 2i} \right|^{2} \right), \tag{3}$$

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

Thermal conductivity and specific heat capacity

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

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$$\lambda = \frac{\sum \phi \times e}{2 \times \Delta T},\tag{4}$$

with λ : thermal conductivity of the material (W. m⁻¹. K⁻¹), e: sample thickness (m), $\Sigma \varphi$: sum of both recorded heat flux at the sample surface (W.m⁻²), and ΔT : temperature gradient (°C).

The specific heat capacity is also an important parameter 342 for construction materials. It depends on the density and the 343 nature of the material in study. It characterizes the ability of 344 a material to store heat and represents the amount of heat per 345 mass unit required to raise the temperature by one degree. 346 To determine the thermal capacity of a material, the method 347 is based on the process of heat storage of the material. In 348 the initial state, the system is isothermal: the exchange 349 plates are at temperature (T_{init}) . The final system is also in 350 an isothermal state (T_{fin}) . The sample stores an amount of 351 energy that represents the system's internal energy variation 352 between these two states (Q). The material's specific heat 353 $(C_{\rm p})$ can then be calculated using Eq. (5). A higher specific 354 heat capacity value means that the material can store more 355 heat and contribute more effectively to building insulation. 356

$$C_{\rm p} = \frac{Q}{\rho \times A \times e \times \Delta T},\tag{5}$$

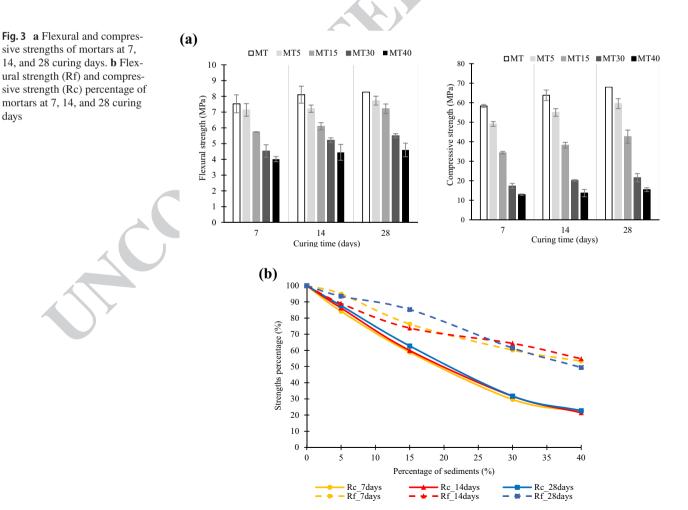
with C_p : specific heat capacity (J. K⁻¹. kg⁻¹), *e*: sample 359 thickness (m), ρ : density (kg.m⁻³), *A*: surface of the material 360 in contact with the exchange plates, *Q*: stored energy (J), and ΔT : temperature gradient (°C). 362

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. 370



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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.

383 Elastic modulus

The average elastic modulus of three measurements for each 384 formulation of sediment-based mortars are presented in 385 Fig. 4, for 7, 14, and 28 curing days. The errors on the meas-386 urements are less than 5%. The elastic modulus E decreases 387 when the percentage of replacement of sand by sediment 388 increase independently of the curing time. The mortars 389 became more deformable with the increase of substitution 390 percentage. The elastic modulus of the reference mortar is 391 equal to 35000 MPa, while the sediment-based mortar MT40 392 has a value around 8000 MPa at 28 days. 393

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 discussed in Interpretations and discussion.

405 Mercury porosity

The porosity of a cement matrix is a fundamental measure-406 ment for characterizing the microstructure and determin-407 ing the material's durability. This test yields a global result 408 ("total porosity"), which is an indicator of the quality of the 409 material [42]. It evaluates the percentage of connected voids. 410 The test results at 28 curing days are given in Table 4. The 411 results confirm that the presence of sediments modifies the 412 pore structure. The average pore diameter is smaller for mor-413 tars having high percentage of sediments. However, the total 414 intrusion volume and total pore area are higher for MT40 415 than for mortars with low percentage of replacement. This 416 result can be refined by presenting the pore size distribu-417 tion of mortars in Fig. 5. The mortars with high percentage 418 of sediments (MT30 and MT40) have higher proportion of 419 small pores but lower large pores. This highlights the porous 420 nature of the used marine sediments. The total porosity of 421

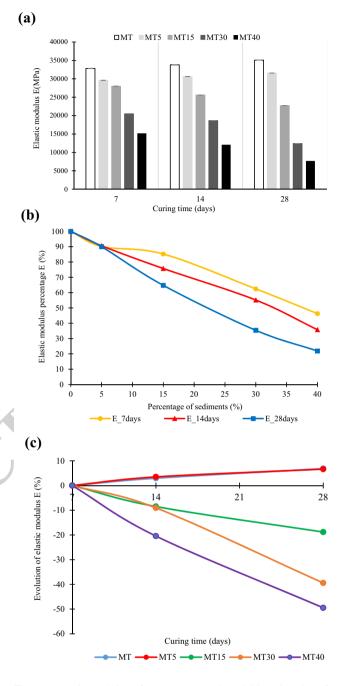


Fig. 4 a Elastic modulus of mortars at 7, 14, and 28 curing days. **b** Percentage of elastic modulus of mortars at 7, 14, and 28 curing days. **c** Evolution in time of the elastic modulus (%) of mortars

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. 424

Heat of hydration measurements

The results of the hydration kinetics observed on the five 426 formulations of mortars are presented in this section. The 427

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Table 4Mortars porosity usinga mercury porosimetry analysisat 28 curing days

Formulations	MT	MT5	MT15	MT30	MT40
Average pore diameter (4 V/A) (µm)	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 (m ² /g)	3.41	4.63	9.05	17.42	19.75
Porosity (%)	9.92	9.85	11.95	13.45	16.56

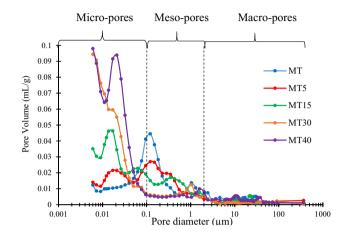


Fig. 5 Pore size distribution of mortars measured by MIP at 28 days

428 total flux and the heat of hydration Q (J/g) of the formulated mortars at the three imposed temperatures 20, 30, and 40 °C 429 are presented in Fig. 6a-f, respectively. The heat of hydra-430 tion Q(J/g) can be determined by an integration of the heat 431 flux. The start time of the hydration reaction is calculated 432 for each mortar at 20 °C and 30 °C (Table 5). It represents 433 the end of the "latent period" where the flux changes are 434 very small and the beginning of the "acceleration period" 435 where the flux starts to evolve. It is difficult to determine 436 such period for the formulated mortars at 40 °C due to the 437 accelerating effect of temperature on the hydration reac-438 tions of cement as it is clearly shown in Fig. 6a-c. Thus, 439 the latent period is very short at 40 °C compared to 20 °C. 440 It can be noticed a delay when the percentage of sediments 441 increase for both temperatures. At 20 °C, the hydration reac-442 tion started after 163 min for the reference mortar MT while 443 after 200 min for the sediment-based mortar MT40. Nearly 444 the same delay effect was observed at 30 °C, the reactions 445 begins after 54 min for MT while 96 min for MT40. 446

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.

454 Comparing the heat curves of the formulated mortars, it 455 is apparent that the heat released of mortars increases with

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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 phe-463 nomena, a parameter was determined which refers to the 464 apparent activation energy E_a described previously in 465 Results. In this paper, a superposition method for determin-466 ing this parameter for the formulated mortars is presented. 467 This method is based on the evolution curves of the heat 468 released which define the maturity of the hydration reaction 469 of cement. 470

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.

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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. 481

The evolution of the apparent activation energy is high-482 lighted in Fig. 7. A quasi-linear evolution of E_a is observed 483 as function of sediment percentage for the three pairs of 484 temperature. This evolution is small up to 5% of substitu-485 tion. Therefore, the hydration reaction is little affected by 486 this introduction of sediments. However, the evolution of 487 E_{a} is much more important for MT15, MT30, and MT40. E_{a} 488 increases around 9%, 15%, and 18%, respectively, for each 489 pair of temperature. One can say that the effect of sediments 490 is not modified with the increase of temperature. 491

Thermal conductivity and specific heat capacity

Figure 8 presents the evolution of the thermal conductivity493and the specific heat capacity of the reference mortar (MT)494and sediment-based mortars.495

The thermal conductivity of (MT) is 1.25 W.m^{-1} . K⁻¹ in 496 conformity with the values obtained in literature [44–46], 497 while it decreases to 0.66 W.m⁻¹. K⁻¹ for MT40. This 498

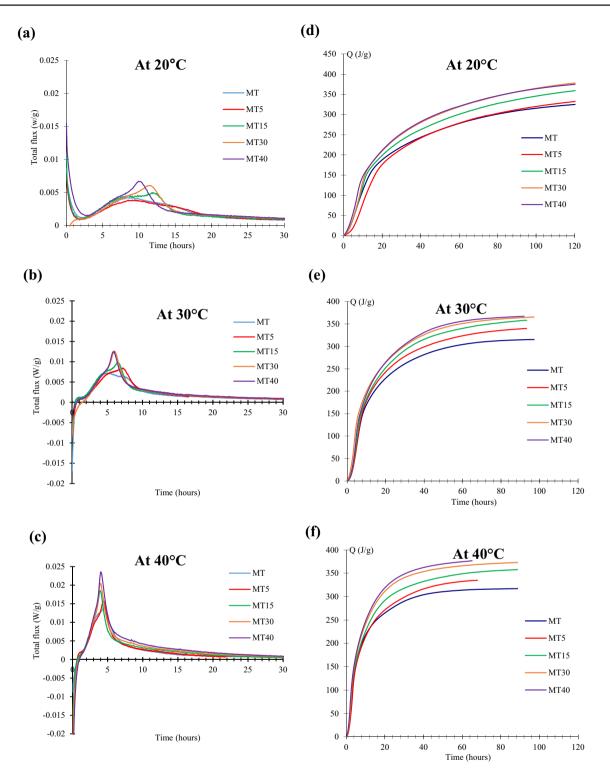


Fig. 6 Total flux (a,b,c) and heat of hydration Q(J/g) (d,e,f) of the formulated mortars at imposed temperatures 20°, 30° and 40 °C

decrease is linear with the percentage of sediments incorporated in the formulated mortars. It ranges from 10 to 50%
for MT5 and MT40, respectively.

Contrary to the thermal conductivity, the specific heat502capacity increase in the presence of sediments. C_p increases503from 821 J/kg. K for MT to 1247 J/kg. K for MT40. An504increase of 50% was observed for 40% of sand replacement.505

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 tem- peratures (°C)	МТ	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

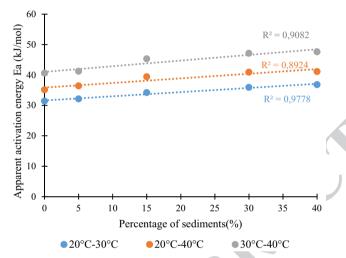


Fig. 7 Evolution of the apparent activation energy of mortars

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Microstructural properties: scanning electron microscopy observations.

The microstructures of the reference mortar MT and the
sediment-based mortar MT30 were observed by Scanning
Electron Microscope (SEM) with back-scattered electron
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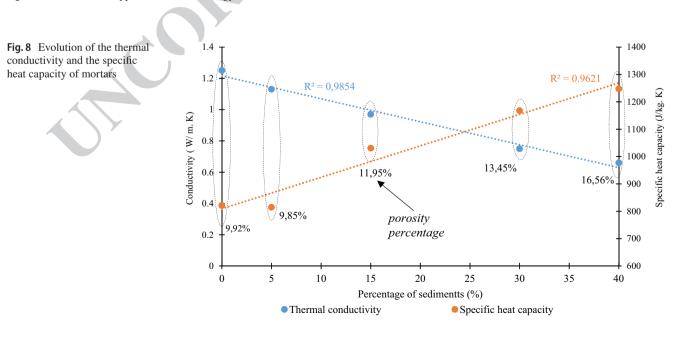
After 28 days curing in water, pieces of 1cm³ were cut from the core of the mortar samples. All samples were epoxy impregnated and polished according to a standardized process. 512

Figure 9 displays the textures and cementitious phases 516 microstructures obtained for the studied mortars MT and 517 MT30. 518

From a qualitative point of view, no major differences 519 in terms of cement matrixes can be observed between 520 the reference and the sediment-based mortar in the SEM 521 images. Crystalline portlandite, needle-shaped ettringite 522 and amorphous calcium–silicate–hydrates (C-S-H) can be 523 clearly recognized. Pores of various sizes can also be seen 524 among the solids. 525

However, C-S-H gel was found in greater amounts in AQ5 6 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.

Diverse CH crystallization was observed in the sediment-based mortar MT30, with thin hexagonal and globular-shaped portlandite crystals (alite and belite). 536



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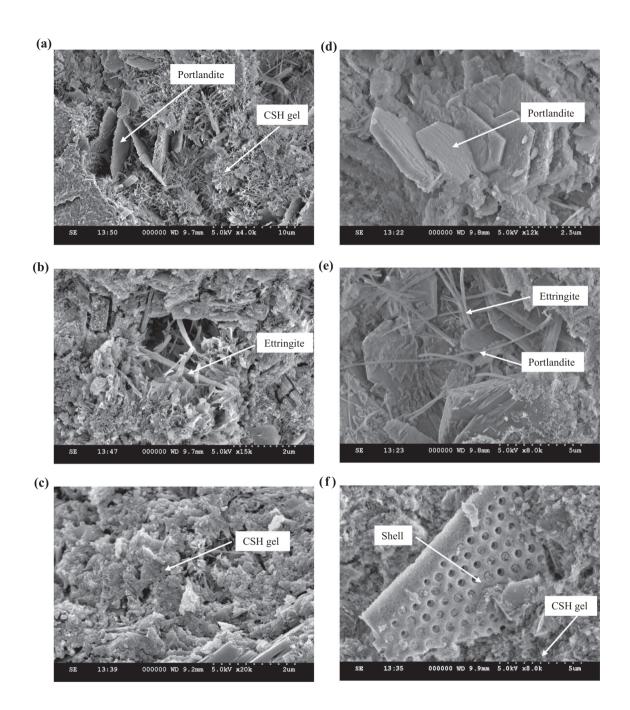


Fig. 9 SEM with back-scattered electron pictures of MT (a, b, c) and MT 30 (d, e, f) samples at 28 days of curing time

537 Interpretations and discussion

The feasibility of the uncontaminated marine sediments used in the formulation of mortars as a replacement of sand aggregate was assessed.

The mechanical strength is an important characteristic 541 that has to be determined for the purposes of valorization. 542

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543 It allows the evaluation of the material's physical integrity 544 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 554 (Table 2). Their high salinity has a negative impact on the 555 mechanical strength development of cement-based materi-556 als. The influence of salts was the subject of a previous study 557 conducted by Kamali et al.[13]. The authors have found a 558 low mechanical performance of mortars mixtures made with 559 unwashed sand. They demonstrated that the effect of chlo-560 ride contained in sediments can be reduced by a washing 561 process. 562

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), Cad-566 mium (Cd), or Chromium (Cr), may alter the hydration 567 processes and have a negative impact on the mechanical 568 strength by affecting the mortar's average porosity accord-569 ing to Arliguie and Grandet [49]. Zinc presents the largest 570 impact out of all the other pollutants. Its presence causes a 571 significant reduction in the compressive strength by affecting 572 the hydration of C₃S and C₃A [55, 56]. Bhatty [52] previ-573 ously investigated the impact of all trace elements in cement 574 production and consumption. The mechanisms that normally 575 lead to the development of hydrates (CSH) or the pores net-576 work can be disturbed [51, 53, 54]. 577

Same trend was observed for the elastic modulus which 578 decreases in the presence of sediments (Fig. 4). It is known 579 that the elastic modulus is highly influenced by the aggre-580 gates modulus that compose the cemented materials as well 581 as by their form and texture [55]. The decrease with the sedi-582 ment substitution can be explained by the means of two fac-583 tors. Sediments are known to have low resistance compared 584 to natural aggregates like sand. In addition, sediments are 585 high porous by nature. This high internal porosity generates 586 an increase of the mortars porosity as it is given in Table 4 587 where the sediment-based mortar MT40 has the highest total 588 porosity (16.56%). 589

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 596 and a progressive decalcification in time of the CSH leading 597 to an increase in their porosity [56, 57]. These phenomena 598 cause internal cracking, which explains the decrease of the 599 elastic modulus as a function of time, particularly for mor-600 tars MT30 and MT40 [58–60]. The elastic modulus is more 601 sensitive to these variations compared to the resistances. For 602 the latter, these effects are masked due the continuation of 603 hydration and the increase of the bonds between the CSH. 604 It is noticed that we have almost no evolution of resistances 605 for the mortars with high substitution rates of sediments. 606

The micro-porosity of the sediments can be observed 607 when comparing the distribution of porosity (Fig. 5). The 608 pore structure can be divided into three main ranges as it 609 was reported in Cheng et al. [61]. The micro-pores which 610 correspond to the pore diameter $< 0.1 \,\mu\text{m}$, the meso-pores 611 for pore diameter ranging between 0.1 µm and 2 µm, and 612 the macro-pores for pore diameter > 2 μ m. The mortars with 613 high percentage of replacement have a porosity distribution 614 ranged in the first domain representing micro-pores. This 615 can be explained by the internal porosity of the used marine 616 sediments. 617

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.

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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 631 in porosity ratio of 1% can result in a 10% loss in elastic 632 modulus. Same result was obtained in this study where the 633 elastic modulus of MT40 decreases by 80% when its poros-634 ity increases by around 8%. The resistance of mortars is 635 primarily a consequence of their porosity. Up to 30% of sand 636 replacement by sediment, the decrease of the mechanical 637 properties is significant. Between 30 and 40%, this effect 638 is reduced. Similar results were obtained by Couvidat et al. 639 [18], who fully replaced sand by sediments. The authors 640 obtained a linear correlation between the increase in total 641 pore area and the decrease in compressive strength. 642

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 µm and mineralogical characteristics of 648

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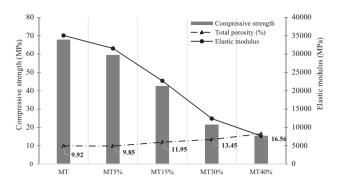


Fig. 10 Compressive strength of mortars linked to the total porosity and elastic modulus at 28 days of curing time

sediments revealed that the major elements are calcite and 649 quartz. Regarding these findings, it seems that sediments 650 are not totally considered as inert admixture. However, 651 they have a certain chemical activity linked to their gran-652 ulometry and mineralogical composition. They can par-653 ticipate in the exothermic reactions of hydration. It seems 654 also that the quality of the bonds between the hydrates 655 656 formed in the presence of sediments are altered despite the increase of heat released. This assumption is confirmed by 657 the degraded properties determined previously. 658

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 for-664 mulated mortars. An increase was observed when the 665 percentage of replacement of sand by sediments increase. 666 This can be explained by the fact that higher energy is 667 necessary to activate the hydration reaction of cement in 668 the presence of the used sediments. This result confirm the 669 decrease of the mechanical strengths observed previously. 670 Bresson [64] have found a correlation between the activa-671 tion energy of a cement and its actual class at two days. 672 Waller [65] compared values of the activation energy for 673 674 different types of cement and demonstrated that the activation energy is inversely proportional to the mechanical 675 strength. 676

The high porosity observed in the sediment-based 677 mortars has also an influence on the thermal conductiv-678 ity and the specific heat capacity (Fig. 8). The available 679 voids inside the cement-based materials and the sediment's 680 internal porosity have a significant effect on the thermal 681 conductivity due to the low thermal conductivity of air 682 683 [66]. In addition, the rise of the specific heat capacity when substituting sand by sediment can be explained by 684 the decrease of the density of mortars as shown previously 685 in Table 3 [67, 68]. 686

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 ($70\% < 63 \mu m$). They were classified as uncontaminated sediments containing high amount of salts. They were mainly composed of quartz (SiO₂) and calcite (CaCO₃). 698
- 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.
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- The total porosity increased as the sediment substitu-703 tion content increase, but the pore size distribution was 704 shifted toward smaller particles. This highlights the 705 porous nature of the used marine sediments. MT is the 706 least porous mixture (9.92%). The increase in total poros-707 ity is linked to the decrease of the mechanical strengths. 708 The total flux and heat released measured were higher 709 in the presence of sediments. A delay of the exothermic 710 hydration reactions was also observed. Sediments had 711 a certain chemical activity linked to their granulometry 712 and mineralogical compositions. 713
- A linear evolution of the apparent activation energy E_a 714 was observed as function of sediments percentage. An 715 increase of 18% was observed for MT40. Higher energy 716 was necessary to activate the hydration reactions in the 717 presence of sediments which confirm the decrease of 718 the mechanical strengths. 719
- The thermal conductivity λ of mortars decreased, while 720 the specific heat capacity Cp increased with the per-721 centage of sand replacement. An evolution of 50% was 722 observed for MT40. This can be attributed to the high 723 porosity and low density of the sediment-based mor-724 tars. MT is the mixture having the highest density cor-725 related to the lowest value of Cp and highest value of λ . 726 The higher the C_p value, the more the material retains 727 the stored heat and contributes to the insulation of the 728 construction. 729
- 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 735

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

One can mention that the origin of sediments (marine or fluvial) could influence the properties of recovered materials especially concerning the salinity in presence. Supplementary experiments would be needed to assess the durability of mortars.

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748 **Declarations**

749 Conflict of interest The authors declare that they have no known com-750 peting financial interests or personal relationships that could have ap-

peared to influence the work reported in this paper.

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