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## Stabilisation/solidification of soils contaminated by chlorinated pesticides

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This article describes laboratory tests carried out to identify an effective stabilisation/solidification method for soils contaminated by chlorinated pesticides at a disused industrial site. The effectiveness of the treatment was evaluated using leach tests. Powdered activated carbon (PAC) was used as the primary amending agent, thanks to the strong affinity for organic molecules and high sorption capacity. For some areas of the site, test results showed that treatment with PAC was decisive in reducing leaching of the pollutants below the prescribed limits. For areas with higher concentrations, treatment with PAC alone was not sufficient, although the reduction was significant. According to a detailed programme, further tests were performed: different percentages of cement were added to the soil–PAC mixture as a binding/solidifying agent. For all the agents, the optimum dosage that led to dissolved phase concentration standards for pesticides was identified. The test results are evaluated in view of possible field applications. Factors to be further addressed for an overall evaluation of the technology are discussed.

**Keywords:** stabilisation; chlorinated pesticides; activated carbon; cement; laboratory test; field applicability

### 1. Introduction

Soil pollution in former industrial sites requiring remediation is a common, relevant problem in many industrialised countries [1]. The study described in this article is part of the soil remediation programme of a disused industrial plant located in northern Italy. The site (16 ha in size) comprises main buildings, paved areas and a green area that formerly hosted slurry ponds. Production at the site was mainly focused on chlorinated pesticides. Several decades of industrial activity resulted in widespread soil pollution. The main contaminants detected are chlorinated pesticides, some heavy metals and heavy hydrocarbons, all resulting from the industrial processes. Routine groundwater monitoring was performed within the site and downstream. The results excluded significant spreading of the contamination from the soil to the water table below. Indeed, the major contaminants present low water solubility. Nevertheless, in compliance with Italian regulations, periodical monitoring and a stabilisation programme to reduce the release of pollutants from soils

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were required to prevent future migration to groundwater and/or health hazards in view of the reuse of the site.

Amendment of soils and sediments with activated carbon (AC) has been shown to reduce aqueous concentrations of organics [2,3], and thus be beneficial for the environment. AC has also been used as a sorbent for pesticides in agriculture to reduce phytotoxicity to crops on formerly treated fields [4]. The advantage of AC amendment is that it can be viewed as an *in situ* remediation technique that does not require extensive soil excavation [5]. Stabilisation/solidification processes have been in use for many years and widely applied for containment of metals in soils and sludges [6–10]. Only recently, have stabilisation/solidification techniques been applied to the stabilisation of organics detected in contaminated soils and sludges [6–10,11].

To select a viable remediation technology for a given contamination problem, the effectiveness of a treatment method is usually evaluated through a preliminary review of the possible treatment options, a laboratory stage to test the candidate method and eventually, if the treatment has proven positive, a pilot-scale stage. This article describes the laboratory stage aimed at selecting the best available technology economically viable (BATEV) in the preliminary design phase of the remediation programme. AC amendment was first used as a stabilising procedure to reduce the release of chlorinated pesticides into the aqueous phase. A two-step process, consisting of amendment with AC and solidification by cement addition, was then tested to improve the results of the first testing phase. The introduction of new Italian legislation on polluted sites caused a temporary impasse in the remediation programme. As a consequence, the pilot-scale stage of the treatment is still pending. Nevertheless, the results stimulated some considerations about the field applicability of the technology. Finally, the economical viability of the treatment is discussed to balance the environmental protection requirements, the feasibility and reliability of the technology, as well as the design and implementation costs.

## 2. Materials and methods

### 2.1. Soils

Soil characterisation comprised > 100 boreholes and grab samples; in total, > 300 soil samples were analysed for pesticides, metals and hydrocarbons. By coupling the results of chemical analysis with information about the plant activities (production processes, layout of critical infrastructures and historical development of site), the site was divided in four 'homogeneous' areas (A, B, C, D). The soil quality of each area was assimilated to the result of the borehole with the highest concentrations. For each representative borehole, we selected the sample with the highest concentrations of pesticides (Table 1). In general, soil up to 3 m from the ground level was the most contaminated by pesticides. This was later confirmed by an independent geostatistical analysis of the contaminant concentrations, performed after this set of experiments. About 20 kg of soil were excavated at each representative location (i.e. close to the original borehole) within the most contaminated soil level and were mixed thoroughly. By adopting this procedure both experiments represented the worst case scenario, and the number of tests met the budget constraints. The collected soils were stored in air- and watertight containers at room temperature prior to leaching tests.

According to the Italian legislation in force at the time of the study (D.M.471/99), the regulatory limits (CLA) for soils from industrial sites were exceeded by some metals, chlorinated pesticides and heavy hydrocarbons. The soils were mainly fine-grained and were roughly described as sandy silts. Grain size analyses and common classification tests requiring manipulation of soils (e.g. Atterberg limits) were not carried out owing concerns about the safety of laboratory operators.

Table 1. Concentrations of pollutants measured in soils from the four areas.

	Area A (mg · kg <sup>-1</sup> )	Area B (mg · kg <sup>-1</sup> )	Area C (mg · kg <sup>-1</sup> )	Area D (mg · kg <sup>-1</sup> )	CLA (mg · kg <sup>-1</sup> )
Lead	64	18	14	17	1000
Copper	5500	140	30	39	600
Alachlor	nd	nd	nd	0.02	1
Aldrin	0.2	0.12	nd	nd	0.1
Atrazine	nd	0.03	0.02	nd	1
α-HCH	1.3	nd	nd	nd	0.1
β-HCH	nd	nd	nd	nd	0.5
γ-HCH (Lindane)	23.7	nd	nd	nd	0.5
Chlordane	3.03	2.24	0.02	0.83	0.1
DDD + DDT + DDE	1	0.34	nd	0.01	0.1
Dieldrin	0.2	0.29	nd	nd	0.1
Endrin	nd	nd	nd	nd	2
Hydrocarbons C < 12	124	1	nd	nd	250
Hydrocarbons C > 12	13,904	167	208	90	750

Note: CLA, limit of Italian regulation for groundwater; nd, below detection limits; HCH = hexachlorocyclohexane; DDT = dichlorodiphenyltrichloroethane; DDE = dichlorodiphenyldichloroethylene; DDD = dichlorodiphenyldichloroethane.

## 2.2. Leach tests

Leach tests were performed following the method described in UNI 10802. In brief, soil clods at their natural water content were broken up and passed through a 2-mm aperture sieve. The appropriate amount of wet soil to contain 100 g of dry solids was then mixed with deionised water to a water/soil ratio of 1 L · 100 g<sup>-1</sup>. The mixture was agitated in an overhead mixer for 24 h and then centrifuged at 2000 rpm for 30 min. The supernatant was vacuum-filtered through a 0.45 μm membrane. The analytical procedure to determine the concentration of pesticides consisted of: preliminary purification by acetonitrile, liquid-phase extraction by *n*-hexane/dichlorometane mixture, desulfuration, purification/fractioning by chromatography on silica gel, and finally chromatographic analysis by electron capture detector. Metals were determined by atomic adsorption spectrophotometry.

In 'blank' tests (i.e. performed on untreated soils), all parameters listed in the regulatory look-up tables and exceeding the regulatory limits for soils (Table 1) were determined. In the leach tests on amended/stabilised soils, only substances belonging to the pesticides group were determined. In agreement with the local Environmental Agency, the regulatory limits for groundwater (C.L.A. *ex* DM 471/99; Table 2) were assumed as the maximum allowable concentrations in the leachate. These limits were used to define the appropriate detection limits of the analytical methods. Given the preliminary nature of the study and budget limitations, no replicates were performed (one leaching test for each group).

## 2.3. Amendment with activated carbon

Because blank tests had revealed that pesticides were the only contaminant group released in concentrations above the adopted limits, activated carbon (AC) was selected as sorbent for pesticides [11–14]. To ensure a uniform distribution of the amending agent within the soil matrix, powdered activated carbon (PAC) was preferred over granulated activated carbon (GAC). Moreover, PAC was found to reduce the freely dissolved concentration more effectively than GAC when amended to contaminated soils and sediments [5].

The used PAC is a commercial product (VWR Intl., Milan, Italy), with a sorption capacity of ≥ 30% by weight, expressed as *n*-hexane. No further characterisation of PAC was carried out.

Table 2. Concentrations of pollutants in the leachate of blank tests.

	Area A ( $\mu\text{g} \cdot \text{L}^{-1}$ )	Area B ( $\mu\text{g} \cdot \text{L}^{-1}$ )	Area C ( $\mu\text{g} \cdot \text{L}^{-1}$ )	Area D ( $\mu\text{g} \cdot \text{L}^{-1}$ )	CLA ( $\mu\text{g} \cdot \text{L}^{-1}$ )
Lead	nd	nd	nd	nd	10
Copper	nd	nd	nd	nd	1000
Alachlor	nd	1.40	0.66	nd	0.1
Aldrin	nd	nd	nd	nd	0.03
Atrazine	nd	nd	nd	nd	0.03
$\alpha$ -HCH	0.10	nd	nd	nd	0.1
$\beta$ -HCH	0.30	nd	nd	nd	0.1
$\gamma$ -HCH (Lindane)	6.40	0.13	0.52	1.4	0.1
Chlordane	nd	nd	nd	nd	0.1
DDD	nd	0.59	nd	nd	0.1
DDE	nd	0.80	nd	nd	0.1
DDT	nd	0.59	nd	nd	0.1
Dieldrin	0.30	0.29	nd	nd	0.03
Endrin	nd	nd	nd	nd	0.1
Sum of listed pesticides	7.10	3.80	1.18	1.4	0.5
Sum of pesticides	11.80	3.80	1.18	1.4	–
Total hydrocarbons (as <i>n</i> -hexane)	nd	nd	nd	nd	350

Note: CLA, limit of Italian regulation for groundwater; nd, below detection limits; HCH = hexachlorocyclohexane; DDT = dichlorodiphenyltrichloroethane; DDE = dichlorodiphenyldichloroethylene; DDD = dichlorodiphenyldichloroethane.

The total amount of pesticide bound to the soil (Table 1) and the reduction in the sorption capacity of AC mixed with soil [13] were considered to set the PAC dosage. A minimum dosage of 0.002 g PAC per g of soil (0.2% by weight) was estimated to potentially adsorb the pollutants. PAC (1 g) was added to 500 g of soil from each area previously passed through a 2-mm aperture sieve; the soil–PAC mixtures were sealed in 1 L glass bottles and agitated on an overhead mixer at 5 rpm for 1 h. The mixtures were allowed to cure for 4 days at room temperature ( $20 \pm 2^\circ\text{C}$ ); 100 g were then sampled from each mixture and subjected to leach tests.

#### 2.4. Stabilisation/solidification with activated carbon and cement

Based on the results of leach tests, for some areas (see Results section), treatment with PAC alone was not sufficient to reduce the release of pesticides below the prescribed limits. Class IIIB, slag furnace cement (Cementir, Spoleto, Italy) was used in addition to PAC as solidifying agent. Slag furnace instead of Portland cement was used because its better performance against chemical attack [15] is generally recognised. The treatment was conducted on soils from area A, which showed the highest residual release in leach tests. The PAC dosage was fixed: 0.2 g of PAC were added to 100 g soil and mixed in an overhead tumbler for 1 h. Two test series were performed. In the first, the cement dosage was fixed (3% of soil by weight) and the mixing sequence of cement and water (20% of soil by weight) varied: (1) PAC–cement–water, (2) PAC–water–cement, respectively. In case 1, PAC was added first, then cement was applied, and the mixture was agitated in a rotary tumbler. Next, water was gently sprayed into the mixture. In case 2 water was first sprayed onto the PAC–soil blend. The moist soil was stored overnight in a watertight container to facilitate uniform hydration. Next, cement was added to the soil and mixed thoroughly. The soil was allowed to cure for 5 days at room temperature, and then subjected to leach tests.

In the second test series, the cement dosage was increased (4 and 10%). In an attempt to optimise the performance of the treatment by enhancing the sorption of organics onto PAC, the soil–PAC mixture was allowed to cure for 10 days. Then cement and eventually water were added. The treated soils were cured for 5 days at room temperature and finally subjected to leach tests.

The efficiency of the treatments was assessed qualitatively by a ‘pass–fail’ approach, based on comparison between the concentrations leached in blank and amended tests. The statistical significance of the results could not be addressed because of the limited number of tests. A more complete investigation taking into account the statistical significance would certainly be desirable in a later stage of the study.

### 3. Results and discussion

#### 3.1. Blank tests

The results of blank tests are listed in Table 2 and illustrated in Figure 1. Substances below the detection limit in all areas (e.g. Atrazine) are not shown for simplicity. The contaminants released in detectable concentrations in deionised water belonged exclusively to the pesticide group; the substance type and concentration varied with area. Therefore, the stabilisation focused on the release of pesticides. Lindane was the only substance exceeding the adopted limits throughout the site. The parameter ‘pesticide sum’ in Table 2 exceeded the sum of individual concentrations because it accounted for additional substances in the pesticide group not included in regulations, but detected by the analytical method in the dissolved phase. Unregulated substances were disregarded.

#### 3.2. Soil amended with activated carbon

Addition of PAC to the soils did not cause significant changes in the consistency or size of soil aggregates. The results of leach tests on the soil–PAC mixtures are reported in Table 3. A comparison of leach results for unamended and PAC-amended soils for areas A and B is shown in Figures 2 and 3, respectively. A systematic reduction in pesticides released into the water phase was observed, with the exception of Alachlor in area A, which increased with PAC amendment. For areas C and D, all leached concentrations were below the regulatory limits and even below the

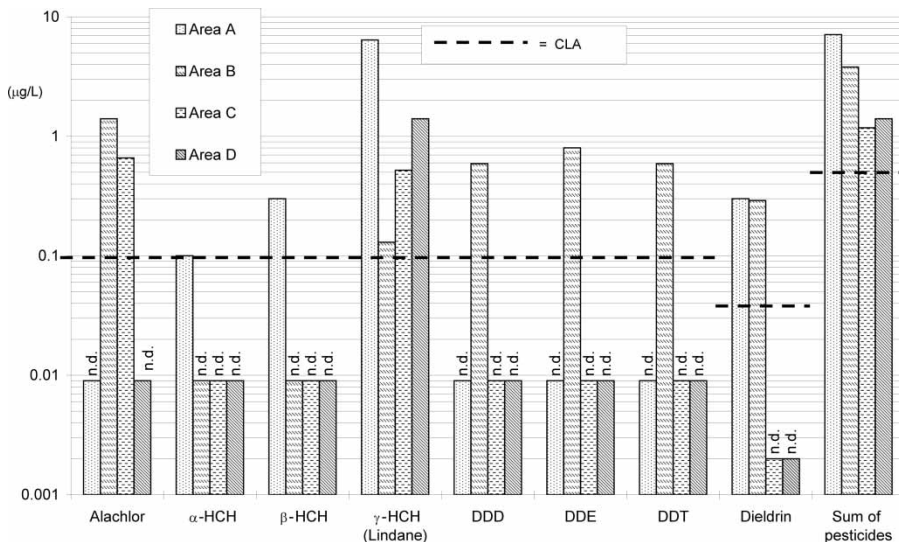


Figure 1. Results of ‘blank’ leach tests (nd, below detection limit).

Table 3. Concentrations of pesticides in the leachate of soils stabilised with PAC.

	Area A ( $\mu\text{g} \cdot \text{L}^{-1}$ )	Area B ( $\mu\text{g} \cdot \text{L}^{-1}$ )	Area C ( $\mu\text{g} \cdot \text{L}^{-1}$ )	Area D ( $\mu\text{g} \cdot \text{L}^{-1}$ )	CLA ( $\mu\text{g} \cdot \text{L}^{-1}$ )
Alachlor	0.44	0.08	nd**	nd	0.1
$\alpha$ -HCH	nd	nd	nd	nd	0.1
$\beta$ -HCH	nd	nd	nd	nd	0.1
$\gamma$ -HCH (Lindane)	0.9	nd	nd	nd	0.1
DDD	nd	0.34	nd	nd	0.1
DDE	nd	0.19	nd	nd	0.1
DDT	nd	nd	nd	nd	0.1
Dieldrin	nd	0.14	nd	nd	0.03
Sum of pesticides	1.33	0.67	nd	nd	0.5

Note: CLA, limit of Italian regulation for groundwater; nd, below detection limits; HCH = hexachlorocyclohexane; DDT = dichlorodiphenyltrichloroethane; DDE = dichlorodiphenyldichloroethylene; DDD = dichlorodiphenyldichloroethane.

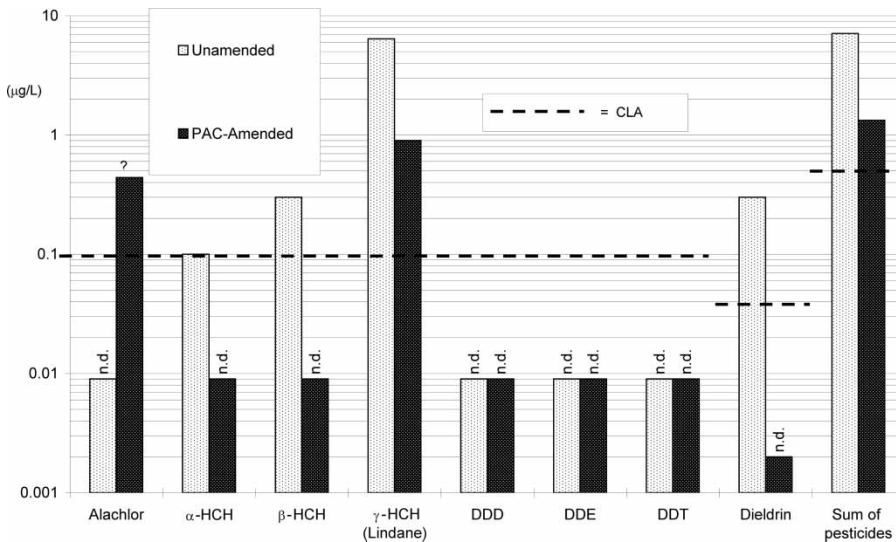


Figure 2. Leach test results for unamended and PAC-amended soils: area A.

detection limits of the analytical method. Therefore, amendment with PAC (0.2% by weight) can be considered an effective stabilisation method in areas C and D. For areas A and B, the concentrations of some pollutants and the sum of all concentrations remained higher than the regulatory limits, with area A showing the maximum release in terms of sum of total concentrations.

Increasing the dosage of PAC was first attempted to reduce leaching, but negligible reduction in the release of pesticides was observed with a double PAC dosage (0.4%). Thus other PAC dosages were not further investigated. Rather a solidification step with cement was added to the treatment procedure. Soils from area A, which had the highest release, were used.

### 3.3. Stabilisation/solidification with activated carbon and cement

The purpose of the solidification was to further reduce the release of pollutant by encapsulation of PAC–soil mixtures in a solid matrix. Regardless of the mixing sequence, cement hydration and mixing activity induced a significant increase of the average size of soil aggregates, with a consequent reduction in specific surface (Figure 4).

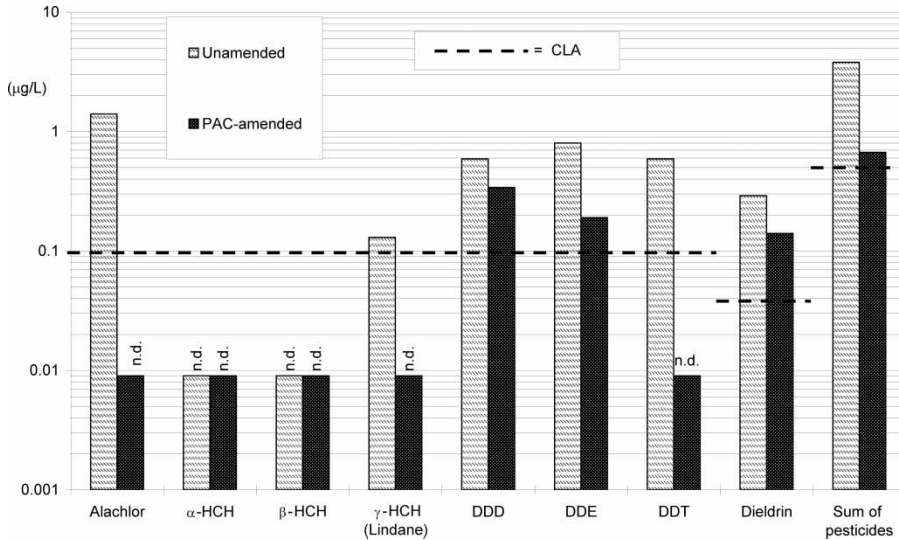


Figure 3. Leach test results for unamended and PAC-amended soils: area B.

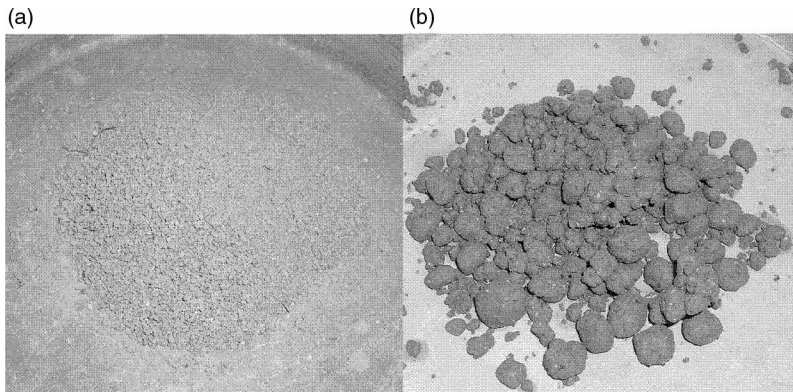


Figure 4. Effect of cement hydration on the size of soil aggregates: (a) PAC-amended soil, (b) after the addition of cement and water.

Lindane was the only substance found in detectable concentrations in the leachate after treatment with cement. Test results (Figure 5) indicated that the mixing sequence has no significant influence on the release of pesticides for given cement content. Therefore, the mixing sequence of water and cement does not appear a significant variable in the stabilisation method. Compared with treatment with PAC only, a slight increase in Lindane concentration was observed. The sum of all concentrations decreased with respect to PAC amendment only. This result may be influenced by the anomalous increase of Alachlor, which was registered in PAC-only treated sample with respect to the blank test. The slight increase in Lindane may have been favoured by disruption of soil/cement aggregates caused by the screening through a 2-mm sieve prior to the leach test. This operation was done to be consistent with previous tests. Therefore, in the subsequent phase of the study, soil aggregates formed upon cement hydration were left unaltered. Moreover, in this stage, the contact time between PAC and soil alone was much shorter (1 h) than in PAC-amended soil (4 days). A shorter contact time might have reduced the uptake of organics from PAC. Data from the literature [16] suggest that the sorption of pure gaseous organics onto AC is completed within a couple of days; however, for organics adsorbed onto soils a longer contact time may be



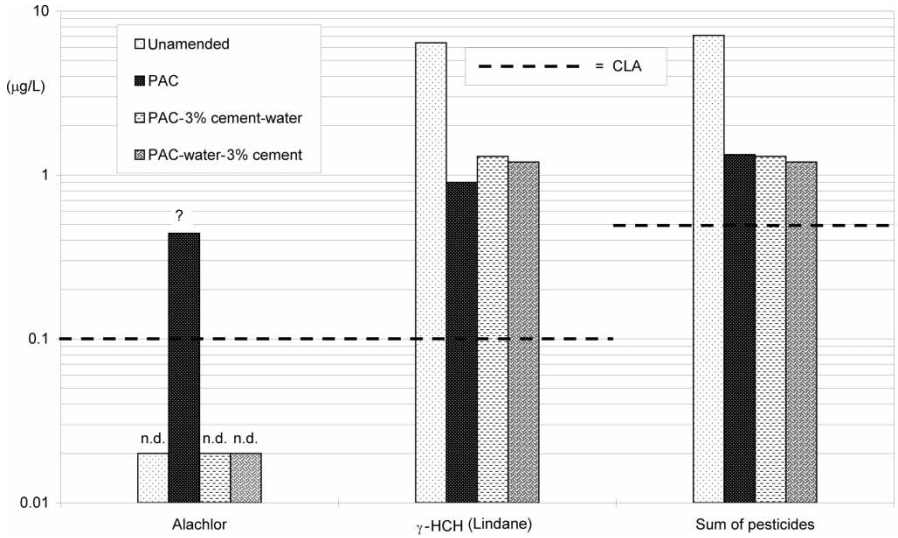


Figure 5. Effect of PAC-amendment and mixing sequence with cement (3%) on the dissolved phase concentrations.

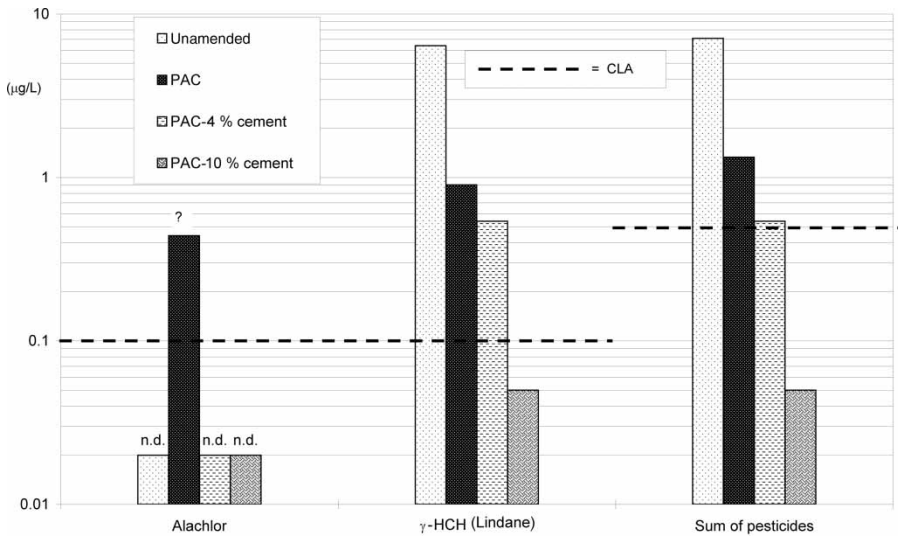


Figure 6. Effect of PAC and cement dosage on the dissolved phase concentrations.

necessary because desorption from soil and adsorption to AC must take place. The actual contact time of soil and PAC may thus play a role in the efficiency of the treatment.

The results of the second test series are illustrated in Figure 6. A significant reduction in Lindane concentration occurred at a cement content of 4% by weight compared with blank tests, but the concentrations remained higher than the limit. A decisive reduction occurred at 10% cement content, at which the Lindane concentration was well below the prescribed limit. It must be noticed that the contact time of PAC and soil in the second test series was 10 days, which may have positively contributed to the sorption of pollutants. Combined treatment with PAC and cement also reduced the sum of concentrations below the prescribed limits. An overall 99% efficiency of the treatment can be calculated.

To assess the long-term release of the stabilised soil, one specimen prepared with 4% cement and cured in a sealed glass bottle for about 3 years was subjected to the leach test. The results of the long-term cured sample are very similar to those of samples cured for 5 days. The only substance with a concentration higher than the limit was Lindane: the measured concentration was slightly higher ( $0.6 \mu\text{g} \cdot \text{L}^{-1}$ ) than that registered in the short curing test ( $0.54 \mu\text{g} \cdot \text{L}^{-1}$ ). In short, the combined stabilisation/solidification technique appeared essentially permanent.

### 3.4. Applicability

The results obtained in the laboratory stage are promising if the relatively low cost of the reagents and the established experience in stabilisation/solidification procedures are considered. However, some aspects related to mix-design and optimisation of the process parameters need further insight before a pilot-scale test can be set up. First, the PAC dosage used in this study (0.2%) is relatively low; indeed it is much less than the dosages generally used in soil remediation projects (5–10%). A very low PAC dosage would probably be more difficult to disperse homogeneously within the soil than in the laboratory. Higher dosages of lower-quality AC could be used to reduce costs as long as the efficiency of the treatment is preserved. As for the *in situ* mixing procedure, it would be more convenient and economically sustainable to mix AC and cement in one single step and then add the reagents to the soil. Although significant interaction of AC and cement in the dry state is not expected, depending on the moisture content of the soil, cement may quickly hydrate and encapsulate PAC, and this would hinder the sorption. A specific test in which pre-mixed PAC and cement are added to the soil would verify the possible reduction in efficiency in terms of pollutant retention. Although treatment with cement alone is not normally sufficient to stabilise organics, based on practical considerations, it could be useful to test the addition of cement alone. The cement dosage should be higher than those used in the previous tests, for example, 15–20% content by weight. A 20% content is generally used to stabilise polluted soils. If the cement treatment alone proves to be efficient, the *in situ* implementation of the treatment would be easier.

## 4. Conclusions

A combined PAC–cement stabilisation procedure was applied to reduce the release of pesticides from polluted soils. Based on the laboratory results, it was possible to outline an optimal procedure in terms of dosages, mixing method and curing time. As a result, the release of pesticides in pore water was reduced below the prescribed limits. To assess the feasibility of the technology a pilot stage is required since the dosage and mixing procedure are usually less accurate in the field than in the laboratory tests.

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