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Effects of a physico-chemical treatment of a dredged sediment on its ecotoxicity after discharge in laboratory gravel pit microcosms

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ABSTRACT

In France, dredged sediments may be dumped into submerged gravel pits. As a consequence, adverse effects may be expected. In addition, groundwater quality may be impacted due to hydraulic communications with gravel pits. The immersion of dredged sediments into gravel pits should thus be restricted to clean or slightly contaminated sediments to minimize the impacts on aquatic ecosystems and human safe. For highly contaminated sediments, alternatives may be treatments aiming at removing or/and neutralizing contaminants. The Novosol® treatment was aimed at neutralizing metals by complexation with orthophosphoric acid and discarding organic pollutants by calcination. The efficiency of the Novosol® treatment was assessed in a scenario of sediment immersion into experimental laboratory gravel pits (LGP). A 180 L water compartment was set up in each system so as to simulate the gravel pit, and various living organisms were introduced. Following a period of colonization and stabilization, raw and treated sediments were introduced into two different LGPs, and the fate and effects of pollutants were studied during the period of deposition and post-deposition. The treatment had positive effects on survival and development of benthic populations and reproduction of pond snails but the introduction of the treated sediment was followed by an increase in salinity (phosphates, sulphates) and a peak of hexavalent chromium at concentrations above drinkability limits and likely to have impaired invertebrate populations of the water column.

The results of this study suggest that discharge of contaminated sediments at a high solid:liquid ratio (1:10) in gravel pits or equivalent aquatic ecosystems may have only limited effects on biota and ground water quality. The Novosol® treatment should, however, be improved so as to increase efficiency of oxidised chromium complexation during the phosphatation step.

1. Introduction

In France, 2.5–3.5 millions of tons of sediments are annually dredged from rivers and canals [1–4]. Dredged sediments of navigation canals are often contaminated wastes which pose a threat for the environment when they are disposed in unsafe conditions. Most sediments are simply stored on soils and exposed to rain precipitations, which generates a production of percolates with impacts on soil ecosystems, groundwater as well as surface aquatic ecosystems [5,6]. Other modes of disposal are sometimes used, such as disposal of dredged sediments in submerged gravel pits [7–9]. Important quantities of dredged sediments might also be valorized for the building of dikes and banks in rivers, canals and lakes. Submerged gravel pits, in relation with groundwater, are often colonized by an aquatic biocenosis finding good conditions for life. Moreover, human activities such as fishing, develop in such artificial lakes. When dredged sediments from navigation channels are deposited into such aquatic ecosystems, adverse effects may be expected [9], and risk assessment based on ecotoxicological bioassays is needed. Risks for water supply resource must also be addressed through assessment of groundwater contamination from the deposited materials. As a matter of fact, since groundwater flows through the gravel pit, contaminants may be leached out and diffuse into groundwater downstream the pit, and finally reach a catchment of undergroundwater.

As a consequence, the immersion of dredged sediments in submerged gravel pits or their valorization in aquatic scenarios should be restricted to clean or slightly contaminated sediments to minimize the impacts on the concerned aquatic ecosystems and on human health. For highly contaminated sediments, alternatives may be physico-chemical treatments aiming at removing or/and neutralizing contaminants, such as the Novosol® treatment pro-

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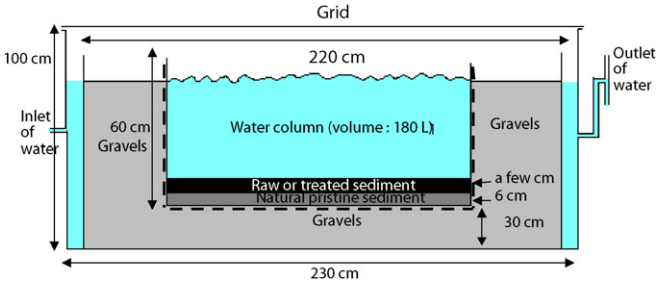

Fig. 1. Outline of one laboratory gravel pit.

cess. This treatment, proposed by the Belgian chemical company SOLVAY, is based on three steps: (i) a phosphatation step consisting in complexing metals under the form of hydroxiapatites by addition of orthophosphoric acid, (ii) a drying step where sediment water is removed in the ambient air, and (iii) a calcination step where organic contaminants are removed by oxidation in an oven at 650–900 °C.

The objective of this study was to assess the efficiency of the Novosol® treatment in a scenario of sediment immersion into experimental laboratory gravel pits (LGP), i.e. 1 m^3 tanks filled with gravels through which water flew continuously. A 180 L water compartment was set up in each system so as to simulate the gravel pit, and various living organisms (cladocerans, benthic invertebrates, gasteropods, aquatic plants, microalgae) were introduced. Following a period of colonization and stabilization, non-treated and treated sediments were immersed into two different LGPs, and the fate and effects of pollutants were studied during the period of deposition and post-deposition.

2. Materials and methods

2.1. General design of laboratory gravel pits

The study involved three different laboratory gravel pits (LGP). The first one (called G1) was filled with pristine lacustrian sediment and received in a second step raw contaminated sediment (called RS). The second one (G2) was filled with the same lacustrian sediment and then received treated sediment (called TS), resulting from the physico-chemical treatment of RS. The third one (G3) received only the same pristine lacustrian sediment as the other LGPs, and was used as the reference laboratory gravel pit.

Each LGP (Fig. 1) was made of inox steel rectangular tank (dimensions: 2.30 m length, 0.50 m width, 1.00 m height) divided into three compartments:

- a central compartment (2.20 m length × 0.50 m width) filled with material mimicking gravels found in gravel pits; inside this compartment, a zone of 1.50 m length × 0.30 m width × 0.60 m height was not filled with gravels and received an inox steel grid of same dimensions of mesh size 1 mm. This grid was filled with pristine lacustrian sediment (6 cm height) and delimitated a volume of water of 180 L. A microcosm was set up in this water volume to mimic the aquatic ecosystem of gravel pits.
- two lateral compartments (0.10 m length × 0.50 width) receiving only water to ensure mixing of input and output water, separated from the central compartment with a rectangular grid of mesh size 1 mm.

Each LGP was continuously supplied with modified tap water (see details hereafter) at a flow rate of 5 L/h. This flow rate corresponded to a groundwater flowing at a speed of ca 112 m/year, a value lower than values found for alluvial grounwaters of rivers such as Rhône and Rhin (1–2 km/year). Note that this very low speed, equivalent to 0.013 m/h, corresponds to lentic ecosystems such as gravel pits. The output water was discharged in the wastewater network.

Table 1
Physico-chemical composition of raw (RS) and treated (TS) sediments.

Sediment	RS	TS
Water content (%)	64.5	0.6
TOC (%)	5.9	<0.1
Mean diameter (μm)	27.0	57.6
% particles <2 μm	5.8	3.3
% particles from 2 to 20 μm	40.5	20.1
% particles from 20 to 50 μm	20.4	18.2
% particles from 50 to 200 μm	19.1	37.9
% particles from 200 to 2000 μm	14.2	20.5
PAHs mg/kg dry weight		
Acenaphtene	0.19	<0.05
Acenaphtylene	<0.1	<0.1
Anthracene	0.63	<0.03
Benzo (a) anthracene	1.06	<0.03
Benzo (a) pyrene	0.97	<0.03
Benzo (b) fluoranthene	2.22	<0.03
Benzo (k) fluoranthene	0.65	<0.03
Benzo (ghi) perylene	0.92	<0.05
Chrysene	1.48	<0.03
Di benzo (a,h) anthracene	0.37	<0.05
Fluoranthene	2.56	<0.1
Fluorene	0.43	<0.05
Indeno (1,2,3-cd) pyrene	0.91	<0.05
Naphtalene	0.15	<0.1
Phenanthrene	2.62	<0.03
Pyrene	2.35	<0.03
Sum of 16 PAHs	17.51	0.79
Metals mg/kg dry weight		
Arsenic	10.9	12.0
Cadmium	6.8	7.1
Chromium	231.6	216.4
Copper	213.2	172.3
Mercury	1.5	0.22
Nickel	257.4	286.4
Lead	300.7	323.0
Zinc	2147.1	2393.0

2.2. Characteristics of gravels and sediments

The gravels were natural non-contaminated materials of diameter 10/20 mm, ensuring a water permeability of 10^{-7}–10^{-8} m s^{-1}. It was stored outside for a few weeks and washed by the rain.

The pristine sediment was collected in the Lake of Aiguebelette (Savoie, France), on the first 20 cm. Its physico-chemical composition is summarized in Table 1. This sediment was previously used as a reference sediment in microcosm tests [10–13].

The raw contaminated sediment (RS) was collected in July 2004 in a canal located in the north of France (Wasquehal, Lille, sampling station referenced as no. 17000 by Voies Navigables de France, the public company in charge of the management of the navigation network). The canal is no longer used for navigation, due to accumulation of a highly contaminated sediment which should be dredged in the future. RS was stored at 4 °C until treatment.

The treated sediment (TS) was the result of the treatment Novosol® of RS. The treatment was carried out by SOLVAY in September 2004. Both sediment were stored (at 4 °C for RS and at ambient temperature for TS) until their immersion into the LGPs in May 2005. From September 2004 to January 2005 they were used in other biological and physico-chemical assays.

2.3. Characteristics of water

The water used to fill and continuously supply the LGPs was tap water filtrated on activated carbon in order to remove chlorine, and enriched with nutrients (N, P). The filtrated tap water was collected

in a reservoir where a nutrient solution (NH_4NO_3 (2.915 g L^{-1}), KH_2PO_4 (0.3315 g L^{-1}) was continuously added using a peristaltic pump (Minipuls 3, Gilson) so as to obtain a constant concentration of 1.352 mg N L^{-1} and 0.2 mg P L^{-1}. Then the stored water was distributed *via* a peristaltic pump (Masterflex® L/S Easy-Load model 7518-00) to the three GLPs at a flow rate of 5 L/h/GLP. The water in the reservoir was continuously aerated using pipet Pasteurs collected to an air pump. The water entered the GLP through a 6 mm tube located in the center of the upstream side at 45 cm from the bottom, and went out through 6 mm tube located in the center of the downstream side at 75 cm from the bottom. A siphoning device allowed to stabilize the level of water inside the GLPs. Pore waters inside the microcosms were collected using a U-shape glass tube equipped with a sintered material at its lower end deepened into the sediment. At the upper end a plastic tube allowed to aspire the pore water. One tube was connected to the lacustrian sediment, another one was connected to the raw or treated sediment.

2.4. Biota of gravel pit ecosystems

Various organisms representing different trophic levels (primary producers and primary consumers) were introduced in the gravel pits: microalgae (*Chlorella vulgaris*, *Pseudokirchneriella subcapitata*), cladocerans (*Daphnia magna, Ceriodaphnia dubia, Simocephalus vetulus*), amphipods (*Hyalella azteca*), chironomids (*Chironomus riparius*), duckweeds (*Lemna minor, Spirodela polyrhiza*), rooted macrophytes (*Elodea canadensis, Myriophyllum spicatum*), gasteropods (*Limnaea stagnalis*, *Physa acuta*).

Cladocerans, amphipods and chironomids were reared in groundwater. Cladocerans (20 individuals for *Daphnia magna* and 40–50 for *Simocephalus vetulus*) were kept in 1 L glass flasks at 19–20 °C under low illumination; a mixture of *Pseudokirchneriella subcapitata* and *Chlorella vulgaris* (10^6–10^7 cells/daphnid) was added to each daphnid culture every other day for food. Amphipods (*Hyalella azteca*) and chironomids (*Chironomus tentans*) were maintained in 3 L plastic aquaria at 22–23 °C under low illumination. They were fed twice a week with TetraMin® (around 1 mg/individual/d). The sediment for chironomids larvae consisted of a layer quartz sand, 3–4 cm deep. Water in the amphipod and chironomid cultures was aerated and changed partially (half volume) each week. The density of mixed-age amphipods was maintained around 100/L by discarding some of the young individuals. For chironomid cultures, the density of larvae was maintained around 100/L by depositing a new egg mass when emergence started to decline.

Pseudokirchneriella subcapitata, a green algae, was cultivated under 72 μE m^{-2} s^{-1} at 21–22 °C in oligo L.C. medium [14]. The medium and flasks were autoclaved before re-inoculation and the medium was bubbled with air to keep cells in suspension and provide carbon dioxide.

A stock culture of duckweeds (*Lemna minor*) was maintained under axenic conditions by transferring 6 two-frond colonies into 250 mL conical flasks containing 150 mL of modified Hoagland culture medium [15] every 14 d. The plants were cultivated at 22–23 °C under a light intensity of 72 μE m^{-2} s^{-1} supplied by daylight fluorescent tubes. The *Lemna* cultures were kept in an air-conditioned room.

The macrophyte *Elodea canadensis* was sampled in the outdoor 4 m^3 microcosms of Université de Savoie (Pr Gérard Blake) and maintained a few weeks before the start of test in laboratory aquariums with enriched ground water used for several cultures of invertebrates. The macrophyte *Myriophyllum spicatum* was kindly provided by Ute Feiler who uses it in sediment tests [16].

The gasteropod *Limnaea stagnalis* was sampled in river Ain and cultured in the lab a few weeks before the start of test. Snails were maintained in the local ground water and fed with fresh lettuce. *Physa acuta* was collected in the outdoor microcosms of Université de Savoie and maintained in similar conditions.

All organisms developed freely in the system, excepted the gasteropods *Limnaea stagnalis* which were maintained in a lateral compartment separated from the main compartment by a piece of mosquito net, allowing the flowing of water and small organisms. This was necessary to prevent *Limnaea* from feeding rooted macrophytes, while *Physa* was grazing microalgae attached on walls and plants but did not damage rooted macrophytes. Lettuce was brought on each other day to the gasteropod *Lymnaea*. Grounded fish food flakes (TetraMin) were also brought each other day to the benthic invertebrates which could not grow in the system feeding on the sole sediment.

Light energy was provided by fluorescent tubes placed above the systems and delivering 2000 lux 12 h/d. The temperature of the laboratory was kept constant at 20 ± 1 °C.

2.5. Schedule of operations

The whole assay lasted 140 days. The three LGPs were used as reference systems, with only pristine sediment, during 70 days, in order to let the populations colonize the ecosystems. First the systems were left 35 days without any organism inoculation, so as to reach stable physico-chemical conditions. On day 35, organisms were added. The populations developed on the three non-contaminated systems during 35 days. On day 70, 18 kg of sediment RS and 18 kg of sediment TS were immerged in the water of respectively G1 and G2, using a sieve to ensure uniform distribution of the sediment over the whole surface of the pit, while G3 received no sediment and was used as reference system in the following of the assay. The impacts of sediment immersion were monitored during 70 d.

2.6. Measurements

Granulometric analyses of sediments were carried out using a laser granulometer (Coulter LS130) at the Laboratoire de Génie Electrique et Ferroélectricité of INSA de Lyon. The Total Organic Carbon contents were determined at the LAEPSI of INSA de Lyon with a carbon analyzer OI Analytical, following the ISO standard 10694 [17]. Sediment PAHs analyses (16 priority PAHs of US-EPA) were carried out by SGS WOLFF Environnement according to the standard XP X 33-012 [18], by HPLC with fluorimetric and UV detection for the treated sediment, and by GC/MS for the treated sediment, following an accelerated extraction step (ASE) in a mixture acetone/hexane. Sediment PolyChloro-Biphenyls (PCBs, congeners PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180) were dosed by SGS WOLFF Environnement according to the standard XP X 33-012 [18] by GC/MS following an accelerated extraction step (ASE) in a mixture acetone/hexane.

The mineral elements of sediments were dosed in LAEPSI by ICP-AES (Inductively Coupled Plasma Atomic Emission Spectrometry) applied after mineralisation carried out with the standard NF ISO 11466 [19]. The measured elements were Ag, Al, As, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, Hg, In, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, Si, Sn, Sr, Th, Tl, V, Zn.

The monitoring of water quality was adapted to the type of water (see Fig. 1):

- Input (enriched) water in the reservoir, upstream the GLPs: pH, conductivity, $\%O_2$, temperature, cations and anions contents (NH_4^+, Na^+, K^+, Ca^{2+}, Mg^{2+}, NO_3^-, PO_4^{3-}, Cl^-, SO_4^{2-}), DOC,
- Microcosm water and output water: pH, conductivity, $\%O_2$, temperature, cations and anions contents (NH_4^+, Na^+, K^+, Ca^{2+}, Mg^{2+}, NO_3^-, PO_4^{3-}, Cl^-, SO_4^{2-}), heavy metals contents (Cr, Cr(VI), Cu, Ni, Zn), DOC, SSM,

- Interstitial water: pH, conductivity, cations and anions contents (NH_4^+, Na^+, K^+, Ca^{2+}, Mg^{2+}, NO_3^-, PO_4^{3-}, Cl^-, SO_4^{2-}), heavy metals contents (Cr, Cr(VI), Cu, Ni, Zn), DOC.

Conductivity, temperature, pH and dissolved oxygen were measured using portative electrodes (pIONneer 65, Radiometer Analytical SAS, France).

Heavy metal contents were measured on filtrated waters (Whatman, GF/C, mesh size 1.2 μm) acidified with nitric acid at pH 2 and stored at 4 °C in polyethylene flasks until measurement. The analytical method was ICP-AES [20]. For Cr(VI) content, the HACH method was used.

Anions and cations contents were determined on filtrated waters (Whatman, GF/C, mesh size 1.2 μm) stored at 4 °C no longer than one-week before analysis. Ammonium, nitrate and phosphate contents were measured using HACH methods.

Suspended Solid Matters (SSM) were measured by filtrating waters (Whatman, GF/C, mesh size 1.2 μm) and measuring weight difference after drying (100 °C) of filter and constant weight.

Dissolved Organic Carbon on filtrated waters (Whatman, GF/C, mesh size 1.2 μm) was measured using the standard NF EN 1484 [21] on waters acidified with orthophosphoric acid at pH < 2 and stored at 4 °C in polyethylene flasks no longer than 1-month before analysis.

The biota was monitored as followed. Algal growth in the water column was measured through chlorophyll *a* content determined using the trichromatic method [22]. Duckweed growth was assessed by regular counting of fronds and colonies. Rooted macrophytes growth was measured via the number of inter-nodes, the length of shoots and, at the end of test, the dry mass of plants. Cladocerans were counted twice a week after collection by siphoning the water column. All individuals were reintroduced into the system so as to follow the development of populations. Amphipods were collected at the end of test by sieving the upper part of sediment (1 cm). Chironomid imagos were trapped during the assay under a cap made of mosquito net placed above each LGP. The cap was removed during sampling and measurements which took a few hours per day, and then placed again. Chironomid larvae were collected at the end of test by sieving of the upper part of sediment (1 cm). Gasteropods were individually counted once a week. *Lymnaea* gasteropod egg masses were collected every other day, number of eggs were counted, egg masses were transferred into flasks containing clean water in order to measure the rate of eclosion. Shell length of *Lymnaea* gasteropods was measured each week so as to assess growth. The consumption of food by *Lymnaea* gasteropods was determined by difference between final and initial dry masses of lettuce. At the end of test, all molluscs were dried and their mass was determined.

Acute (48 h survival of neonates) and chronic (21 d reproduction) toxicity tests on *Daphnia magna* were carried out in parallel of microcosm tests on surface and ground waters of LGPs. Waters tested in the reproduction test were sampled in LGPs from day 1 to day 22. The aim was to consolidate ecotoxicological assessment obtained in a complex ecosystem submitted to a sudden stress by simple standard methods.

3. Results

3.1. Physico-chemical characterisation of sediments

The granulometric analysis showed fine sediments (Table 1). The treatment generated a higher proportion of particles >20 μm and a mean diameter multiplied by 2.

The raw sediment was highly contaminated with organic compounds (PAHs) and heavy metals (Table 1). The main metals, apart from major elements such as iron, aluminium and magnesium, were Zn, Pb, Ba, Mn, Sr, Cr, Cu, Ni, Cd. The treatment removed almost 100% organic compounds but conserved the total contents of heavy metals, whereas phosphorous content was multiplied by 4, due to the phosphatation step.

3.2. Innocuousness of microcosm water

Due to a higher availability, tap water was used for the continuous feeding of GLPs, and was consequently the water compartment of microcosms. Although this water was aerated and dechlorinated by filtration through activated carbon, its quality as a medium for life and its innocuousness had to be controlled by means of bioassays with the cladoceran *Daphnia magna*, the amphipod *Hyalella azteca* and the green microalga *Chlorella vulgaris*.

Daphnia magna reproduced slightly less in tap water than in the ground water used for daphnid rearing (25 neonates per mother in tap water vs. 35 neonates per mother in groundwater, on average, within 21 days). The 14 d survival of *Hyalella azteca* in water above reference sediment was almost 100% in both waters, and their size was similar (2.50 ± 0.25 mm in tap water vs. 2.31 ± 0.27 mm in ground water). A 72 h growth assay with *Chlorella vulgaris* showed that enriched tap water (1.3 mg N/L and 100 μg P/L) was convenient for algal growth (3.2×10^6 cells/mL vs. 4.5×10^6 cells/mL for synthetic algal medium, after 72 h and an initial density of 50 000 cells/mL). The addition of nutrient was necessary since non-enriched tap water did not support algal growth (2.5×10^5 cells/mL).

These bioassays show that, although enriched tap water could not be considered as an optimal medium for life, it was sufficient for high survival of organisms and reproduction or growth.

3.3. Physico-chemical equilibration of LGPs

Physico-chemical characteristics of water column (noted EGi) of LGPs and downstream groundwater (noted ESi) of LGPs (water collected at the outlet of LGP, see Fig. 1) were measured during the pre-contamination phase (from day −40 to day 0). The conductivity varied between LGPs during the first 20 days due to adjustment of hydraulic conditions and reaching of equilibrium, then was constant around 445 ± 0.58 μS/cm for all LGPs, very close to the conductivity of enriched tap water, with very slight differences between water column and groundwater.

The pH decreased quite regularly from day −28 to day 0 but reached similar values, around 7.51 ± 0.04 pH units, in all LGPs. Note that pH of enriched tap water was always around 7.8–7.9. Although enriched tap water (ETW) used to feed the systems was constantly aerated in the distribution tank, oxygen content in LGP waters decreased from 90 to 40–50% between day −20 and day 0. This was probably due to absence of aeration in the LGPs and increase of oxygen demand of sediment and gravel microbial communities. Nevertheless, this oxygen level was still sufficient for invertebrates.

The enrichment of tap water through continuous addition of N and P in the mixing reservoir prior to distribution of enriched tap water in LGPs led to a rather good conservation of total N, whereas NH_4^+ and PO_4^{3-} contents decreased more or less by flowing through the gravels (NH_4^+: 1.36 ± 0.31 mg/L in enriched tap water, 0.09 ± 0.04 in LGP water; PO4: 0.49 ± 0.08 mg/L in enriched tap water, 0.24 ± 0.044 mg/L in LGP water), probably due to retention of these nutrients and/or microbial activities inside the gravels. The resulting water quality was nevertheless suitable for growth of primary producers.

Finally, as shown by monitoring of water quality during the pre-contamination phase, the waters of LGPs reached similar and steady-state values reflecting the equilibration of systems under

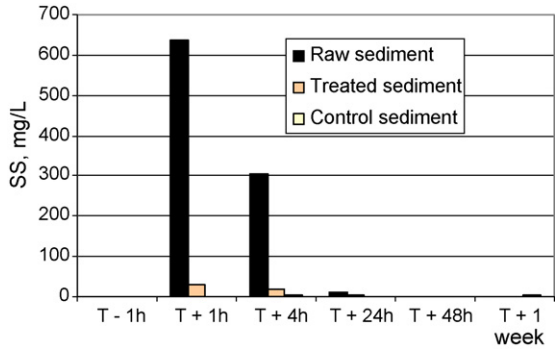

Fig. 2. Evolution of water turbidity (suspended solids content) of the three LGPs following addition of sediments.

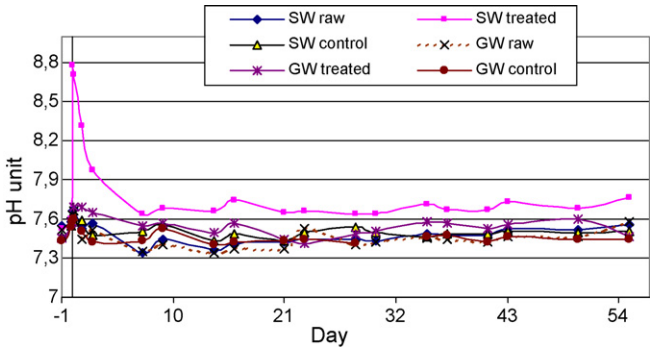

Fig. 4. Evolution of water pH following addition of raw and treated sediments (SW: water column of LGP; GW: downstream groundwater of LGP).

continuous circulation of water. Note that the quality of groundwaters downstream (GWi) was not different from that of LGP waters.

3.4. Evolution of physico-chemical characteristics of waters following sediment addition

The addition of sediments into the water compartment of LGP1 (raw sediment) and LGP2 (treated sediment) was followed by a peak of turbidity (Fig. 2) and clarification of water within 48 h. The sedimentation of materials was faster with the treated sediment, probably due to a lesser proportion of fine particles and absence of organic and colloidal matters.

Conductivity of LGPs surface waters increased following sediment addition, but the increase was far higher for the treated sediment (Fig. 3). The same pattern was observed for groundwaters but, whereas conductivity of surface water above treated sediment returned to normal values within two weeks, it remained higher in groundwater until day 40. The renewal of surface water probably explains this decrease, whereas the settled treated sediment continued to generate salinity towards interstitial and ground waters.

The increase of pH was only significant in surface water of LGP2 (treated sediment) (Fig. 4). Although a rapid decrease was observed within one-week, the pH remained significantly higher in this LGP until the end of experiment.

As expected, the addition of the raw organic sediment led to a significant decrease of water oxygen content, whereas water above the treated sediment was slightly more oxygenated than in the control LGP (Fig. 5). Moreover, we observed a positive gradient of oxygen in the treated sediment-LGP upstream to downstream (difference of 13–17% oxygen on day 1 and 2 following sediment addition). This phenomenon might be explained by chemical reactions inside water (see further).

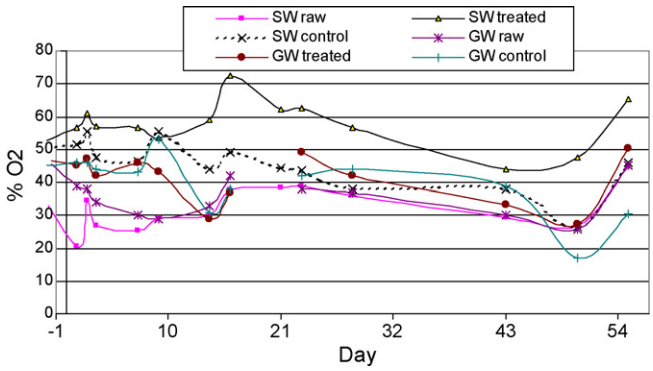

Fig. 5. Evolution of oxygen content of water following addition of raw and treated sediments (SW: water column of LGP; GW: downstream groundwater of LGP).

Analyses of metal (Cr, Cr VI, Cu, Ni, Zn) contents of filtered surface and groundwaters were carried out before and after addition of sediments. Zinc was immediately released by the raw sediment and returned to normal values, whereas the results for the treated sediment suggest a delayed release with significantly higher concentrations after three weeks (Fig. 6). Unfortunately, metals monitoring was stopped after four weeks. Anyhow, zinc concentrations were never at ecotoxic levels. More worrying was the behaviour of chromium. Almost non-detectable concentrations of total and hexavalent chromium were found in surface water above raw sediment, but total and hexavalent chromium was found at significant and ecotoxic levels in the surface water above treated sediment (Fig. 7). However, chromium concentrations had returned to non-detectable values after two weeks. The evolution of hexavalent chromium in the water column and the oxygen gradient observed upstream to downstream in the treated sediment-LGP

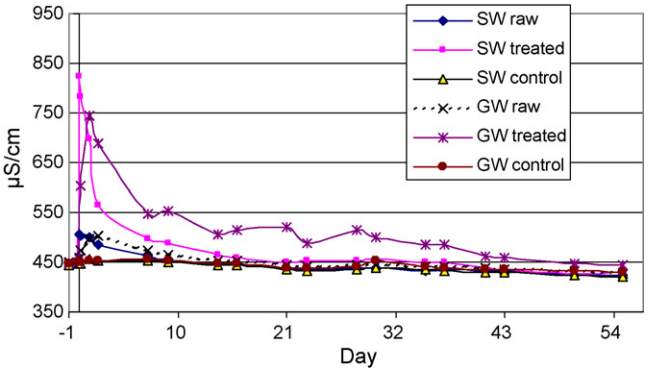

Fig. 3. Evolution of water conductivity following addition of raw and treated sediments (SW: water column of LGP; GW: downstream groundwater of LGP).

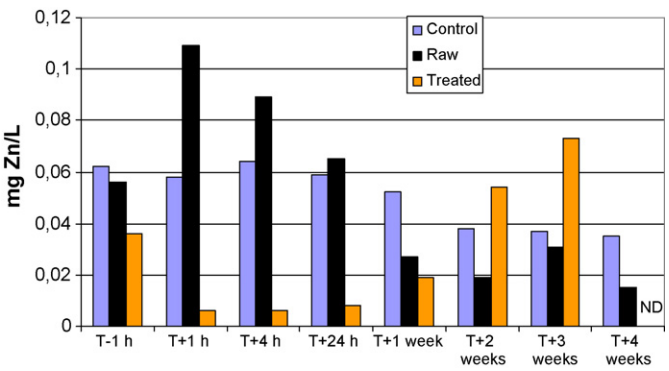

Fig. 6. Evolution of zinc content of surface water following addition of raw and treated sediments (analyses on GF/C filtered waters).

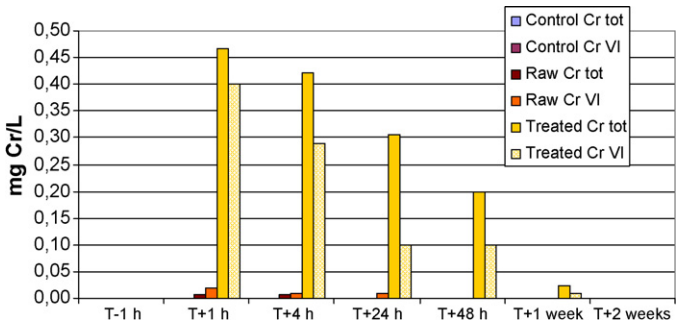

Fig. 7. Evolution of chromium (total and hexavalent) content of surface water following addition of raw and treated sediments (analyses on GF/C filtered waters).

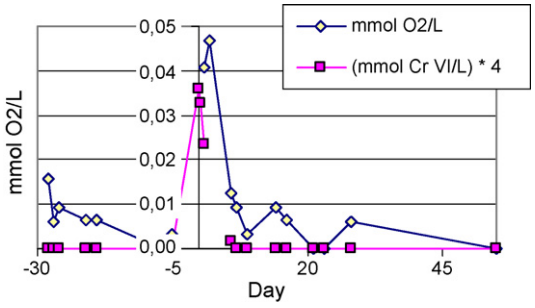

Fig. 8. Evolution of positive upstream–downstream oxygen gradient and hexavalent chromium molar concentration in water column of TS LGP.

were compared (Fig. 8). It appears that the observed production of oxygen might have been due to the reduction of Cr(VI) into Cr(III), this reduction corresponding to the production of 4 moles of oxygen for one mole of reduced chromium.

Apart from Zn and Cr, Ni was only found at trace concentrations (36 μg/L) above raw sediment, and Cu was not found.

The behaviour of metals in groundwaters (Table 2) was the same as in surface waters, with a peak around 24 h and recovery of normal values after one or two weeks.

As expected, the addition of raw sediment led to an increase of ammonia, with a peak after a few hours (Fig. 9). Maximum values (8.9 mg/L) were not considered as toxic at observed pH. A release of orthophosphates was observed with the treated sediment, which could be attributed to the phosphatation step. Return to normal conditions was observed after two weeks, although phosphate content above treated sediment was kept around 0.5 mg/L until the end of experiment. Nitrate concentrations were close to 10 or 16 mg/L in all waters, with no significant change due to sediment addition. The immersion of treated sediment led to an increase of ions (K^+, Na^+, Mg^{2+}, Ca^{2+}, SO_4^{2-}, Cl^-) of surface and ground waters, corroborated by the observed increase of conductivity, whereas the raw

Table 2
Evolution of groundwater quality (detectable metals) downstream LGPs following sediment addition ((): potability limits; DL: detection limit).

	Cr total, μg/L (50)			Cr VI, μg/L			Zn total, μg/L (5000)		
	Control	RS	TS	Control	RS	TS	Control	RS	TS
T−1 h	nd	nd	nd	<DL	<DL	0	23	25	16
T+1 h	9	12	10	<DL	<DL	10	20	140	16
T+4 h	<DL	<DL	195	<DL	<DL	110	0	20	13
T+24 h	<DL	<DL	302	<DL	<DL	210	0	16	14
T+48 h	nd	nd	nd	<DL	<DL	130	nd	nd	nd
T+1 week	<DL	<DL	35	<DL	<DL	20	15	12	9
T+2 weeks	<DL	<DL	8	<DL	<DL	<DL	10	6	4
T+3 weeks	<DL	<DL	7	<DL	<DL	<DL	15	11	21
T+4 weeks	<DL	<DL	6	<DL	<DL	<DL	6	5	9

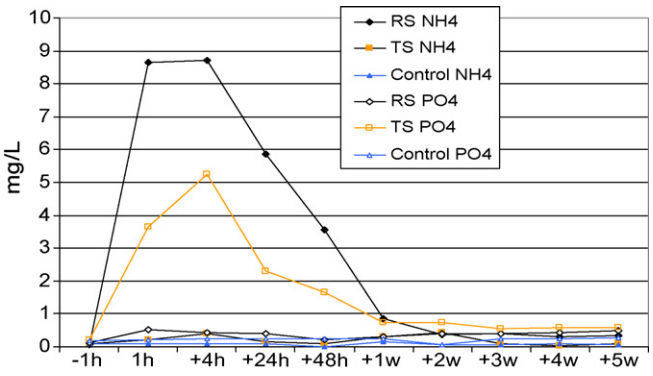

Fig. 9. Evolution of ammonia and orthophosphate contents (mg/L) of surface waters of LGPs following sediment addition (RS: raw sediment; TS: treated sediment).

sediment did not significantly modify water salinity. The release of SO_4^{2-} and Cl^- from the treated sediment was particularly significant (Table 3). The peaks were observed within the first 24 h, then concentrations decreased and returned to values similar to control values between 7 and 40 days depending on the parameter and the water.

Chemical analyses were performed on pore waters of initial (lacustrian sediment) and added sediments (TS or RS). For the RS LGP, sampling of RS pore water was not possible due to clogging of the U-shape glass tube designed to collect water. For major ions, concentrations were highest for the treated sediment (TS) and the lacustrian sediment beneath, especially for Mg^{2+}, Ca^{2+}, Cl^- and SO_4^{2-}, whereas moderate concentrations of RS lacustrian sediment indicate significant but moderate release of salts from RS.

3.5. *Effects on living populations following sediment addition*

Acute bioassays were carried out on the waters of LGPs at various times after sediment immersion. Effects on cladocerans exposed 48 h to surface waters (Table 4) were clearly significant in contaminated LGPs after 1 h and 4 h, with a higher intensity in the presence of treated sediment. Results were far less consistent after one week, with no clear effects.

Daphnid reproduction in surfac and ground waters of LGPs sampled from day 1 to day 22 following sediment immersion was not different between LGPs when considering total cumulated neonates per mother (Fig. 10). However, a significant inhibition was observed in surface water of TS during the first week of reproduction. No effect was observed in ground waters. The second reproduction test launched on the same waters sampled from day 7 to day 28 following sediment immersion showed no effect.

Daphnid populations, free in water columns of LGPs, were monitored following sediment immersion (Fig. 11). In control LGP, daphnid population developed well but started to decline on day 21, probably due to starvation. In RS LGP, the sediment immersion led to a high mortality within 48 h, which necessitated a re-inoculation (+100 individuals on day 3). Then daphnids reproduced at a high rate (around 5000 individuals after 1-month). In TS LGP, reproduction was lower, despite several re-inoculations.

Response of *Ceriodaphnia dubia* populations was quite different: whereas populations developed well in control and TS LGP, the growth of population was far much slower in RS LGP.

Hyalella azteca was sensitive to surface waters of RS and TS LGPs sampled from day 8 to day 22, with a slightly higher toxicity in TS LGP (14 d survival: controls: >80%; RS: 60%; TS: 52%), whereas no toxicity was found in waters sampled from day 27 to day 41 (survival >80%). Free amphipods *Hyalella azteca* did not develop well in LGPs. Whereas 100 individuals were introduced on day −18, only

Table 3
Sulphate and chloride contents of surface water and groundwater of LGPs following immersion of raw and treated sediment (from 1 h to 69 days).

	Groundwater						Surface water					
	Control		Raw sediment		Treated sediment		Control		Raw sediment		Treated sediment	
	Cl^-	SO_4^{2-}	Cl^-	SO_4^{2-}	Cl^-	SO_4^{2-}	Cl^-	SO_4^{2-}	Cl^-	SO_4^{2-}	Cl^-	SO_4^{2-}
T−1 h	12.1	24.3	12.4	23.6	11.8	24.0	12.4	24.4	12.7	24.7	12.8	25.0
T+1 h	12.8	25.1	13.6	26.5	12.5	28.9	14.2	25.6	12.4	26.2	21.7	169.0
T+4 h	13.4	23.4	12.8	24.8	17.0	103.7	14.2	44.6	13.2	26.2	21.0	163.9
Day 1	13.8	27.5	12.6	26.9	17.0	128.0	13.6	27.7	13.4	27.3	18.3	120.0
Day 7	13.5	28.4	nd	nd	13.5	28.4	13.1	26.8	13.7	29.4	14.6	49.0
Day 14	13.7	25.2	nd	nd	13.3	57.6	nd	nd	12.7	26.9	13.4	36.7
Day 21	13.4	29.4	13.5	30.6	13.3	73.8	13.2	29.8	12.8	29.3	13.3	36.6
Day 28	12.5	30.7	13.1	30.3	13.0	66.9	12.9	32.4	13.3	31.1	13.2	39.5
Day 41	13.4	35.0	13.8	35.3	14.7	48.3	13.3	35.5	13.9	35.6	14.0	39.4
Day 49	13.2	36.3	13.3	35.2	13.6	47.2	13.1	35.9	13.3	36.3	13.3	38.4
Day 55	13.0	38.0	13.8	47.4	13.4	48.8	13.2	39.7	12.9	35.8	12.5	40.1
Day 69	13.7	39.2	16.1	48.1	13.2	47.6	13.0	37.6	13.2	41.2	13.8	40.0

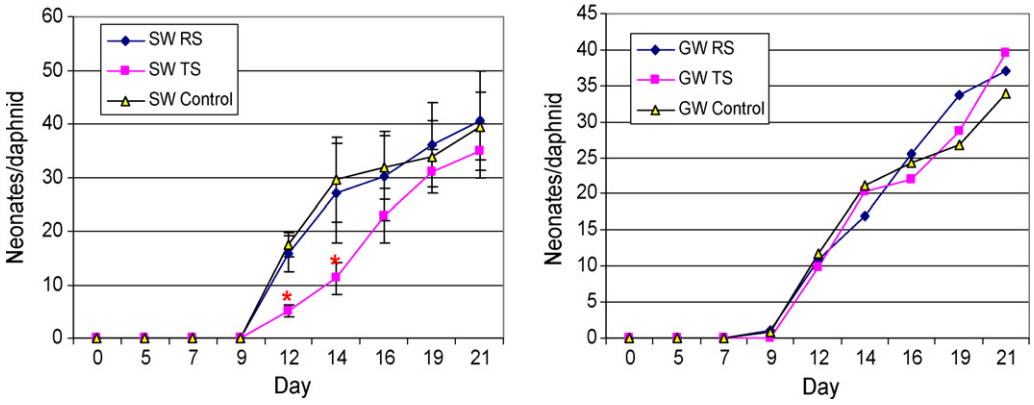

Fig. 10. Daphnid reproduction in surface (SW) and ground (GW) waters sampled from day 1 to day 22 following sediment immersion (mean ± SD error bar, $n = 10$) (RS: raw sediment; TS: treated sediment).

64 and 61 individuals were found at the end of assay respectively in control and TS LGPs. Only 22 were found in RS LGP, which indicates that the raw sediment was slightly toxic for this amphipod. So as to confirm this toxicity, *H. azteca* tests (survival and growth) were carried out on sediments collected in the LGPs after the assay. The results confirm that the raw sediment impaired survival and growth of this amphipod, which was not the case for the treated sediment (Fig. 12). Note also that pristine lake sediment initially introduced in RS and TS LGPs was safe at the end of assay, whatever the contaminated sediment the LGP received.

Among the 250 chironomid larvae introduced during the course of the assay and free in the LGPs, very few emerged (emergence rates between 2.4 and 12%). It is assumed that conditions inside LGPs were not optimal for this insect larva, due to insufficient food (fish food flakes were nevertheless regularly brought) or/and competition with other benthic invertebrates brought with the lake sediment (dipter larvae, oligochaetes, ...) or predation by ephemeropter larvae (a few individuals were observed). Like for amphipods, an emergence chironomid test was carried out on sediments collected in the LGPs at the end of microcosm assay. The results show that the treated sediment was safe for survival and emergence of chironomids, whereas the raw sediment delayed emergence (Fig. 13). As for the amphipod test, all reference sediments of LGPs were not toxic.

The gasteropod *Limnaea stagnalis,* introduced 20 days before sediment immersion, survived well in all LGPs (survival rate on the duration of assay: 80–100%). Their growth, measured as shell size, was the same in all LGPs (+17–18 mm within 80 days), and

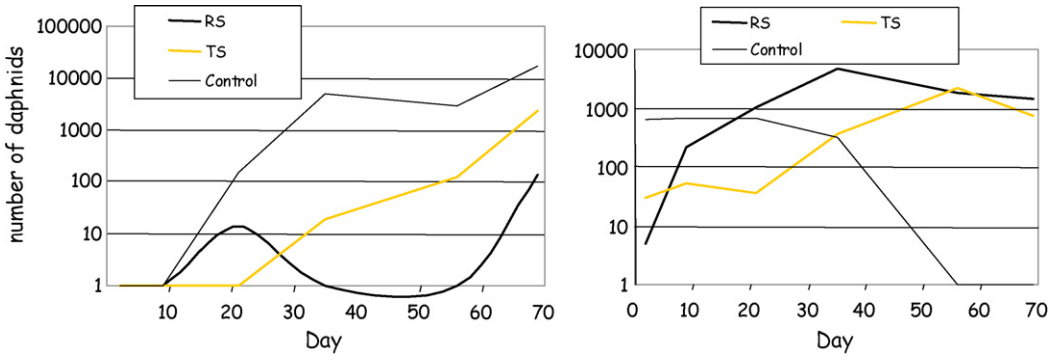

Fig. 11. Evolution of cladoceran populations (left: *Ceriodaphnia dubia;* right: *Daphnia magna*) in LGPs (RS: raw sediment; TS: treated sediment) (*y* axis: number of daphnids in log scale).

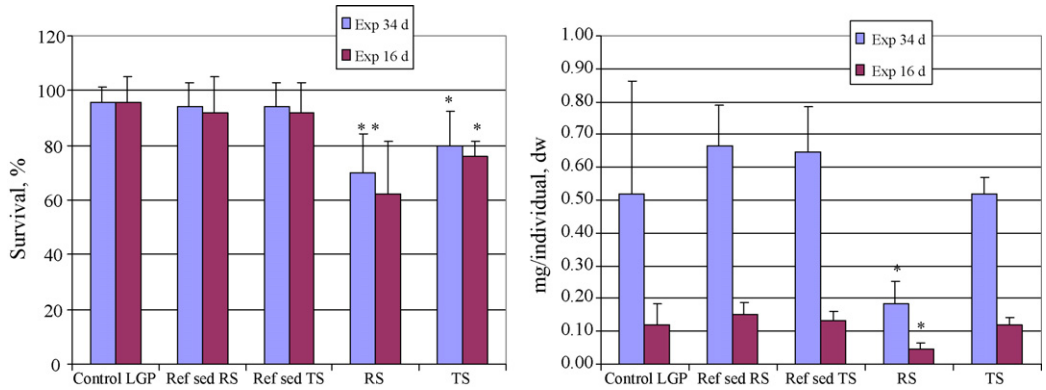

Fig. 12. Results of *Hyalella azteca* survival tests in sediments collected in LGPs at the end of assay, with exposure durations of 35 days (1st test) and 16 days (2nd test) (controls: lake sediment at the bottom of each LGP; * significantly different from the controls).

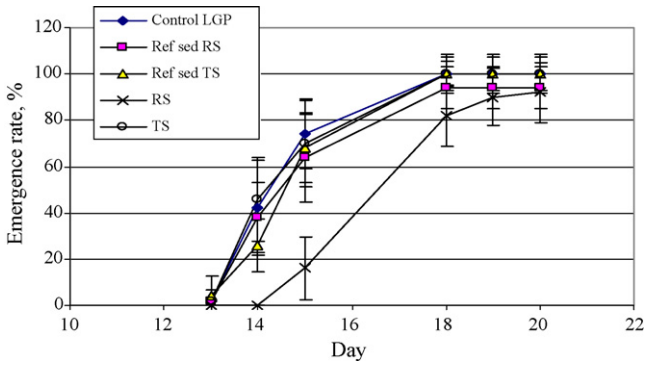

Fig. 13. Results of *Chironomus riparius* 20 d emergence test in sediments collected in LGPs at the end of assay (controls: lake sediment at the bottom of each LGP).

Table 4
Inhibition of cladoceran mobility after 48 h exposure to surface waters sampled in LGPs 1 h, 4 h and 1-week following sediment immersion (RS: raw sediment; TS: treated sediment).

	T+1 h	T+4 h	T+1 w
	Daphnia magna		
LGPcontrol	0%	5%	10%
LGP RS	10%	20%	65%
LGP TS	25%	20%	5%
	Simocephalus vetulus		
LGPcontrol	0%	0%	64%
LGP RS	0%	0%	69%
LGP TS	69%	7%	67%
	Ceriodaphnia dubia		
LGPcontrol	13%	0%	27%
LGP RS	25%	19%	7%
LGP TS	50%	44%	7%

their food consumption was also similar in all LGPs (from 10 to 20 mg dw/d at the start of test to 55–65 mg dw/d at the end). The cumulated number of egg masses per snail was nevertheless lower in the RS LGP (Fig. 14a), whereas the TS sediment showed no effect at all. When considering the cumulated number of eggs per snail, the data were still lower for the RS LGP but the difference was not so high (Fig. 14b).

The gasteropod *Physa acuta* introduced on day-19 in LGPs (10 individuals) reproduced well during the assay (Table 5). However, the immersion of sediments seems to have impaired population during the first two weeks following contamination. But, whereas

Table 5
Number of gasteropods *Physa acuta* in LGPs during the course of the microcosm assay (day0: immersion of sediments in RS and TS LGPs).

	Control LGP	RS LGP	TS LGP
Day−19	10	10	10
Day+17	140	45	57
Day+31	355	316	285
Day+56	500	530	410

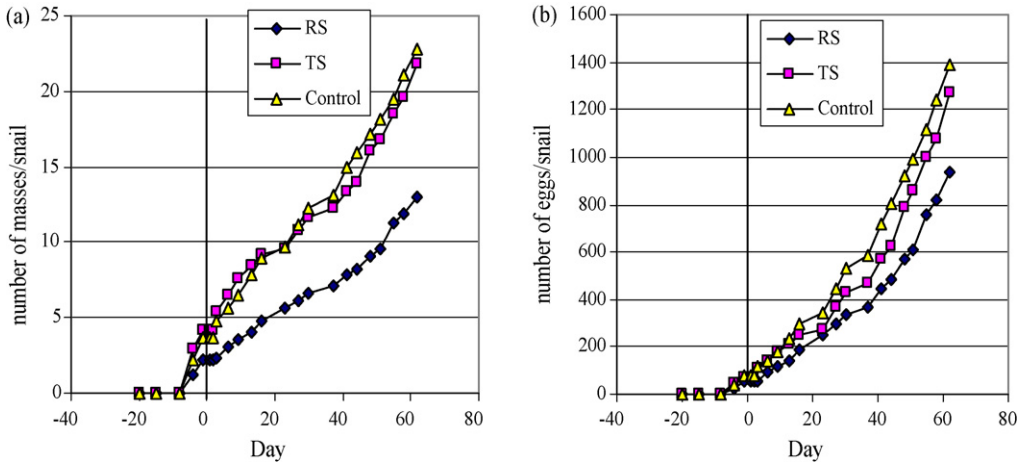

Fig. 14. Evolution of mean cumulated numbers of egg masses/snail (*L. stagnalis*) (a) and of mean cumulated numbers of eggs/snail (*L. stagnalis*) (b) (immersion of sediments on Day 0).

Table 6
Doubling time (unit: days) of frond number (species: *Lemna minor (Lm)* and *Spirodela polyrhiza* (Sp)) during the course of microcosm assay (note that on day 21 all duckweed colonies were discarded and replaced by 4 two-frond colonies).

Day	Control LGP		RS LGP		TS LGP	
	Lm	*Sp*	*Lm*	*Sp*	*Lm*	*Sp*
−39–0	10.1	6.5	10.4	8.1	11.0	10.5
0–21	11.4	8.7	8.1	7.5	16.5	12.7
23–57	7.9	17.9	5.1	6.7	86.6	200

the delay was compensated in the RS LGP, the population growth of TS LGP seemed to have been inhibited by 20%.

Floating duckweeds (*Lemna minor* and *Spirodela polyrhiza*) showed similar responses to sediment immersion. Their growth was inhibited in TS LGP but stimulated in RS LGP, as shown by higher (inhibition) or smaller doubling times (stimulation) (Table 6).

Rooted macrophytes (species: *Myriophyllum spicatum*) were introduced into the LGPs 10 days after sediment immersion. The plants grew by direct contact with sediments. Measurements of shoot length (Fig. 15a) and numbers of inter-nodes (Fig. 15b) showed that growth was higher in the contaminated sediments, with a very clear stimulation in the RS LGP.

Measurements of chlorophyll content of surface waters provided low values in all LGPs, before and after sediment immersion (<2 μg/L), despite regular re-inoculations of *Chlorella vulgaris* and *Pseudokirchneriella subcapitata*.

4. Discussion

No experiments similar to this one have been reported in the literature. There are many studies on desorption of solid phase bound pollutants from sediments suspended in water ([23–30]. A few studies focussed on the toxic effects of pollutants from sediment suspensions, most often in batch experiments where static conditions prevailed [8,31–33].

In the present experiment, sediments were added to water columns overlaying a natural sediment in a flow-through system, so as to mimic a scenario of gravel pits partially filled with contaminated sediment. According to literature results, the fate of pollutants was likely influenced by several processes acting more or less simultaneously: rapid desorption of the most labile contaminants from sediment, dilution of pore water (for the raw wet sediment), oxidation of organic matter and reduced forms such as sulphurs (for the raw sediment), immediately after sediment immersion, sedimentation of most particles during a short lapse of time (less than 24 h), continuous leaching of pollutants by water flowing through the systems, establishment of a new equilibrium between solid phase and water phases (pore water and surface water) following sediment deposition, a period during which diffusion of contaminants from the deposited sediment may continue but contamination of the water column is reduced by continuous renewal of water. As a result of all these processes, peaks followed by a decline were observed for physico-chemical parameters of the water column such as turbidity (sediment deposition), conductivity (dilution of pore water during the immersion phase, leaching of salts from the solid phase of sediments), pH for the treated sediment, metals. Although only a few contaminants were monitored, the results suggest that, due to the continuous renewal of water, the quality of water column was recovered after periods depending on the chemicals considered and on the sediment. However, the comparison of raw and treated sediments shows differences in behaviour. As expected, the immersion of raw organic-rich sediment was followed by a higher turbidity of the water column, a slight decline in dissolved oxygen due to organic matter, and a peak in ammonia. However, low variations of pH and conductivity and low emissions of dissolved metals (Zn < 0.12 mg/L, Cr < 0.02 mg/L, Ni < 0.036 mg/L, Cu < detection limit) were observed, suggesting that oxidation of organic matter and sulphurs was not sufficient to acidify the water and to enhance metals mobility, processes reported in the literature [23,30,34–37], or that free metals were rapidly sorbed by iron and maganese oxides and hydroxides [23,37]. The effects of the immersion of treated sediment on the surface water quality were quite different: a lower turbidity, a significant increase of conductivity due to emissions of chlorides, orthophosphates and especially sulphates resulting from oxidation of sulphurs during the treatment process, and leaching of hexavalent chromium. These results suggest that the physico-chemical treatment, although it eliminated organic pollutants, was not completely successful in reducing the leachability of metallic pollutants. Moreover, the phosphatation step generated excessive phosphates diffusing from the sediment during the whole duration of the assay. The calcination step was probably responsible for the oxidation of chromium which was leached out and reached concentrations of concern in the surface water of the TS LGP. This emission of chromium also impacted the quality of groundwater (water collected downstream the TS LGP after filtration through gravels), where a peak of 210 μg Cr(VI)/L was observed after 24 h, a value far above the limit value of total Cr (50 μg/L) for drinkable water [38].

Surprisingly, the introduction of the highly contaminated raw sediment did not severely impact the biota, at least at the population level. Although acute effects were observed on cladocerans during the immersion phase, the population of *Daphnia magna* grew after re-inoculation and stabilized at high number, whereas control populations finally declined after 50 days. By contrast, the *Ceriodaphnia* population appeared to be much more affected. Results regarding the epibenthic amphipod *Hyalella azteca* suggest effects on the survival of introduced individuals, and this was confirmed by a final assay on the raw sediment sampled at the end of the assay.

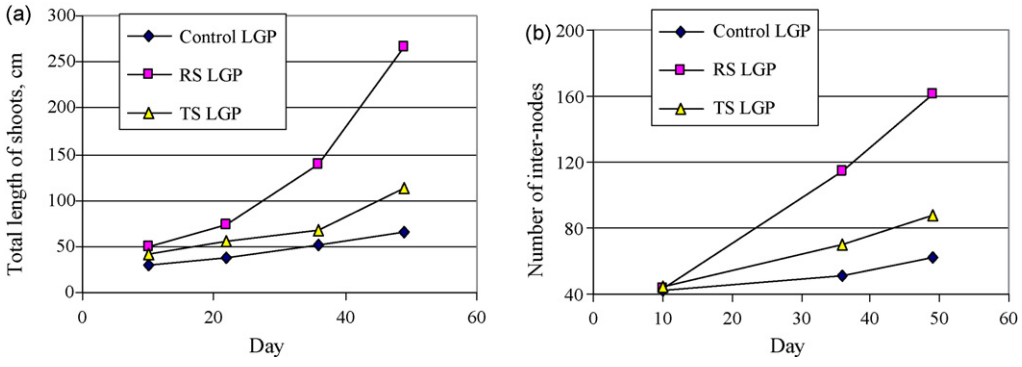

Fig. 15. Growth of *Myriophyllum spicatum* in LGPs during the assay (immersion of sediments on day 0).

The effects on chironomids could only be measured through an emergence test on the sediment at the end of assay, which showed only a delayed emergence with no effect on the total emergence rate. Moderate sublethal effects were observed on gasteropods: eggs production for *Limnaea stagnalis*, delayed reproduction for *Physa acuta* but with final numbers comparable to that of control LGP. Plant (floating duckweeds and rooted macrophytes) growth was enhanced by nutrients brought by the sediment.

Pollutant emissions from the treated sediment underlined in the physico-chemical analysis were corroborated by observed effects on various organisms. It seems that the hexavalent chromium peak could have been responsible for effects on cladoceran reproduction, although populations could finally grow after re-inoculation. Amphipods were less affected than in the raw sediment, but survival was slightly impaired at the end of assay, whereas chironomids could survive and emerge normally. Among gasteropods, only *P. acuta* population was slightly impaired with a lower final number. Surprisingly, duckweed growth was inhibited, whereas growth of rooted macrophyte was stimulated, though much lesser than in the RS LGP. Observed effects can be at least partly explained by the high concentrations of Cr(VI) observed during the first 48 h following sediment immersion, ranging between 200 and 460 μg/L. As a matter of fact, the NOEC-7d (reproduction) for *C. dubia* is 100 μg/L [39], the NOEC-21d (reproduction) for *D. magna* is 18 μg/L [40], the LC50-48 h of Cr(VI) for *D. magna* is 162 μg/L [41], the NOEC-7d (growth) for *Lemna minor* is 112 μg/L [42]. In addition, an aquatic PNEC of 1.5 μg/L has been proposed for Cr(VI) [43].

The experimental device used in this study allowed to obtain data on the behaviour of sediments immerged into aquatic ecosystems mimicking the functioning of gravel pits. The use of real gravels and natural tap water flowing continuously through those materials brought a great dose of realism in this laboratory study. Furthermore, the realism was reinforced by the use of a lacustrian sediment introduced in the water body. However, resulting conditions were not optimal for all organisms. Microalgae grew only slightly in the water column, resulting in the necessity to reinoculate microalgae several times. Since water speed was very low (0.013 m/h), and algae have the ability to settle and grow on sediment, as observed in static microcosm assays currently carried out in our laboratory, the low algal growth might be due to a limiting factor, e.g. the non-optimal composition of water. Populations of chironomids and amphipods failed to develop in the systems, though the sediment had proved its capacity for the development of benthic organisms [10–13]. Conversely, cladoceran populations, gasteropods and macrophytes (rooted plants and duckweeds) grew normally, and brought valuable informations on biological responses to sediment immersion. The lack of development of chironomids, amphipods and algae should not be attributed to the fact that these species are typically those adopted in laboratory standard bioassays, according to EPA and OECD protocols, and would not be suited to complex microcosm assays. As a matter of fact, we currently use such species in microcosm assays, either simple microcosms (volume: 2 L) [10–12] or more complex microcosms (volume: 100 L) with synthetic or ground water and artificial or natural sediment [13]. In this study, interferences might have been due to the presence of gravels, to the quality of water, or/and to the lack of organic matter for benthic organisms creating sub-optimal conditions for these species.

Due to the size of the systems, their cost and the time spent, there was no replication during the contamination phase. However, the three systems were used as replicates during the pre-contamination phase, and physico-chemical data obtained (not detailed here) showed a close behaviour of the systems. This rather high level of replicability was probably due to the continuous renewal of water which was decisive for the functioning of the ecosystems.

5. Conclusion

Undoubtedly, the physico-chemical treatment did not allow to transform a highly contaminated sediment into a pristine one which could be used totally safely in an aquatic scenario such as filling of gravel pits, or, by extension, building of banks and dikes. The immersion of the treated sediment had negative temporary and permanent effects. As temporary negative effects, we observed an increase of salinity, a leaching out of hexavalent chromium, sulphates and phosphates at high levels in the water column as well as in the groundwater, leading to acute effects on cladocerans and amphipods. Note that temporary means that effects were observed on periods from a few days to a few weeks. The permanent effects were linked to the presence of phosphates in the sediment with a continuous emission towards water column, and toxicity to duckweeds. However, positive effects should be underlined: reduction of sediment mass, materials easier to handle, reduction of visual and odour nuisances, better settability, removal of organic compounds and associated ecotoxicological risks, removal of organic matter and associated risks of anoxia, removal of ammonia emissions and associated ecotoxicological risks, removal of zinc and nickel emissions, reduction of sediment toxicity to amphipods and chironomids. Improvement of the treatment process, especially the phosphatation and calcination steps, should help to reach a higher acceptability regarding the use of treated sediments in aquatic scenarios.

Acknowledgements

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