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## Percolation as a single test to assess pollution potential of a waste

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Abstract A new test of percolation under pressure is proposed to estimate the pollution potential of wastes in various scenarios. Depending on the use foreseen for the material, samples can be analysed in a granular or monolithic state. The proposed trial keeps the physical structure of the samples and accelerates the phenomena by applying a pressure variation in order to obtain many important data, such as permeability and the environmental release of species. In this paper, some results of this percolation test are presented for hydraulic-binder-treated gravels and sands that include polluted treated sediments. The results are repeatable and a steady flow is reached for each kind of sample. Incorporation of the waste leads to a drop in the permeability of road materials even though their characterisation shows that they are more porous. The capacity to resist aggressive ion penetration can then be estimated. The release kinetics of heavy metals are studied and the environmental release results are compared with data from a regulatory leaching test. Differences can be observed and the percolation test seems to be more sensitive for most of the heavy metals measured.

Keywords: environmental assessment; percolation test; monolithic materials; waste valorisation road materials.

## Introduction

At present, the choice of the tests to assess the potential pollution due to waste materials is dependent on European directives reported in the standard EN 12-920+A1 [1]. It recommends a methodology taking both the specific properties of the waste and the disposal scenario into consideration. A waste can be produced in granular form and can later be incorporated into monolithic material (for disposal or reuse). This difference in the physical form of the waste has to be taken into account. For instance, road structures, which need large quantities of granular materials could readily accept granular wastes. Hydraulic-binder-treated materials are widely used for foundation layers. They are monolithic and, in real conditions, will suffer rain water infiltration. If wastes are to be put to use in such a way, their release kinetics have to be estimated. This scenario suggests that infiltration of water is one of the most likely phenomena the material will face, as the roadway is exposed to the elements. The water will flow by gravity into the layers below the surface, including the layer of waste enclosed in a cement matrix. In consequence, the main risk is the release of some pollutants into this percolating rainwater. Pollution of both the soil and the water table by way of this polluted water are then possible. Several methods are available to study the release and the behaviour of road materials, such as leaching or percolation tests. Until now, current regulations governing the estimation of environmental impact have focussed on leaching criteria. Most leaching tests use granular materials, which does not suit our purpose. There are also monolithic leaching tests [2,3] but the main drawback to these is that they are wash-off tests, and reactions can take place only at the surface of the materials. These tests are not suited to a road scenario since they propose either crushing the material or keeping it in contact with the liquid.

Building a lysimeter or a road at 1/1 scale could be an option. These solutions are convenient since the material is in the same conditions as it would be in a road structure. However, it is not always desirable to use these possibilities since they need time to obtain results and are very expensive.

A percolation test remains the most suitable for a road scenario. However, few percolation tests exist at present. The only standard one in France is the NF CEN/TS 14405 test [2]. It is a characterisation test that determines the release from compacted granular materials subjected to upward axial percolation of demineralised water. The upward direction was chosen to prevent preferential paths being taken by the fluid. However, only granular materials can be tested in this column and crushing monolithic materials would increase the contact area and induce changes in the reactions. The American standard ASTM D4874-95 [3] proposes a similar test, the NEN 7343 Column Test [4]. It is like the French one except for the nature of the fluid: it requires water that has been acidified to pH 4. The main disadvantage of these tests is their non-polyvalence; **they do not suit the scenario that is forecasted since they do not respect the future material state or the conditions do not represent the aggressiveness of the fluid that will go through the real material.**

The development and technology of a new percolation test are reported in this study. It assesses the pollution potential of a waste in various scenarios. Granular or monolithic materials can be tested by applying radial (convergent or divergent) or an axial percolation. Many kinds of materials have already been tested: fly ashes, siliceous and calcareous filler, concretes [5], polluted treated sediments [6] and road materials [6,7].

In this paper, only results for road materials, such as hydraulic-binder-treated gravels and sands, are presented. Reference samples were tested but other formulations were also made: a polluted sediment that had been treated but was still considered as a waste was introduced into road test materials as a partial replacement of the sand. The results obtained from various

tests carried out to gain a better understanding of the phenomena occurring during this original test are also reported.

## **Materials and methods**

### ***Percolation test device***

The initial idea was to follow the same thought process that, in the 1950s, led Capdecombe et al. [8], cited by Nguyen [6], to find a laboratory method for accelerating processes that happen in nature. During the tests, many factors were modified, such as the pH of the percolating fluid, the granular state of the material, the temperature and the pressure difference used to increase the flow. Our aim was just to accelerate the phenomena without changing too many parameters. The most relevant way to accelerate the phenomena seemed to be to increase the flow by means of a pressure variation. It was also possible to change other parameters but we preferred not to change the aggressiveness of the fluid so as to stay closer to reality. Thus, we decided to keep the structure of the materials, to accelerate the phenomena by a pressure difference and not to change the aggressiveness of the fluid.

The percolation device was a permeameter, improved thanks to many works [9–11,5,6]. It consisted of a pressure generator, a box and a collecting device. Details are given below.

### ***Pressure generator***

The experimental device is presented in Figure 1. The pressure generator consisted of a reservoir in which the fluid was kept under constant pressure by means of nitrogen. A confinement pressure could also be applied. The reservoir and the associated piping and taps were manufactured in stainless steel to avoid corrosion. This generator worked with gate valves and air/water drainage of the lines. The fluid supply system allowed the injection pressure to be set between 10 kPa and 4 MPa.

### *Percolation boxes*

Depending on the cohesion of the sample, the flow could be axial or radial. Three configurations could theoretically be employed: axial flow and convergent/divergent radial flow.

The box for monolithic materials subjected to downward axial percolation is presented in Figure 2.

This box was used for materials that could only undergo axial percolation, such as compacted powders, weak concretes, materials with high permeability and road materials. For materials with low permeability, this configuration would take too much time. Using a radial configuration could be a solution since it would allow the thickness of the materials to be reduced. This is suitable and required for samples that need a high pressure to be percolated. Such a configuration needs the samples to be perforated so as to obtain an axial hole to allow a large mass of material with a small thickness to be tested. It also implies that the samples need to have sufficient strength to undergo coring. The advantage is that the pressure applied can be smaller, thus reducing sealing problems.

Many joints (in black in figure 2) were used to ensure the sealing of the box. A lateral confinement pressure could also be applied for samples requiring high pressure to be percolated. A spacing ring was placed above the sample to ensure that there was sufficient water in contact with the upper side of the sample and also to let the fluid spread out uniformly. To ensure that the samples used were representative, two boxes were available, which could accept aggregates of different diameters. For hydraulic-binder-treated sands, the maximal diameter of the samples was 70 mm; for gravels, a larger box, with a maximal

diameter of 100 mm, was available. The percolation boxes could be adapted to the length of the samples with a maximal length of 60 mm.

#### *Collecting system*

The flow was estimated using a computer linked to a balance that recorded the quantity of percolates that passed through the sample. For the chemical composition of the percolates to be measured, they first had to be collected. Since the samples were not protected from carbonation, percolate collection was not continuous.

#### *Materials tested*

Road materials, such as 0/2 hydraulic-binder-treated sands (STLH) and 0/20 gravels (GTLH) were made according to road standards [12–18]. “Waste” was also used to partially replace the sand. The waste was originally a polluted sediment classified “B” in Belgium, which means it cannot be reused without treatment to make it inert. It was therefore treated by the Novosol® process proposed by Solvay. This waste has been fully studied in other works [19,7]. Its main characteristics are a high percentage of fine particles (30-40% smaller than 80 µm) and a high heavy-metal content (the three main ones being zinc [3,737 mg/kg], lead [777 mg/kg] and chromium [450 mg/kg]). Other studies have already considered the valorisation of polluted sediments (treated or not) in many kinds of materials (concrete, brick, road materials...)[20–22]. It has also been shown that a cement-based solidification is beneficial from the environmental and mechanical points of view [19,23]. Building hydraulic-binder-treated materials has the same advantages since it also leads to solidification.

In order to respect the granular class of the STLH, the sediment was crushed to a 0/2 particle size distribution. For GTLH, it was used as it was. On the basis of previous works [6], sand replacement percentages of 30% in STLH and 15% in GTLH were chosen. For STLH, two



mixtures were made: a reference one, named ST0, and one with the waste, named SS3. For GTLH, the reference mixture was GT0 and the one with the waste was GS15. The following tables show the composition of each of the road materials.

[Tables 1 and 2 near here]

### ***Experimental protocol***

#### ***Preparation of samples***

The samples were resized to fit into the box [6]. In order to be representative as far as the maximal diameter of aggregates is concerned, it is usual to choose:

- a sample diameter at least four times the aggregate dimension;
- a thickness at least twice the aggregate dimension.

The hydraulic-binder-treated sands were sawn to provide samples with a diameter of 50 mm and a height of 45 mm. Gravels were cored and sawn into samples with a diameter of 93 mm and a height of 50 mm.

In order to force the fluid to go through the sample in the vertical direction only, the samples were laterally waterproofed with vinyl ester and polyurethane resins. PVC pipe was also used for reinforcement (see Figure 3). The seal obtained in this way was tested and gave good results [6].

#### ***Percolation test***

The aim of this test was to apply axial percolation of a fluid percolating under pressure to accelerate the phenomena. Downward percolation was chosen as being closer to reality. Demineralised water was used as the percolating fluid. The pressure was chosen so as to obtain a liquid/solid ratio of 10 L/kg – typical in leaching tests – in a reasonable delay of ten days. The flow was then adjusted by varying the injection pressure. Previous tests had given

an estimate of the pressure for each kind of sample. It is important to note that, in the case of monolithic samples, the stress created in the specimen was a limiting factor. It has been shown that there is a limit pressure that cannot be exceeded without damaging the material [10,5,24].

. Before beginning the test proper, it was necessary to saturate the materials in order to avoid creating preferential flows. Immersing samples in water would have induced unwanted leaching, which is why upward percolation was chosen for this step only [6]. A pressure less than or equal to that chosen for the test was applied. As soon as the fluid emerged from the box, the test was considered to begin and the box was inverted to apply downward percolation.

The minimal quantity of leachates required was estimated at 10g. The criterion for stopping the test was L/S reaching 10 L/kg. Since the pressure was kept constant, the flows were different for each sample, which led to variable test durations. Many data were recorded [7,19] but only the three main pollutants, Zn, Pb, Cr, are presented here.

### ***Complementary tests***

#### *Characterisation of the samples*

In order to better understand the behaviour of the road samples studied, several methods were used to characterise them. Gas adsorption (Micromeritics, ASAP 2010, USA) and mercury porosimetry (Micromeritics AUTOpore III) were employed to characterise porosity. For gas adsorption, nitrogen was chosen, the sample having been degassed for 24h at 105°C before the analysis. For mercury porosimetry, a pressure increment method was used and two cycles of intrusion-extrusion were applied for each sample. However, such methods could only analyse materials that had a maximal diameter of 80 mm, which was quite small compared to the aggregate dimensions of the road materials studied. Therefore, only STLH

samples were tested since characteristics found for GTLH would not have been representative. Measurements were made on various samples to ensure representativeness. For percolated samples, it was necessary to dry the samples at 50°C before coring. For the STLH compound characterisation, thermal analysis by TGA-DSC (TG-DSC 111, Setaram) was also performed over a temperature range of 20-750°C. Considering the results of previous experiments [7], a heating rate of 5°C/min, under air atmosphere with a flow rate of 33.3 mL/min was chosen. Scanning electron microscopy in secondary electron imaging mode was also used on fractures of STLH to study their structure.

#### *Comparison with a standard leaching test*

A standard leaching test was also applied to the four mixtures of road samples so as to compare the results obtained with those from the percolation test. The EN 12457-2 test [25] was chosen since it has similar operating conditions, using demineralised water and an L/S of 10 L/kg as in the percolation test. This test was 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. Only 24 hours' extraction was necessary. The lixiviates were then analysed by ICP-AES. The releases, cumulated until an L/S of 10 L/kg was reached, were then compared for the two tests.

## **Results and discussion**

### ***Results from the percolation test***

Preliminary tests had indicated 10 kPa for ST0, 35 kPa for SS3 and 2.2 MPa for GT0 and GS15 as the best pressure value choices. For gravels, the pressure needed was high, so a lateral confinement pressure was also applied at the same value. Four samples of each GTLH and three of each STLH were tested.

The flow showed a typical curve for all the samples. At the beginning of the test, a decrease in the flow was noted. This phenomenon was more or less pronounced but a steady flow was attained for all the samples before an L/S of 2 L/kg was reached.

The proposed percolation test used a permeameter and it was therefore possible to find a relevant value for porous materials undergoing this kind of test: their permeability. It was assumed that the actual ensemble of grains making up the porous medium was replaced by a “representative continuum for which we can define macroscopic parameters [...] and utilize macroscopic laws [...] to provide macroscopically averaged descriptions of the microscopic behavior” as proposed in [26].

The permeability  $k$  (sometimes called *specific* or *intrinsic permeability*), which represents the ability of a porous material to be crossed by a fluid under a pressure gradient, was measured using Darcy’s law. It was assumed that the pore volume was completely filled with water. Darcy’s law is valid for steady, laminar flow. If a 1D-flow in a homogeneous, isotropic material is considered, the permeability for an incompressible fluid can be obtained from the following equation [27]:

$$k = \frac{\mu * Q_s * L}{A * (P_e - P_s)} \quad (\text{Eq1})$$

where  $A$  is the area of the sample  
 $L$  is the length of the sample  
 $P_e$  is the pressure at the entry of the sample  
 $P_s$  is the pressure at the exit of the sample and  
 $\mu$  is the dynamic viscosity (for water:  $10^{-3}$  Pa.s)

$k$  is a function only of the medium and has dimensions [length<sup>2</sup>].

The results for all the samples are presented in Table 3.

The average flow corresponds to the value when the steady flow is established.

A steady flow was reached for each road material and the time delay was also respected. For the STLH, applying a higher pressure for SS3 ( $3.5 \cdot 10^4$  versus  $1 \cdot 10^4$  Pa for ST0) did not lead to a higher flow (20 mL/h for SS3 versus 35 mL/h for ST0). It was even necessary to wait twice as long to obtain an L/S of 10 L/kg. Thus, comparing the permeability of SS3 ( $36 \cdot 10^{-16}$  m<sup>2</sup>) and ST0 ( $220 \cdot 10^{-16}$  m<sup>2</sup>) revealed that the permeability of ST0 was ten times that of SS3. For GTLH, we noted that, although the pressure applied was the same ( $220 \cdot 10^4$  Pa), the average flow was different (97 mL/h for GT0 and 40 mL/h for GS15). As for the STLHs, the time to reach an L/S of 10 for GS15 samples was twice as long. Therefore, including sediments in the mixtures implies a decrease in the road material permeability ( $0.34 \cdot 10^{-16}$  for GS15 versus  $0.81 \cdot 10^{-16}$  m<sup>2</sup> for GT0). This reduction was less marked for GTLH, possibly because a lower quantity of sediments was introduced (15% for GTLH versus 30% for STLH). These results show that it is not possible to obtain a given L/S in the same time for such different kinds of materials. Many tests have shown that, although the time to reach an L/S of 10 L/kg is different, the release behaviour is similar if we consider the parameter L/S rather than the time [6,7]. Moreover, most standard tests use this unit because it also allows comparisons to be made between tests that have different durations.

### ***Characterisation of STLH samples***

#### *Differences between samples with or without sediment*

From the permeability results, a first impression may be that the reference samples have higher permeability because they are more porous. Measurements made of the porosity accessible to water (see table 4) showed, however, that this was not the case .

In order to understand the origin of this increase of porosity for samples with sediments, BET adsorption methods and mercury porosimetry were used. Both methods showed a major

difference between ST0 and SS3 samples; only the results from mercury porosimetry are presented (see figure 4).

A larger volume of mercury was introduced into SS3, confirming that the samples were more porous. A greater mesoporosity was detected for SS3 samples, with an average diameter of 0.03  $\mu\text{m}$ . A consequent jump on the curve can be observed for SS3 samples between 0.003 and 0.1  $\mu\text{m}$ , which corresponds to the diameters of hydrate pores. These measurements show that the increase in the porosity of SS3 samples was due to the greater presence of hydration products. This phenomenon also explains a gain in strength for mixtures with sediments [7]. Therefore, samples with sediment have a lower permeability but a higher porosity, mainly due to a greater presence of hydrates. It can be concluded that the porosity of samples with sediments is less connected than that of reference samples. This characteristic is an advantage for road materials that incorporate sediments since it gives them higher mechanical performance, brought about by more hydrates, and they are also less sensitive to intrusion of aggressive agents because of their lower permeability.

#### *Differences between percolated/non-percolated samples*

BET adsorption methods and mercury porosimetry were also used to characterise STLH samples before and after percolation. Taking the standard deviation measured on various samples into account, no significant changes were seen. Applying the percolation test until an L/S of 10 L/kg was reached did not seem to have any great impact on the porous medium structure.

The results obtained on ST0 samples by TGA showed three main phenomena: between 30 and 300°C, water departure; between 420 and 480°C, presence of portlandite; between 500 and 750°C, decomposition of carbonates. These phenomena were detected for samples whether or not they had been subjected to the percolation test, although the quantity of portlandite

seemed to be lower in percolated samples (0.4 % for non-percolated samples and 0.2 % for percolated ones) as has already been shown in other works [24]. This shows that portlandite dissolved (as expected) since it is the most soluble hydration product. The analysis of the precipitate found in the percolates in contact with air throughout the test confirmed this result [7]. It can also be concluded that, even after percolation, portlandite is still present. This could explain results from other studies [7] that have demonstrated that the pH values of the percolates decrease only slightly during the test and that the values remain basic (between 11.8 and 12.1) at an L/S of 10 L/kg.

Scanning electron microscopy (SEM) was also used. This method again confirmed the presence of portlandite in ST0 and SS3 samples that had undergone the percolation test (see Figure 5a).

On percolated samples and particularly for ST0, a “new formation” was observed (see Figure 5b). This usually appears on samples that have been subjected to water movement. However, its “airy” structure prevented its analysis. It was less easy to detect on SS3 samples, possibly because of their more dense structure compared to that of ST0.

#### ***Comparison with a standard leaching test***

To make this comparison, results from the percolation test had to be presented as cumulated values. The cumulated release was calculated by assuming that the concentration measured on a percolate was constant from the previous one. The unit ‘mg/kg’ was more suitable for comparing results from the two tests since we wanted to evaluate the release for different elements [28]. Only the results for zinc, lead and chromium are presented in Table 5. The cumulated release for the percolation test ‘P’ was compared to the results of the leaching test ‘L’ by calculating the ratio L/P. If this ratio was lower than 100%, the percolation test was more sensitive than the leaching one; such ratios are in bold in Table 5.

The results show that the percolation test is more sensitive to the release of the heavy metals considered, except for chromium in STLH. Although the L/S and the nature of the fluid were similar between the two tests, we can conclude that the granular state of the material seems to have non-negligible consequences on the release. It seems important to keep the monolithic character of road materials if their environmental behaviour is studied. Crushing them implies changes and the release of some heavy metals can be over- or even under-estimated, which could lead to an incorrect assessment.

## **Conclusion**

In order to respect the scenario for reusing wastes and to mitigate the lack of percolation tests for monolithic materials, a new percolation test has been put forward in this study.

The device used was a permeameter and, in particular, it allowed us to work with monolithic materials and with renewal of the fluid during the test. Fluid under pressure was forced to go through road materials. This way of working was chosen to accelerate the phenomena in the laboratory without using conditions too different from actual practice. Many boxes were developed in order to adapt the shape of the sample according to its permeability and also to use a size that was representative. The measurement of the flow and the chemical composition of the percolates provided better knowledge of the environmental behaviour of the road material. A study case has been presented on monolithic road materials with and without granular wastes. Downward axial percolation with demineralised water was applied to hydraulic-binder-treated gravels and sands. The results are repeatable and show that a steady flow can be reached if the pressure of the fluid is adapted to the kind of samples. Thanks to this test, the permeability can also be estimated and the results show that including sediments as a partial replacement of the sand leads to lower permeability.



The road samples were characterised using BET adsorption, mercury porosimetry, thermogravimetric analysis and SEM, and links have been shown with percolation results. Portlandite passed into solution only partially during the test, which is why the pore structure did not seem to be affected and the pH values remained so basic. Comparison with a standard leaching test also using demineralised water and an L/S ratio of 10 L/kg was made by calculating the cumulated release for the percolation test. Differences were shown in the quantity released and the percolation test was more sensitive than the leaching test except for chromium in STLH.

Taking the recommendations of the EN 12920+A1 [1] standard into account implies respecting the scenario for use. The advantage of the percolation test proposed in this study is that it comes close to this scenario in the laboratory. Even if it is not yet a standard laboratory test, it seems to be promising for studying the release of many kinds of materials.

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## Figures

*Figure 1: Experimental device of the percolation test [6]*

*Figure 2: (a) Circuit diagram of the percolation box for monolithic samples - axial percolation configuration (inspired by Rabreau et al.[5] and Nguyen[6]) – (b) photo of the percolation box [6]*

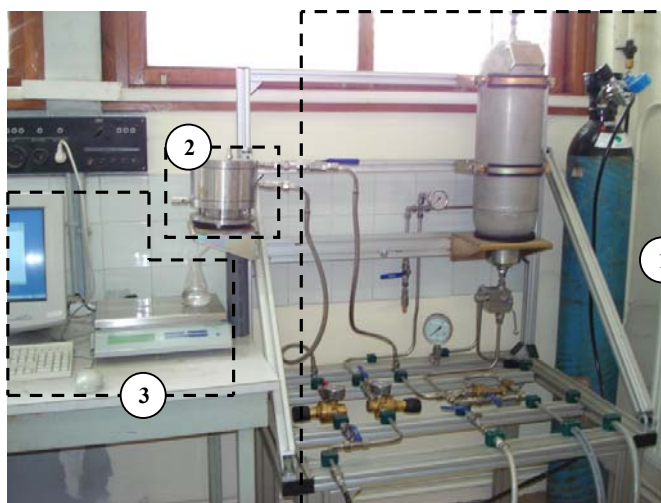
*Figure 3: Photos of samples prepared for percolation; hydraulic-binder-treated sand on the left and gravel on the right*

*Figure 4 : Mercury porosimetry curves for STLH*

*Figure 5: SEM photos of STLH samples*

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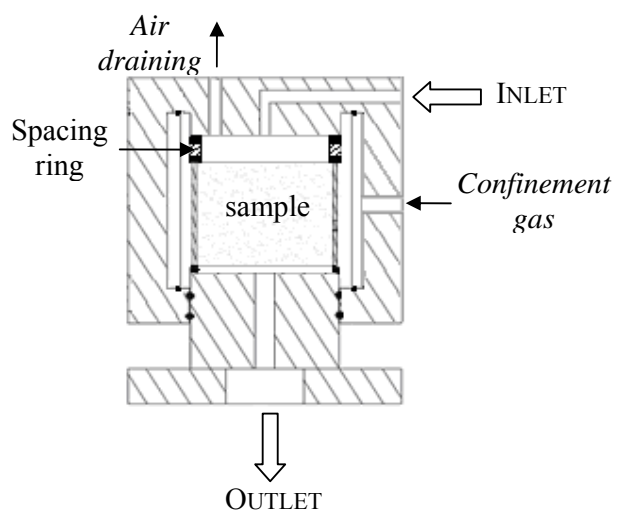
Figure 1.



- (1) pressure generator (0-40 bars);
- (2) box containing the sample;
- (3) device to collect the fluid released from the box.

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Figure 2.



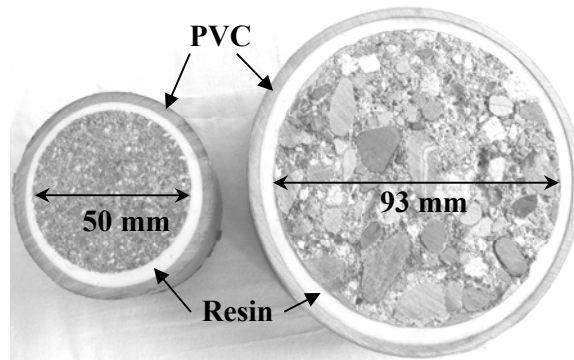
(a)



(b)

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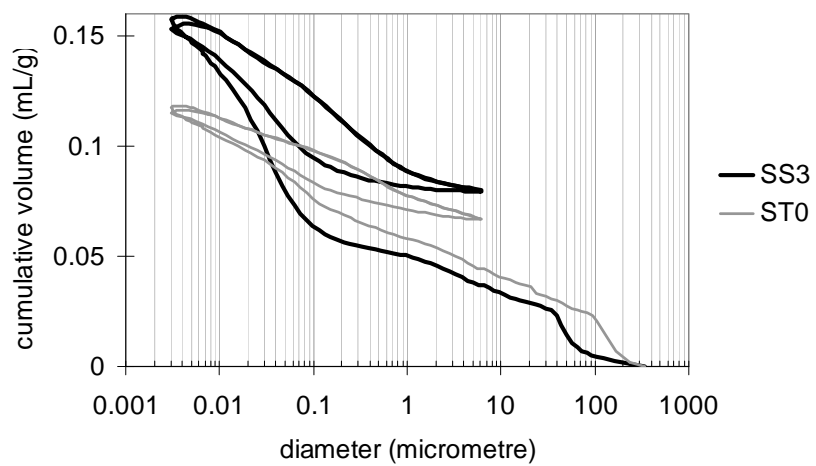
Figure 3.



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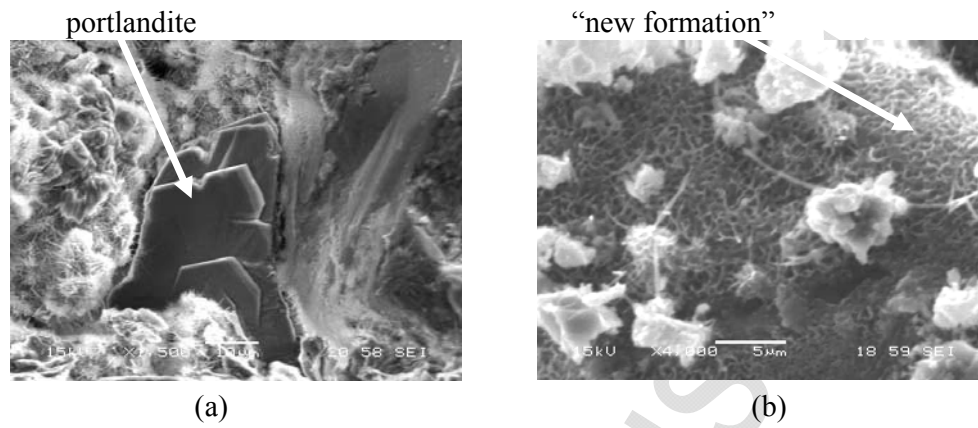


Figure 4.



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Figure 5.



Tables

(%)	0/2 sand	0/2 sediment	cement	Water content	Dried true density (g/cm <sup>3</sup> )
<b>ST0</b>	85.1	-	6.4	8.5	2.051
<b>SS3</b>	55.4	26.4	6.2	12.0	1.898

Table 1: Composition of STLH

(%)	0/4 sand	4/10 gravel	10/20 gravel	sediment	cement	Water content	Dried true density (g/cm <sup>3</sup> )
<b>GT0</b>	37.7	26.3	26.3	-	3.8	5.9	2.344
<b>GS15</b>	23.1	25.8	25.8	13.8	3.7	7.8	2.253

Table 2: Composition of GTLH

STLH		GTLH	
ST0	SS3	GT0	GS15

Saturation pressure ( $10^4 Pa$ )	1	3.5	100	100
Percolation pressure ( $10^4 Pa$ )	1	3.5	220	220
Average flow (mL/h)	35	20	97	40
Time to reach L/S=10 L/kg (days)	2	4.1	4.4	10.9
Permeability ( $10^{-16} m^2$ )	220	36	0.81	0.34

Table 3: Results of the percolation test

	<b>ST0</b>	<b>SS3</b>	<b>GT0</b>	<b>GS15</b>
Porosity (%)	23.9	30.0	14.6	18.3
Standard deviation	0.4	0.0	0.1	0.4

Table 4: Porosity accessible to water for the road materials studied

<b><math>\mu\text{g/kg}</math></b>	<b>ST0</b>			<b>SS3</b>			<b>GT0</b>			<b>GS15</b>		
	<b>P</b>	<b>L</b>	<b>L/P (%)</b>	<b>P</b>	<b>L</b>	<b>L/P (%)</b>	<b>P</b>	<b>L</b>	<b>L/P (%)</b>	<b>P</b>	<b>L</b>	<b>L/P (%)</b>
Cr	24	189	778	105	175	166	577	110	<b>19</b>	1469	135	<b>9</b>
Pb	30*	30*	<b>100</b>	60	30*	<b>50</b>	1937	30*	<b>1</b>	7854	30*	<b>0</b>
Zn	40	31	<b>77</b>	66	2*	<b>3</b>	167	2*	<b>1</b>	530	2*	<b>0</b>

\* : below detection limit

Table 5: Comparison of the release obtained from the percolation and the EN 12457-2 tests