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Use of Treated Dredged Sediments as Road Base Materials: Environmental Assessment

C. Tribout · B. Husson · A. Nzihou

Abstract The treatment and the valorization of polluted dredged sediments is an important issue. The main benefit is the saving of natural resources and the prevention of landfilling. The valorization of sediments into road base materials require an accurate assessment of their characteristics. This study addresses the mechanical and leaching tests. The Novosol[®] process has been used to treat the dredged sediment and consists in the stabilization of heavy metals using phosphate and the degradation of organics by calcination at 700°C. The characterization (chemical, physical, thermal and environmental) carried out on the treated sediment shows that it is mainly composed with fine particles. The analyses have shown a low bulk density in comparison to that for standard sand. A significant total metal content and the absence of organic matter have been concluded. To evaluate the mechanical behaviour of the road materials, tensile strength and elasticity modulus have been measured. It has been seen that the introduction of treated sediments in materials treated with hydraulic binders improves the tensile strength. An environmental assessment has been made on these hydraulic-binder-treated materials thanks to a percolation test and the EN 12457-2 leaching test. The percolation test allows to evaluate the behaviour under accelerated conditions. The standard leaching test is used in order to assess pollution

potential. Both tests show that the release of metallic elements is almost the same for mixtures with or without sediments. The results suggest that the use of treated sediments in road materials is not harmful and brings mechanical benefits.

Keywords Treated sediments · Road based materials · Mechanical performances · Environmental assessment · Valorization

Introduction

Cleaning out ship canals or harbors is currently a necessity to ensure navigation. The sediments that are extracted are often polluted. The place where the sediments are dredged is bound to their pollution rate: if the dredged zone is located near industrial or urban sites, these sediments are in most cases polluted with heavy metals or organic compounds as PAHs [1–3]. It is difficult to manage them since their spread in agricultural land or their environmental release is no longer possible [4]. This contamination leads to classify the dredged sediments as “hazardous wastes”. Therefore, dumping seems to be the only way to get rid of them unless a treatment can favor economic value. Many treatments are available [5]. The process used in this work is a new physicochemical and thermal treatment for polluted sediments, so called the Novosol[®] process, developed by Solvay Company. In order to manage natural resources in a better way, using these treated sediments as a replacing product is proposed in civil engineering. Other studies have already worked on their valorization in bricks, mortars [6, 7]. In this paper, the treated sediments are introduced in road base materials. Building road networks implies an important need of sand and its replacement with treated

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sediments would allow to reduce the quantities that are nowadays extracted from quarries. This solution has been proposed as regard to the availability, the particle size and the chemical composition of the sediments.

In the first part of this paper, physical, chemical, thermal and environmental characterizations have been carried out to determine the main properties of the treated sediment. In the second part, road materials have been made incorporating this treated sediment. A determination of the mechanical performances of these road materials has been undertaken. Furthermore, an environmental assessment has been set up thanks to a percolation test and a standard leaching test.

Part I: Treated Sediment Characterization

Originally, the sediment considered was a polluted fluvial sediment extracted from a Belgium canal at Dampremy; it has been treated using the Novosol[®] process described below.

The treatment consists in 2 successive steps, presented in Fig. 1.

The step A consists in a phosphate treatment by mixing raw sediments with phosphoric acid H_3PO_4 (3–3.5%). This method is currently used to stabilize heavy metals by capturing them in a calcium phosphate matrix ($[Ca_3(PO_4)_2]_xH_2O$) [8, 9]. The sediments are then dried to decrease their water content and to allow their maturation, since calcium phosphates evolve in more stable structures with time.

The step B is a calcination. It consists of calcining the phosphated sediments at 700°C in a rotary kiln, in order to destroy the organic matter (polycyclic aromatic hydrocarbons and pesticides). It reduces the volume of processed materials after treatment and allows a better stabilization of metal phosphates [10, 11].

Gas emissions resulting from the treatment process (essentially H_2S and CO_2 , traces of heavy metals and dust) are removed to comply with the European emission standards [12].

Materials and Methods

The processed material, named “PC sediment”, is a new product and its characteristics have to be determined. Thanks to the different steps of the treatment that have mixed the sediment, we can consider that it is homogeneous. Moreover, a quartering of the sample has been made so the PC sediment is representative of the sediment that has undergone the Novosol[®] process.

Physical Characterization

The sediment can be described as a fine grained powder. It is composed mainly of agglomerates of fine particles.

For the size distribution, the fraction coarser than 80 micrometers was studied by sieving and the other fraction was studied in a laser particle size analyser. Density measures (bulk, absolute) have been made and the specific area has been achieved with the BET method.

The physical characteristics after processing are given in Table 1; in order to compare, those of a standard sand are presented in Table 2.

The size distribution shows that the treated sediment contains a higher proportion of fine particles than a standard sand since, if we compare this sediment with a standard sand that also has a d_{90} of 3.5 mm, the d_{10} of the sediment is lower (0.02 vs. 0.25 mm for the sand).

The bulk density of the sand is higher, that implies that the sediment is lighter than a standard aggregate. Its absolute density is the same. The PC sediment has a higher porosity (intergranular or/and intragranular). The presence of fine particles in the PC sediment induces a higher specific area.

Chemical/Mineralogical Characterization

The elemental composition of the treated sediment is presented in Table 3. Major oxides were measured by inductively coupled plasma-atomic emission spectrometry (ICP-AES) and the minor elements by inductively coupled plasma-mass spectrometry (ICP-MS). The water content

Fig. 1 Novosol[®] process

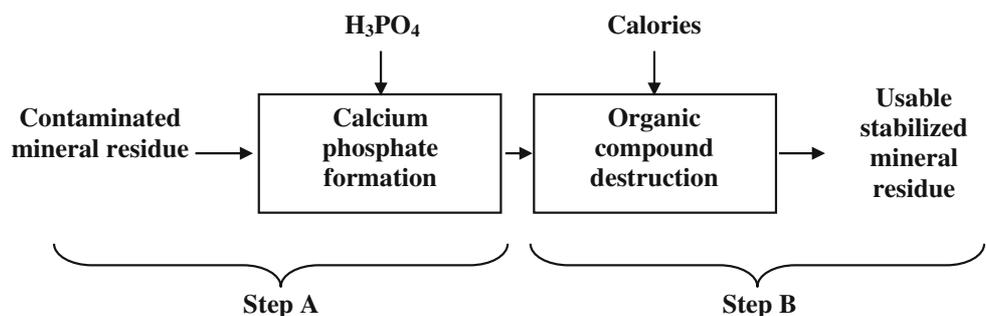


Table 1 Physical characteristics of the Novosol[®] sediments

Size distribution (mm)			Density		Specific area (m ² /g)
d ₁₀	d ₅₀	d ₉₀	Bulk	Absolute	BET
0.02	0.5	3.5	1.0	2.7	2.8

Table 2 Physical characteristics of a 0/4 rounded siliceous sand

Size distribution (mm)			Density		Specific area (m ² /g)
d ₁₀	d ₅₀	d ₉₀	Bulk	Absolute	BET
0.2	0.8	3.5	1.6–1.8	2.7	1.9

(drying at 105°C) and the loss on ignition (calcination at 1,000°C) were also measured. The chemical composition highlights a high quantity of heavy metals.

It can be noticed that the PC sediment has a high rate of pollutant, the three main ones are zinc, lead and chromium. It is mainly siliceous and contains also a high rate of ferrous oxide. The site where it has been dredged may be the origin of this important quantity. The water content is nil that is due to second step of the Novosol[®] treatment. The causes of the high value of the ignition loss can be identified thanks to the thermal analysis.

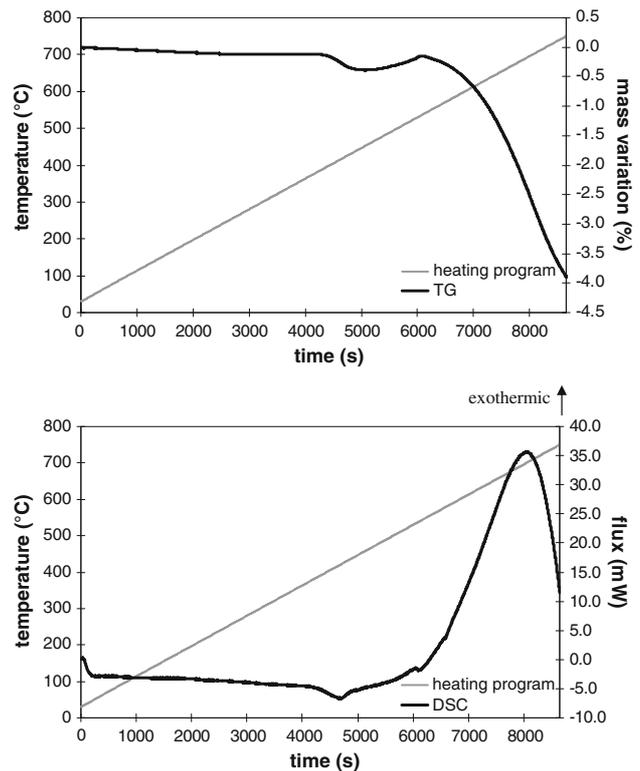
The crystalline phases were identified by XRD (Co K α , $\lambda = 1,789$ Å). The main minerals, in decreasing order, were quartz (SiO₂), haematite (Fe₂O₃), feldspars, gehlenite and lime (CaO). Other phases such as anhydrite (CaSO₄), dolomite (CaMg(CO₃)₂), monetite (CaHPO₄) and possibly whitlockite (β -Ca₃(PO₄)₂) have been detected in less quantity.

Thermal Characterization

Thermal analysis by TGA-DSC (TG-DSC 111, Setaram) was also performed on the treated sediment over a temperature range of 20–750°C. A heating rate of 5°C/min has been chosen since it allows to distinguish the various phenomena and to optimize the test time too. Figure 2 presents the result of the TGA signal and the DSC one; the heating program remains on each graph.

The main phenomenon observed is between 550 and 750°C: a loss in mass of about 4% with a very high exothermic peak. The important loss of mass has already been detected thanks to the ignition loss (see Table 3). This could be the superposition of two phenomena: decarbonation, leading to the mass loss, and calcium phosphate crystallization leading to the exothermic peak. It is well-known that a thermal treatment allows calcium phosphates to change their structure in a more stable and crystalline one [13, 14].

A slight phenomenon can be seen at about 400°C: a weak drop of mass (0.3%) with a small endothermic peak.

**Fig. 2** TGA-DSC curves of the treated sediments

It corresponds to the monetite dehydration (CaHPO₄) to form a calcium pyrophosphate (β -Ca₂P₂O₇) [15].

The absence of strong exothermic peaks between 200 and 700°C shows that there is no longer organic matter in the PC sediment [10, 16].

Environmental Characterization

The aim of the environmental characterization is to assess the pollution potential of the PC sediment. Contaminants leaching is controlled by many factors; one of them is the

Table 3 Chemical composition of the studied PC sediments

Major oxides content (%)		Minor elements content (mg/kg)			
SiO ₂	46.4	Zn	3,737	La	57
Fe ₂ O ₃	19.1	Pb	777	Nd	55
Al ₂ O ₃	9.8	Cr	450	Y	48
CaO	9.7	Ba	412	As	23
P ₂ O ₅	2.9	Zr	255	Co	23
MgO	2.3	V	175	Pr	15
K ₂ O	1.6	Sr	164	Ga	14
Na ₂ O	1.1	Cu	134	Th	13
TiO ₂	0.6	Ce	125	Nb	11
MnO	0.2	Ni	115	Sm	10
Water content	0.0	Sn	70	Cs	10
Ignition loss	5.8	Rb	66	Cd	9

pH, which is the main leaching controlling parameter. The pH value of the material and of its environment are crucial in determining the release of many constituents.

Among the available leaching tests, two of them have been chosen: a “characterization” test and a “compliance” test, defined by the CEN/TC 292 framework [17].

The « pH dependence test » (prEN 14429) [18] appears to be one of the principal characterization tests. It is recommended by many studies [19–22] to cover large pH differences in the environment, to get the buffer capacity, in order to have a sight of the product sensitivity in external conditions.

The test consists of parallel extractions of the crushed material at an L/S = 10 (L/kg) for 48 h at a series of pre-set pH values. Various amounts of acid and base are added to obtain a final pH. After an equilibration period of 48 h, concentrations of elements are measured in the eluates. The test also gives an indication of the buffer capacity of the product. The test is designed to represent chemical and physical equilibrium conditions.

The Toxicity Characteristic Leaching Procedure (TCLP) has been chosen as the compliance test. Most of the compliance leaching tests use demineralised water as extraction liquid but the TCLP test uses acids. The aim of this environmental characterization is not to represent real conditions but rather to estimate the PC sediment release in hard environmental conditions, that’s why the TCLP has been used.

It was performed as specified in the EPA method [23]. Prior to extraction, the solid material was passed through a 9.5 mm standard sieve. Then it was extracted for 18 h with an amount of extraction liquid equal to 20 times the weight of the solid phase.

The extraction liquid employed was chosen on the basis of waste alkalinity (CH₃COOH, pH = 2.88 ± 0.05). Following the extraction, the liquid extract was separated from the solid phase by filtration and analysed.

Until yet, a regulation permitting valorisation and including thresholds does not exist. In this study, the results obtained through the TCLP test will be calculated thanks to the METOX, which is a parameter created by water agencies to calculate toxic pollution fees. This parameter concerns 7 metals and 1 metalloid: arsenic, cadmium, chromium, copper, mercury, nickel, lead and zinc. It is calculated by the weighted sum (in g/L) of these metals and metalloids, using weighted coefficients that take the element toxicity into account [24].

Table 4 Balancing index of the metals used for the calculation of the METOX parameter

Hg	Cd	As	Pb	Ni	Cu	Cr	Zn
50	50	10	10	5	5	1	1

The list of coefficients of each element is presented in Table 4.

It is important to notice that the METOX parameter is not a global pollution indicator and has many limits. It does not take all pollutants into account and disregards synergies or antagonisms that exist between pollutants or with other substances. It takes neither speciation nor bioavailability into consideration.

All these reasons induce that the results obtained thanks to this parameter should be used with caution.

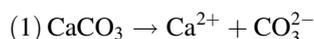
The PC sediment results will be compared with those obtained on the sediment that has just been phosphated (“P sediment”).

Results and Discussion

The solubility of elements is investigated using the pH dependence test so the behaviour of the sediment under various pH has been assessed. Figure 3 represents the pH versus the quantity of acid added. The acid neutralization capacity (ANC) can be derived from the amount of acid/base used to reach a given pH.

The natural pH of the PC sediment is 12.2. Other studies have already shown that natural pH of such treated sediments is between 10.3 and 12.6 [11].

The first plateau at pH 12 confirms that the PC sediment contains lime. The drop of the pH between pH 12 and pH 7 after adding 0.6 meq H⁺/g of PC sediment corresponds to the neutralization of OH⁻ and CO₃²⁻ ions. A new plateau is reached at pH 6–7; this fact is due to the carbonates dissolution. For instance, for calcite :



The pH of the mixture will not drop anymore until carbonates vanish whatever the amount of acid added.

The next drop of pH up to pH 4 corresponds to a reaction between bicarbonate ion and acid to form carbon dioxide:

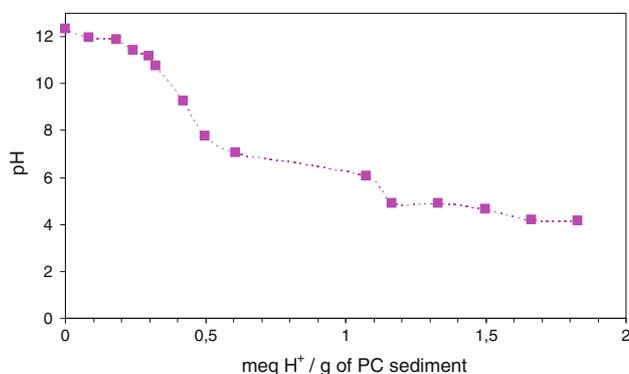


Fig. 3 pH versus added acid quantity



The last inflexion point is at pH 5.5. This zone is the end of the acid neutralizing capacity of the PC sediment. A quantity of 1.1 meq H^+ /g is necessary to reach this point. This value is quite low in comparison with bottom ash for example that have shown a neutralizing capacity of about 3–5.5 meq H^+ /g [25].

The stabilization thanks to an hydraulic binder such as cement matrix is very beneficial to this kind of materials [19].

A particular focus on the behaviour of three main heavy metals that have been detected in the PC sediment (zinc, lead and chromium) has been made. The results obtained are presented in Fig. 4.

The thick continuous lines represent the initial total composition of each element in the PC sediment. The thin lines are the release that has been obtained for the different pH values. Release curves are similar and systematic for different groups of element (salts, cations, anions) [26] and the results obtained for the PC sediment follow the general leaching behaviour.

The result of a pH dependence test informs on the amounts of metals released under different exposure conditions. It can be noticed that the behaviour of each species that is presented here is different: zinc and chromium present a higher availability for acid pH values whereas chromium has its maximum for basic ones. These results show the importance of the pH of the environment in which the waste

will be introduced. The PC sediment will be exposed to a basic environment as a cement matrix; thanks to Fig. 4, each element availability can be estimated by making the ratio between the recorded release and the total composition :

- 0.02% for lead;
- 0.07% for zinc;
- 0.3% for chromium.

In the foresee future environment of the PC sediment, the availability for leaching of these heavy metals will be almost non-existent, even for chromium whose availability was higher for basic pH.

The behaviour of the sediment has been also investigated at constant pH using TCLP leaching test. The results obtained as well as the impact of each metal using a ME-TOX index are presented in Table 5.

The total release is always higher for the P sediment. These results show that the calcination allows to reduce the environmental impact of the sediment.

Conclusion of Part I

The study of the characteristics of the PC sediment have shown that it has a high rate of fine particles and an important heavy metals content. Its buffer capacity is not important making favourable the use of this sediment in basic environment such as cement matrix in general. This beneficial use is discussed in part II.

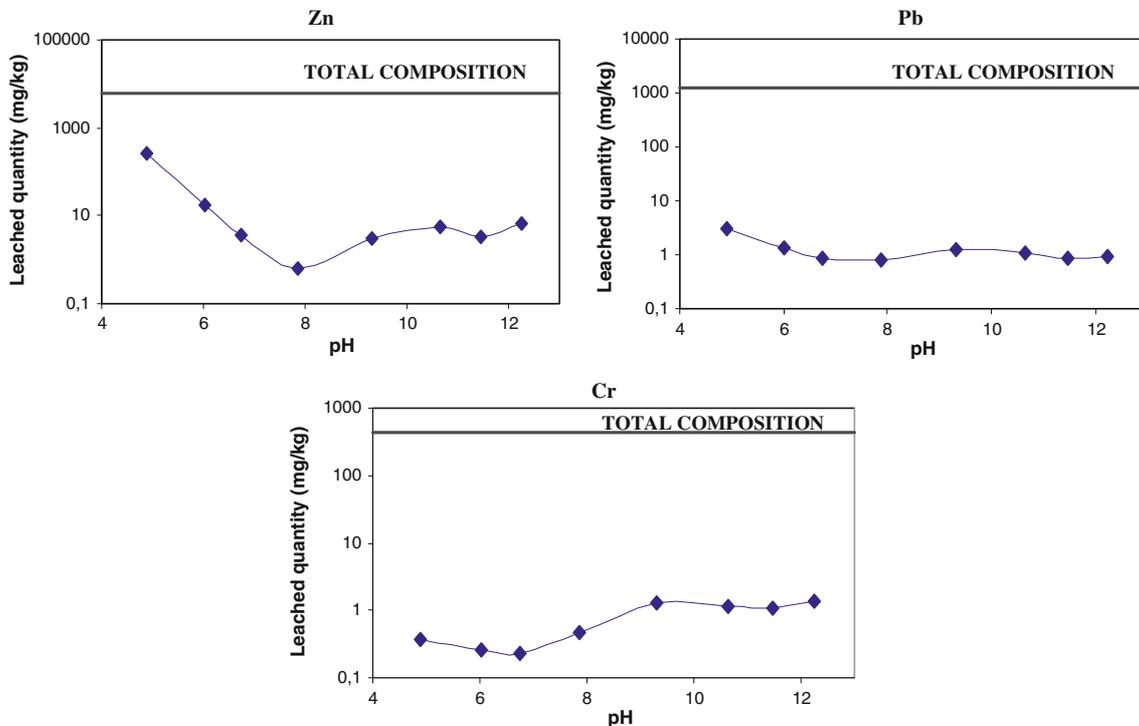


Fig. 4 Heavy metals leaching as a function of pH

Table 5 Release of the METOX elements through the TCLP test

ppm	PC sediment	P sediment	METOX coefficient	PC sediment—METOX	P sediment—METOX
Hg	<0.1	<0.1	50	<5.0	< 5.0
Cd	0.6	2.1	50	30	105.0
As	1.6	2.2	10	16	22.0
Pb	5.9	5.0	10	59	50.0
Ni	2.8	5.9	5	14	29.5
Cu	1.8	2.6	5	9	13.0
Cr	1.0	0.6	1	1.0	0.6
Zn	158.2	739.2	1	158.2	739.2
Total	172	757.7		292.2	964.3

Part II. Valorisation of Treated Sediments in Road Materials

The aim of this second part is to replace a fraction of the sand used in road materials by the sediment.

Materials and Methods

Composition of Road Base Materials

Hydraulic-binder-treated materials are often used for road base buildings. In this study, 0/2 hydraulic-binder-treated sands (STLH) have been made. The constituents of the STLH were 0/2 sand, Portland binder and Novosol[®] sediments. The sediment was crushed so as to respect the size distribution (0/2 mm). Reference STLH (ST0) were made in order to be compared with the test material which was made by incorporating Novosol[®] treated sediments in the formulations as a replacement for a fraction of the sand (SS3). The aim was to make formulations which would satisfy road use criteria and which would have mechanical characteristics comparable to standard materials.

The percentages of all granular constituents have been chosen according to the EN 14227-1 standard [27]. For the binder, a value of 7% has been fixed. The treated sediments replaced 30% of the sand because beyond this value, mechanical performances decrease [28]. Furthermore, a test road was made in [29] with the introduction of 30% of Novosol[®] sediments with good results.

The granular compositions are presented in Table 6.

Table 6 Granular compositions of STLH

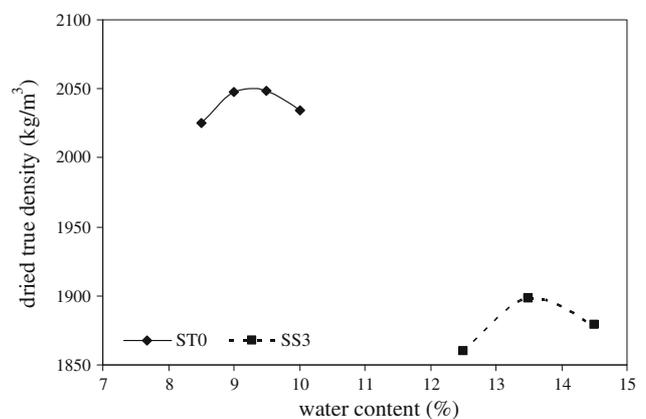
	STLH	
	ST0 (%)	SS3 (%)
Treated sediment 0/2	–	30
Siliceous standard sand 0/2	93	63
Binder (CEM I 52.5R)	7	7

The water content and the bulk density were determined by the Modified Proctor test (OPM) in accordance with the EN 13286-2 standard [30]. The curves obtained by the Modified Proctor test are presented for each STLH in Fig. 5.

The presence of treated sediments induced an increase in the water content coupled with a drop in the density.

The introduction of sediments corresponds to an addition of fillers, which could lead to a greater need for water. The density decrease for mixtures incorporating sediments is explained by a lower density of sediment particles compared to the sand particles they replaced.

Table 7 shows the various compositions made and already used in other studies [28, 29].

**Fig. 5** Modified Proctor curves for STLH**Table 7** Summary of the STLH compositions

	ST0 (%)	SS3 (%)
Treated sediment	–	26.4
Siliceous standard sand 0/2	85.1	55.4
Hydraulic binder (CEM I 52.5R)	6.4	6.2
Water	8.5	12
Dried true density (kg/m ³)	2.1	1.9

The samples are made according to the in-force standards [31, 32]: STLH are cylindrical samples ($\Phi = H = 5$ cm) made by static compression.

Evaluation of Road Materials

In a road scenario, the performance levels to be recorded concern the tensile strength, R_t , and the modulus of elasticity, E .

An indirect tensile strength test was chosen so as to obtain R_t and E together. In this case, EN 13286-42 [33] and -43 [34] describe the experimental procedure for the test.

For R_t , indirect tensile strength R_{it} is converted into direct tensile strength R_t according to (1).

$$R_t = 0.8 \times R_{it} \quad (1)$$

For E , 2 strain gauges should be used but, for such type of samples, it is too difficult. Other studies [35] have shown that it is possible to use only one strain gauge.

Both tests have been made on three samples of STLH.

For the environmental assessment, two tests have been used:

- a percolation test which is not standard but allows to work on monolithic materials;
- the EN 12457-2 leaching test [36] which is standard but needs crushed materials.

A percolation test has been chosen to evaluate the environmental behaviour of the materials. Previously, this test was used to forecast the long term behaviour of rocks in civil engineering [37, 38]. In order to accelerate the processes and to reduce the time scale, various parameters

had been modified (temperature, fluid aggressiveness,...). The test presented in this paper is based on the pressure variation. Recently, it has been adapted to allow to work on cementitious materials [28]. The equipment is a permeameter (Fig. 6).

It is made up of :

- (1) a pressure generator to adapt the pressure to the sample. It is made of a reservoir which is kept under pressure thanks to compressed gas. The available range is 0–40 bars;
- (2) a box, containing the sample, which allows percolation (axial or radial). It is made of stainless steel and the watertightness is performed thanks to various joints.
- (3) a device to collect the fluid released from the box.

Percolating samples have been submitted to a downward axial percolation, using demineralised water as fluid. The samples have been resized to enter the box and we make them laterally waterproof in order to force the fluid to go through the sample only in the vertical way.

To avoid creating preferential flows for the fluid, the box is returned, a weaker pressure is applied and then the sample suffers an ascendant percolation for saturation. The percolation test begins when the fluid emerges from the cell.

The pressure is chosen so as to obtain a liquid/solid ratio of 10 L/kg in about 10 days. Preliminary tests had been carried out so as to choose 35 kPa for SS3 and 10 kPa for ST0.

During this test, the flow is measured and the fluid is collected for the determination of heavy metals content.

The second test is the EN 12457-2 standard leaching one that has been chosen to estimate pollutant potential. It is

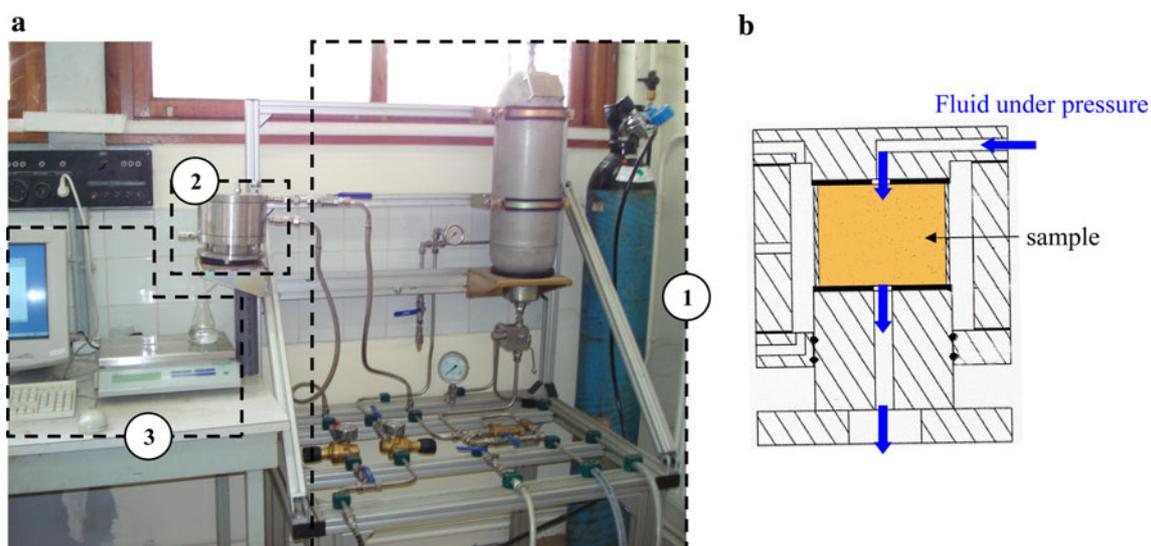


Fig. 6 Experimental device of the percolation test (a)—schematization of the percolation box in axial mode (b)

carried out on a sample crushed to less than 4 mm and immersed in demineralised water at a liquid/solid ratio of 10 L/kg. Extraction during 24 h is necessary to get the leachates that are then analysed using ICP-AES.

Results and Discussion

Mechanical Performances

The EN 14227-1 standard [27] suggests classifying the mixtures according to the values of their performance (E , R_t) at 28 days (Fig. 7). As we can see, both mixtures belong to the T3 traffic class.

In the French standard, road base materials have to belong to the T2, T3 or T4 classes. The suggested STLH are thus relevant for use in roads.

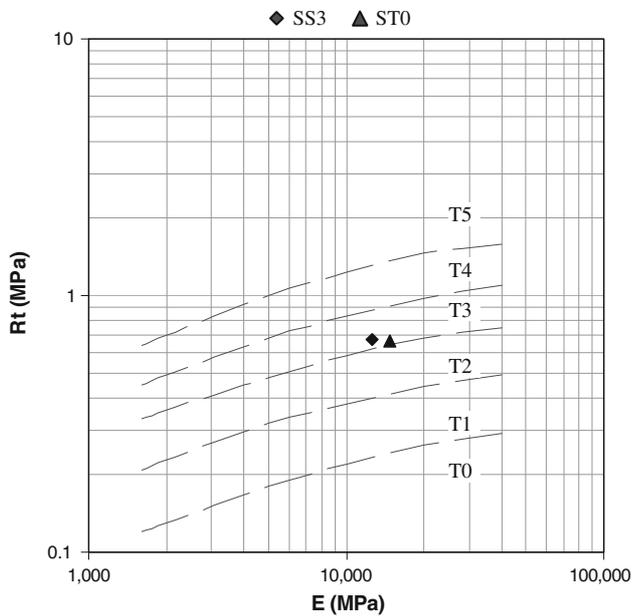


Fig. 7 Classification of the mechanical performances of 28-day-old STLH samples

Environmental Evaluation of the STLH

Percolation Test Figure 8 shows the flow for STLH during percolation and Fig. 9 shows the release for chromium and zinc as a function of L/S.

In Figure 8, an initial peak is observed but all samples reach a steady flow. The value of the steady flow is similar for all the samples.

In Figure 9, the trend is the same for both samples. The release for SS3 is slightly higher than for ST0 but the order of magnitude is quite similar. These results show that the samples with treated sediment do not release more heavy metals than those without.

Leaching Test Results of the leaching test are given in Table 8.

It can be seen that the quantities of heavy metals released are not very high since only chromium (and zinc for ST0) were measurable. The other elements have concentrations below the detection limit.

Moreover, the results show that the amount of heavy metal released was almost the same for both mixtures. The introduction of sediment does not seem to have any negative effect on the release.

Conclusion of Part II

The mechanical performances have shown that all the mixtures are standard so they can be used on roadworks.

The environmental behaviour has been studied thanks to water leaching and percolation tests. The percolation test allows to work on monolithic materials as it is in real working conditions. Both tests show that the mixtures with sediments do not release more pollutants than those without sediments, which are already used in road construction. The same amounts of heavy metals are released from both mixtures.

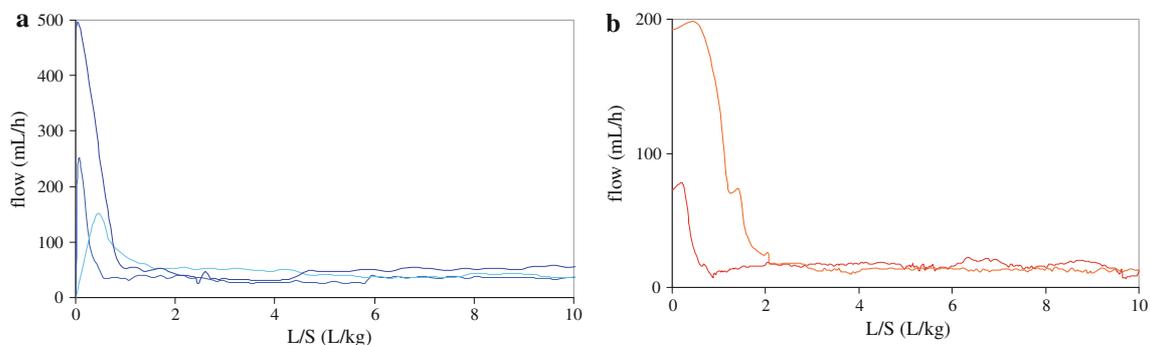


Fig. 8 Flow versus L/S for ST0 (a) and SS3 (b)

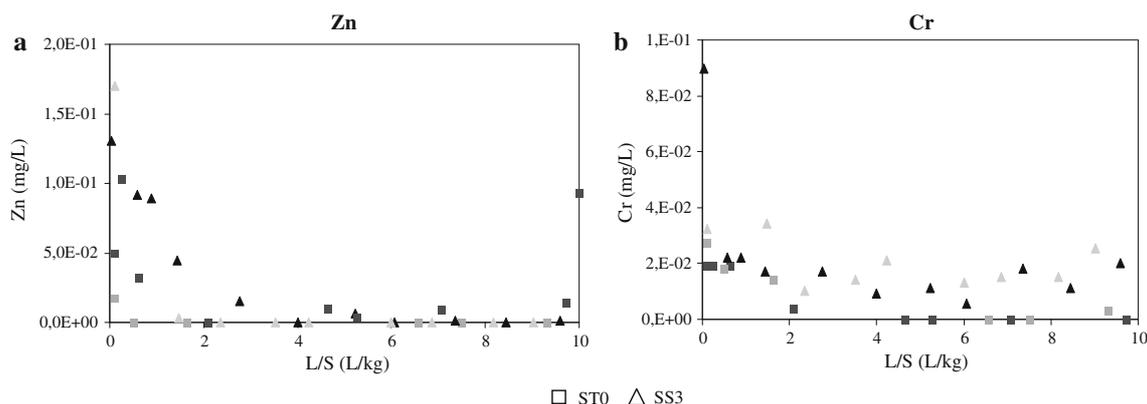


Fig. 9 Release of Zn (a) and Cr (b) as a function of L/S

Table 8 Concentrations of leached elements

	Cd	Cr	Cu	Pb	Zn
Minor elements content ($\mu\text{g/kg}$)					
SS3	<l.d.	175 (\pm 13)	<l.d.	<l.d.	<l.d.
ST0	<l.d.	189 (\pm 7)	<l.d.	<l.d.	30.7 (\pm 0.0)

<l.d.: under the detection limit

Concluding Remarks

Waste valorisation needs to put in place a study strategy.

The knowledge of the main characteristics of the waste is really necessary to imagine an adapted valorisation scheme. The study of the performances of the formulation proposed is useful to discuss the safety of the valorisation.

The results obtained confirm that Novosol[®] sediments can achieve economic value by being used as a substitute for some of the sand in road building techniques. Even considering all the different constraints existing in a scenario of valorisation, their use in road materials is entirely possible.

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