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About a possible valorisation in cementitious materials of polluted sediments after treatment

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Abstract

The disposal of contaminated dredged material has become an economical and environmental issue. Firstly, this study presents the Novosol® process which was used for the treatment of polluted sediments. This process is based on the stabilisation of heavy metals and on the thermal elimination of organic matter. A physical characterization of the processed material reveals high porosity (60%) and water absorption (45%). Moreover, the treated sediment aggregate (TSA) has a relatively low strength and a high content of fine particles.

Secondly, an experimental study on the feasibility of TSA introduction in cement-based materials was performed. Three mortars for which a given sand volume was replaced by the same sediment volume (33%, 66% and 100%) were designed. A strong increase of drying shrinkage was observed (up to 10 times higher, when compared to a reference mortar). Permeability remained virtually constant, though. There was a significant increase in strength for low to moderate substitution, while high incorporated quantity of sediment led to a strength on the same order of that of the reference mortar.

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1. Introduction

Sediments, which result from natural processes, are an essential, integral and dynamic part of river basins, including estuaries and coastal zones. Generally, sediments come from the weathering and erosion of minerals, organic material and soils in upstream areas and from the erosion of riverbanks. They are susceptible to being transported downstream by surface water [1]. Deforestation and urbanisation tend to facilitate the washing of superficial soils by rain which increases the amount of particles down the bottom of rivers, channels and harbours. Nowadays sediments have become an economic and environmental issue. Due to human industrial activities, an important part of sediments is actually polluted. Contamination is mainly due to inorganic and organic pollutants. Among organic ones, PAHs (polycyclic aromatic hydrocarbons), PCBs (polychlorinated biphenyls), TBT (tributyltin) and dioxins are the most toxic. Heavy metals such as lead (Pb), chromium (Cr), mercury (Hg), zinc (Zn), cadmium (Cd), copper (Cu), are considered the worst inorganic pollutants.

Dredging sediments is essential to maintain navigation in ports, harbours, marinas and inland waterways. Dredging operation impact and the effect of dredged material disposal in open water have been largely investigated [2,3]. These operations are a huge threat, not only for the marine flora and fauna, but also for human beings.

France, as other countries, has to deal with sediments issue. Every year, 50 million m^3 are dredged into the five huge maritime port authorities and into the seventeen commercial ports [4], and a high amount of these sediments is polluted. Dredged material has to be managed within international rules (OSPAR Convention [5], London Convention [6], Barcelona Convention [7]). A French order dated June, 14th 2000 [8], details the chemical threshold values that dredged material must respect before dumping and

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the protocol to sediment sampling in ports depending on size, gain nature and volume.

Various alternative ways to the disposal of dredged material and beneficial reuses [9] have been investigated. Among the most common answers are landfilling, use of confined disposal facilities and capping. Those techniques do not really bring a definitive solution. Indeed, landfilling of contaminated materials and confined disposal facilities require space and long term monitoring; and capping does not apply for maintenance and engineering dredging, i.e., dredging operations which are aimed at creating or enlarging existing channels or basins. Several existing treatment methods may be mentioned: thermal treatment, bio-remediation, solidification/stabilisation by hydraulic binders, washing. Nevertheless, due to high costs, treatment is seldom used. In France, less than 100 000 m^3 are treated each year [10]. The process used for this study is the Novosol® process patented by Solvay Company [11], which consists in a chemical inertization of heavy metals followed by a thermal elimination of organic pollutants. In order to make this solution economically and environmentally sustainable it is necessary to find beneficial reuse ways to avoid overcost and useless discharge disposal. Hence, a large experimental study, focused on the valorisation of treated sediments in the Civil Engineering area has been performed in our laboratory.

This paper aims, in its first part, at presenting the Novosol® process and a physical and mineralogical characterization of treated sediments is detailed. In a second part a valorisation feasibility study on cement-based materials is presented. Finally, the results are discussed.

2. The treatment of polluted sediments

2.1. Raw polluted sediments

The sediments used in this study were dredged in the harbour of Dunkirk Authority in the North of France. Pollution is mainly located on the finest particles [12], which removal is very difficult. The sediments studied here were dredged in a basin where damaged ships are repaired. The low sediments granulometry (see Table 1) and the industrial activity in the sampling area explain the high amount of pollutant they contain (see Table 2). In France, observation workgroup on dredging and environment named GEODE has induced a French inter-ministerial decree edited on June 14th, 2000, on reference levels to consider when analysing marine or estuarine sediments in the natural or port environments [8]. Two thresholds for heavy metal and PCBs content of dredged marine sediments were defined. As specified in the memorandum on the conditions of application of this decree and cited by Alzieu [13]: "*These thresholds constitute benchmarks designed to improve assessments of the potential impacts of a planned operation (as open water relocation). Below level N1, the potential impact is regarded, in principle, as neutral or negligible, with "normal" concentrations or comparable to environmental background. Between action levels N1 and N2, further investigations may prove necessary depending on the project considered and on the extent that action level N1 is exceeded. Beyond an N2 level, additional investigation is generally necessary since significant indices suggest a potential harmful impact from the operations*". Table 2 underlines the high amount of pollutant in our sediments and demonstrates the necessity to isolate this material from the environment or to treat it before a possible valorisation. Several sources of harbour contamination may exist. Antifouling paints represent a significant source of copper introduction into port environment since CuO_2 is an active ingredient. These inputs have increased since the early 1980s, due to the ban on tributyltin in antifouling paints. Zinc is introduced into port environments by the dissolution of pure zinc bound to anti-corrosion substances on ship hulls. Furthermore, those paints also contain large amounts of zinc oxide as anti-corrosive additive [34].

Table 1
Mineralogical composition of raw sediments

Particle type (size)	Mass percentage
Clay (<2 μm)	5
Silt (from 2 to 63 μm)	59
Sand (>63 μm)	36

Table 2
Heavy metals content of raw sediment and threshold value

Heavy metal	Content (mg/kg of dry material)	Geode threshold	
		N1 level	N2 level
Cadmium (Cd)	1.9	1.2	2.4
Chromium (Cr)	110	90	180
Copper (Cu)	639	45	90
Lead (Pb)	256	100	200
Zinc (Zn)	785	276	552

2.2. The Novosol® process

The Novosol® process is based on the ability of a natural mineral to fix heavy metals and to make them unleachable. This mineral is the hydroxyapatite, which chemical formula is $Ca_5(PO_4)_3OH$. It reacts with a large variety of pollutants such as Pb, Cd, Cu, Zn, Ni, As, U, F, Br, Cl, which leads to different compounds such as $(Ca,Pb)_5(PO_4)_3OH$, $Pb_5(PO_4)_3Br$, $Zn_5(PO_4)_3F$, $Cu_5(PO_4)_3OH$, $Cd_5(PO_4)_3OH$, $Ca_5(AsO_4)_3OH$. This stabilization mechanism has already been widely studied and approved [14,15]. Our knowledge of involved fixation mechanisms lies on the experience of various waste treatment processes, like municipal solid waste incineration fly ash or sediments, based on phosphatation and experienced by Solvay through its Revasol and Novosol processes. These experiments show that the major produced crystal phases are not pure hydroxyapatite but various types of calcium phosphates and metallic phosphates [16,17,35–37]. Their equilibrium under various conditions, such as temperature or pH, has also been studied.

The process consists of two phases:

- The phosphatation phase: dredged sediments containing 50% water are pumped and mixed with 3% phosphoric acid (H_3PO_4). Raw sediments containing Ca^{2+}, their chemical reaction with H_3PO_4 lead to the formation of apatite-like minerals, which fix heavy metals. This reaction is followed by the formation of foam [38]. After mixing sediments with H_3PO_4, the phosphatized sediment is put in a drying bed in order to reach a given saturation level. Foam allows an increase in drying rate [38].
- The phosphatized sediment is heated in a rotary kiln at 650 °C, in order to eliminate all organic compounds. This phase also increases the amount of heavy metals fixed in the apatite mineral.

The efficiency of the Novosol® treatment has been assessed by leachability tests, with French XP 31-210 [18] and American TCLP [19] standards. Since the treatment process consists in reducing the mobility of pollutants instead of eliminating them, the total content in inorganic pollutants remains the same before and after treatment, while the amount of leachable pollutants is reduced [36,37,39].

2.3. Methodology

Treated sediment aggregates are not acceptable as concrete aggregates according to French standards [20].

Depletion of best quality materials, need for resource conservation and lengthened transport distances have all increased the need to introduce substitute materials to natural aggregates. On another hand, building and other industries produce large amounts of secondary products, which may be suitable for reuse [21]. This underlines the necessity to study the use of substitution raw material.

As regards the making up of cement-based material with large proportions of treated sediments substituted to aggregates, two main questions arise. Is there a practical feasibility (concerning cement setting) in introducing a material which preliminary treatment has been chemical? What would be the main characteristics and mechanical properties of such a new material? Answering these questions is crucial if industrial use is expected, even as a poor concrete.

Hence the experimental study presented in this paper has been set up to study the feasibility of sediment use in cement-based material.

3. Experimental

3.1. Characterization of treated sediment

After treatment, visual inspection shows that the inertized sediments are a brown, light and porous aggregate (see Fig. 1). Their cohesion is very low: they are easily crushed between two fingers. They are covered with fine particles. In fact, from millimetric to centrimetric size, the aggregates appear to be made up from the agglomeration of fine particles. They may have been formed during the beginning of a sintering phase during calcination. Fine particles were studied more thoroughly through SEM micrographs. As Fig. 2 shows, they have various shapes and dimensions, and they are highly porous. Further visual observation does not show any significant difference between river and harbour sediments. Micrographs c, d and e taken from crushed harbour aggregates show the same type of structure at the microscopic level as at the macroscopic one. The smallest aggregates are also made up from the agglomeration of fine particles. This type of structure leads to a high internal porosity.

Fig. 1. Macroscopic aspect of harbour sediments treated by the Novosol® process.

The fine particles size distribution has been studied with a laser granulometer (Coulter LS 230 counter, Small volume module). Samples were immersed in a mix of water and sodium hexametaphosphate. Three different types of treated harbour sediments were tested: one from Livorno (Italy) and two from Dunkirk (France). The curves are similar, quite narrow, with a mean value around 10 μm (Fig. 3).

As intra-granular porosity usually governs the water demand during mixing to obtain cementitious material, this parameter was studied with a BET device using nitrogen. Fig. 4 shows the isotherm adsorption and desorption curves of fine particles of treated sediments in which can be observed a slight hysteresis. IUPAC (International Union of Pure and Applied Chemistry) has proposed to interpret these curves according to the shape of the hysteresis [40]. The shape observed in Fig. 4 corresponds to agglomerates with an undefined meso-porosity (between 0.1 and 0.6 μm). This confirms the SEM observations (Fig. 2). The BET surface area of those particles is 6 m^2/g on average which is about 10 times that of cement.

The physical characterisation of fine particles of TSA revealed typical properties of this new material. The

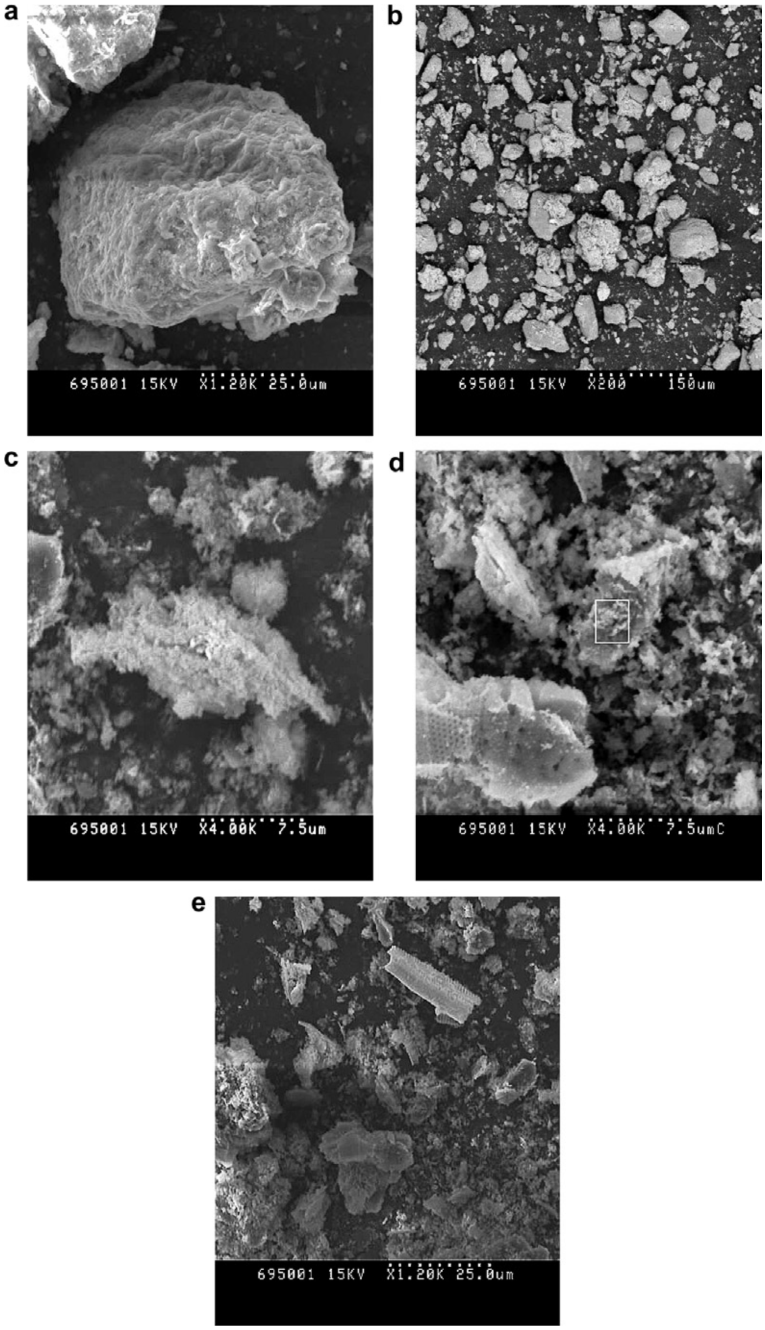

Fig. 2. SEM micrographs of treated river sediment fines (<80 μm) (a and b) and crushed treated harbour sediments (c, d and e).

average diameter is close to 10 μm. Fine particles form a highly porous agglomerate which can be easily crushed. The main consequence of this porosity is the high specific area. Fine particles use in cementitious material would lead to an increased water demand and a lowered workability.

At a macroscopic level, a second type of internal porosity is observed. It is attributed to the foaming which occurs during the phosphatation phase of the treatment. Table 3 summarizes the main macroscopic characteristics of treated sediments. It is observed that treated sediments have a high porosity and a high water demand which increase slightly with the size of the aggregate. This is directly linked with a low volumetric mass. Porosity was measured with demineralised water after saturation under vacuum, and volumetric mass was measured with a pycnometer. Due to the aggregate brittleness and its fine particles coating, an accurate measurement of the aggregate mass is a hard task, especially at the “saturated dry surface” state.

3.2. Mortar design and curing conditions

Four different mortars were designed for this study:

- a reference mortar (“MR”) with classical mixing proportions (see Table 4 for the design of studied mortars),

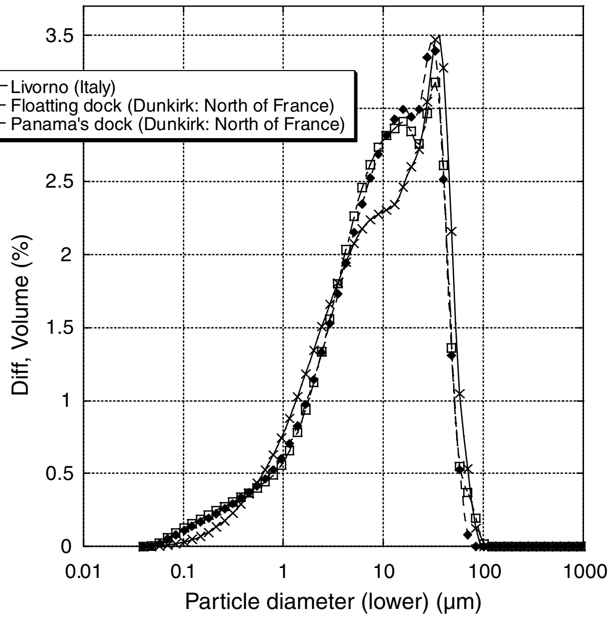

Fig. 3. Laser granulometry of three different treated harbour sediments.

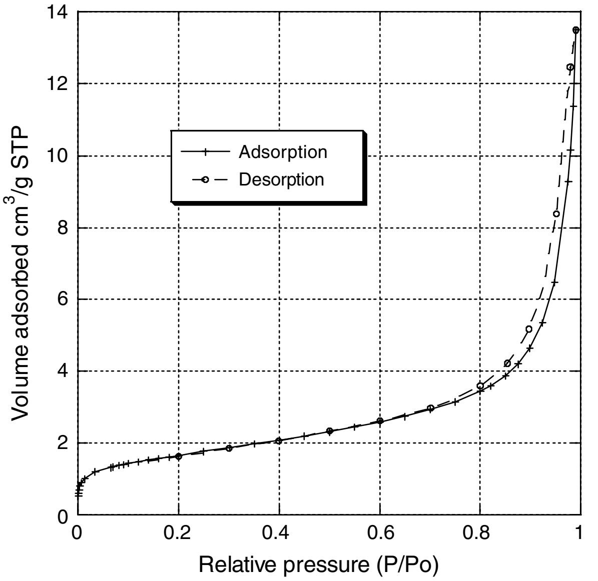

Fig. 4. Isotherm plot of a BET study on treated harbour sediments from Dunkirk.

Table 3
Macroscopic characterization of treated harbour sediments

	Porosity (%)	Water absorption after 24 h (%)	Bulk density (g/cm^3)
Sand fraction (<2.5 mm)	54	41	1.3
Gravel fraction (2.5–8 mm)	66	53	1.1

- three mortars called MS33, MS66 and MS100 for which a given sand volume was replaced by the same sediment volume: respectively 33%, 66% and 100%.

In order to reduce the number of unknowns, the granulometric curve of treated sediments has been artificially designed to match that of the substituted classical sand. Nevertheless some parameters are less known or uncontrollable such as the geometrical shape of aggregates, the texture, the mineralogical composition, the porosity, ...

After casting, samples were protected from dessication for 24 h. Samples were then divided into two batches and submitted to two different curing conditions: under controlled atmosphere (20 °C and 60% relative humidity), and in water at 20 °C.

As described in many studies [22–25], the main issue of porous aggregates is their water absorption capability which influences both the workability during casting and the material properties. A part of the free water necessary for cement hydration is absorbed by aggregates which affects the mix plasticity. Hence aggregates were pre-wetted for 24 h before mixing. This led to an aggregate absorption of about 45% water in mass. Due to the quantity of water added and the uncertainties on the amount of water released during mixing, we chose to work with a constant slump instead of a constant water/cement ratio (W/C). Therefore, water was incorporated into the mix until it reached the same workability as the reference mortar. The amount of water added to the mix is given in Table 4. Slump was measured according to the European standard EN 12350-2 [41] designed for concrete slump measurement, in the absence of a well adjusted test for mortars in the laboratory. The slump results suggest that a part of the pre-saturation water is released during the mixing.

3.3. Sample preparation and experimental testing

Twenty-six liters of each formulation were mixed and casted. For each of these formulations several parameters and properties were measured:

- Length variation of prismatic samples (4 × 4 × 16 cm) equipped with brass studs. Variations were monitored for 120 days with a mechanical retractometer. For each curing two samples conditions were monitored to evaluate shrinkage.
- Compression strength and Young's modulus: uniaxial compression strength tests were performed using a mechanical Instron® testing machine. A prescribed displacement mode was used, the platen displacement being of 2 μm s^{-1}. The axial stress was deduced from the machine load measurement cell (300 kN capacity). To carry out a perfect stress state (without significant bending effects), an original hinged loading platen was designed and placed between the superior machine platen and the sample [45]. Imperfections due to a possible parallelism defect between the two sample surfaces were therefore minimised.
- Gas permeability was measured under steady flow rate conditions using pure Argon gas. Technical devices

Table 4
Experimental composition and characteristics of mortars

	Cement CEM II 32.5 R (kg)	Classical Seine sand (kg)	Treated sediment sand (kg)	Pre-saturation water (kg)	Water added to the mix (kg)	Slump (cm)
MR	11.7	35.1	–	0	5.85	8
MS33	11.7	23.37	5.85	2.63	5.41	9
MS66	11.7	11.7	11.7	5.27	5.15	8.5
MS100	11.7	–	17.55	7.90	5.59	9

and full test analyses can be found in previous studies conducted in our laboratory [26].

- Porosity, volumetric mass, water absorption measured as detailed in [42,43].

Compression strength, Young's modulus, gas permeability, porosity and volumetric mass were all measured on cylindrical samples of 36 mm in diameter and 72 mm in length after 28, 60 and 90 days of curing. Results are averages on three samples for each condition.

4. Results and discussion

4.1. Physical properties of hardened mortar

The main physical properties of the four mortars are indicated in Table 5. They were obtained after 28 days of water curing. Porosity and bulk density undergo a variation almost linear with the substitution ratio. Porosity of reference mortar averages 20% while that of substituted mortars reach 29%, 39% and 50% for 33%, 66% and 100% of substitution respectively. At the same time, volumetric mass is reduced: the volumetric mass of mortars equals 1.95, 1.84, 1.62 and 1.38 g/cm^3 for 0%, 33%, 66% and 100% of substitution respectively. This trend may be due to the characteristics of treated sediments themselves (cf. Table 3). Indeed the increase of porosity and the loss of bulk density are mainly attributed to the internal porosity of TSAs.

A high porosity may be considered a low durability indicator, since it facilitates aggressive species (CO_2, chlorides...) penetration in the matrix. Nevertheless, studies on lightweight concrete durability have shown that the use of lightweight aggregates (very similar to TSAs as regards their high porosity and low bulk density) does not lead to significant effects on durability, except for a saturated material subjected to freeze–thawing cycles [27]. The concrete permeability is governed by that of the matrix, the impact of the porosity of the aggregates not being of great consequence. The low permeability of lightweight concrete is explained by several factors:

- The low W/C of the paste.
- The good quality of the interface transition zone prevents flowing around the aggregates.
- The compatibility of the paste and the aggregate elastic modulus which reduces micro-cracking.
- The water tank role of lightweight aggregates allows the continuation of the hydration process and a reduction of the permeability [27].

Table 5
Physical properties of hardened mortar for water cured samples after 28 days

	Open porosity (%)	Bulk density (g/cm^3)	Intrinsic permeability (10^{-17} m^2)
MR	20.15–20.53	1.98–1.99	0.78
MS33	28.49–28.80	1.84	0.81
MS66	39.81–39.93	1.60–1.61	2.44
MS100	51.22–51.76	1.33	2.78

Permeability tests with Argon gas have been performed on the four mortar formulations. Intrinsic permeability values are given in Table 5. The MS33 permeability is very close to the reference mortar. This confirms the minor role of the aggregate porosity on the global mortar permeability. For higher substitution ratios, permeability values are still comparable with classical mortars even if slightly higher than that of the reference mortar. Low permeability is an indicator of quality and durability of a material. Indeed, every degradation mechanism, even internal, is submitted to transport processes [28]. Substituted mortars can therefore be considered potentially durable materials.

Due to the high total water content of these mortars, particular interest was given to the monitoring of drying shrinkage. Evolution of the latter is given in Fig. 5. Measurements were performed on $4 \times 4 \times 16$ cm^3 prismatic samples. Four samples of each formulation were monitored. Samples were protected from dessication with a plastic layer during the first 24 h after casting to avoid excessive plastic shrinkage which could not be measured. The length of samples aged 24 h was considered the initial length.

Results show that introducing pre-saturated TSAs leads to a strong increase of drying shrinkage. At the age of 110 days, the deformation of the 100% substituted mortar is up to nine times higher than that of the reference mortar. For lower substitution ratios the increase in final deformation is still three to six times higher than that of the reference mortar.

When pre-saturated aggregates are used, increase of drying shrinkage is a widely observed phenomenon. This is due to the higher deformability of lightweight aggregates according to Virlogeux [22]. In highly substituted mortars,

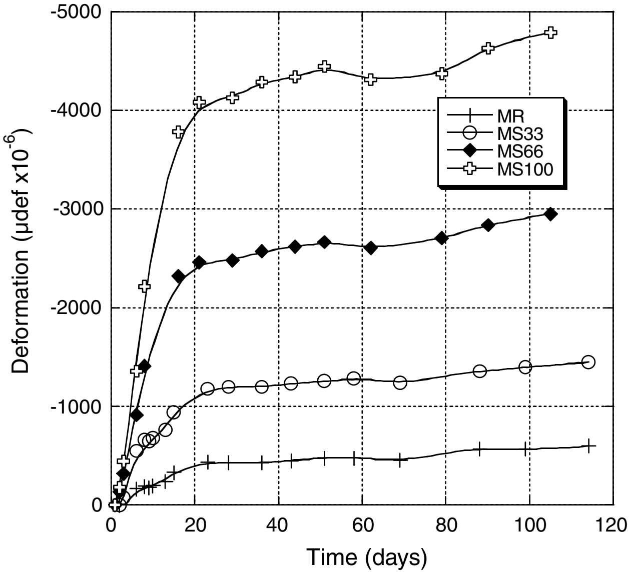

Fig. 5. Evolution of drying shrinkage of 4 × 4 × 16 cm^3 samples subjected to air drying.

the cement paste shrinkage is not impeded by rigid aggregates which leads to high final values. This shrinkage is not detrimental for the durability and the performances of the material as long as it does not lead to stresses and cracks. As Fig. 6 shows, large cracks have rapidly appeared on highly substituted mortars. Nevertheless, this was observed neither on MS33 nor MS66 mortars.

4.2. Compressive strength of mortars

This property is essential for the purpose of valorisation as it allows to assess the physical integrity of the material and to determine its possible reuses. Uniaxial compression tests were performed on cylindrical samples of 36 mm diameter and 72 mm length.

Fig. 7 presents the whole set of results for samples cured in water for 28 days. It is observed that introducing treated sediments as a substitute for sand leads to strength improvement. An increase by 20% is observed for the MS33 samples. For higher substitution ratios, compressive strength almost recovers the value of the reference mortar strength.

Fig. 6. Cracking due to drying on a MS100 beam kept in controlled atmosphere (after 1 month).

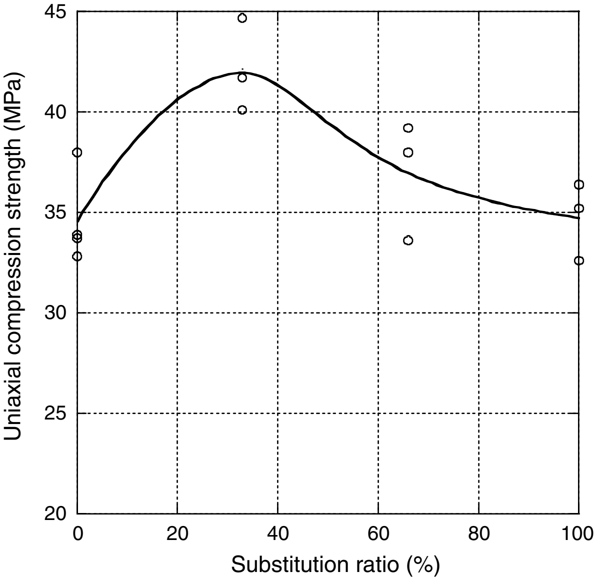

Fig. 7. Evolution of uniaxial compression strength of 28 days old mortars (water cured) as a function of substitution ratio.

An optimal substitution ratio can be therefore observed around 33%. Above this ratio, compressive strength decreases, though it is still comparable with that of the reference mortar. Feasibility tests performed in the laboratory have not revealed any pozzolanic activity. This was confirmed by the absence of vitreous phase in XRD analysis of treated sediments.

We believe that this behaviour can be attributed to two main factors. First, sediments are less resistant to abrasion than natural aggregates and they are naturally coated with fine particles. As a consequence, the mixing leads to a change in the expected granulometry with higher proportions of fines elements. An improvement of the material compactness may explain a part of the compressive strength increase. It is difficult to measure this change in granulometry, since as soon as treated sediments are mixed with water, the particles tend to agglomerate. Further drying leads to a hardly analysable paste.

The second contributing factor may be the difference of morphology between natural and treated sediment aggregates. The later are more porous which improves adherence between cement paste and aggregates [27,29]. The initial water content is supposed to lead to a more efficient hydration around aggregates [22,30,31]. However it is worth mentioning that other studies have reported opposite effects: the bleeding of saturated aggregates leads to a local increase of the W/C ratio and to a strength decrease [25]. Chemical interactions between cement paste and minerals present in the treated sediments may also be part of the strength increase [32,33]. Determination of the activity index according to the European standard EN 206 [44], should bring clues to understand involved mechanisms but this has not been performed in this feasibility study.

These factors may explain the increase in strength observed for the MS33 mortar. For MS66 and MS100 mortars, the decrease is probably due to a competition between the positive effect of TSAs discussed above, and the low strength of aggregates themselves which become the limiting factor at higher substitution ratios.

It is clear from these results that standard levels of strength can be obtained with reasonable ratios of substitution.

5. Conclusion

The Novosol® process presented in this study offers a reliable answer to the issue of polluted sediments. It fixes heavy metals in a matrix of apatite obtained after phosphatation of raw sediments. Organic pollutants are eliminated by additional calcination. This experimental study aimed at characterizing treated sediment aggregate and evaluating its ability to be valorised in cement-based materials.

A chemical, mineralogical and physical characterisation of this new material was performed. Its main characteristics are a low strength, a high porosity, a high amount of absorbed water, and a coating of fine particles. As reported in many valorisation studies on recycled aggregates, the porosity of TSAs has led to difficulties in mixing (necessity to pre-saturate the aggregates) and it has also led to a high drying shrinkage (up to nine times higher than that of the reference mortar for 100% of substitution). Nevertheless, other replacement aggregates (demolition concrete aggregates, scrap tyres, ...) are usually used with moderate substitution ratio. In such a case, the behaviour of treated sediment mortars would be acceptable for various potential reuses. Besides, no significant cracking was observed for 33% and 66% replacement.

Addition of TSAs in cement-based materials has also resulted in a significant compression strength improvement. An optimum substitution ratio has been observed around 33% with up to 20% of strength increase. For higher substitution ratios, compressive strength slightly decreases but remains higher than that of the reference mortar. Despite the high TSAs porosity, gas permeability was on the same order as that of a classical mortar, whatever the replacement ratio. Among the possible explanations are an improvement of the granular skeleton compacity due to TSA's fine coating, an internal curing due to pre-saturation water, and the porosity of TSAs leading to a better adherence between the cement paste and the aggregates.

The study proved the practical feasibility of treated sediments introduction in cement-based materials. Nevertheless, other formulations will be batched and additional tests performed, to improve our understanding. As an example, Neuville [27] underlines the low durability of lightweight concretes using saturated aggregates submitted to freeze–thawing cycles: this point will be investigated in a further study since TSAs also need to be pre-saturated before mixing. The high chloride content of TSAs potentially restrains valorisation pathways to some specific applications such as plain concrete for pre-casted blocks, artificial reefs, coastal defence blocks, ... The durability in aggressive environment (like seawater exposition), the effect of dry/wet cycling, should also be studied.

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