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Research

Experimental system to displace radioisotopes from upper to deeper soil layers: chemical research

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Abstract

Background: Radioisotopes are introduced into the environment following nuclear power plant accidents or nuclear weapons tests. The immobility of these radioactive elements in uppermost soil layers represents a problem for human health, since they can easily be incorporated in the food chain. Preventing their assimilation by plants may be a first step towards the total recovery of contaminated areas.

Methods: The possibility of displacing radionuclides from the most superficial soil layers and their subsequent stabilisation at lower levels were investigated in laboratory trials. An experimental system reproducing the environmental conditions of contaminated areas was designed in plastic columns. A radiopolluted soil sample was treated with solutions containing ions normally used in fertilisation (NO_3^-, NH_4^+, PO_4^{---} and K^+).

Results: Contaminated soils treated with an acid solution of ions NO_3^-, PO_4^{---} and K^+, undergo a reduction of radioactivity up to 35%, after a series of washes which simulate one year's rainfall. The capacity of the deepest soil layers to immobilize the radionuclides percolated from the superficial layers was also confirmed.

Conclusion: The migration of radionuclides towards deeper soil layers, following chemical treatments, and their subsequent stabilization reduces bioavailability in the uppermost soil horizon, preventing at the same time their transfer into the water-bearing stratum.

Background

In the last sixty years, several episodes of artificial emission of radionuclides into the atmosphere have occurred as a result of nuclear weapons tests or as a consequence of nuclear power plant accidents (e.g. Chernobyl, 1986). Release of radionuclides to the environment represents a risk to human and animal health both as a source of irradiation and, above all, for the toxicity exerted at the cellular level by mutagenic, teratogenic and oncogenic actions [1,2], because of their presence in the food chain [3]. However, natural or artificially released hard gamma emitters can be easily found as minimum detectable activity is very low.

Essentially, radioactive elements can be introduced in the food chain by:

1. direct consumption of contaminated vegetables;

2. consumption of contaminated foods of animal origin, obtained from animals fed with radioactive fodder [4];

3. contamination of groundwater and direct or indirect human assimilation [5].

Contamination presently found in polluted areas is mainly due to Cesium 137 (^{137}Cs) and, to a lower extent, to Strontium 90 (^{90}Sr), although traces of other high atomic mass radioisotopes may be found [6,7]. The distribution of these radio-emitting nuclides in the soil profile is graphically represented by a curve starting from the ground surface and decreasing gradually toward deeper levels [6,8,9], suggesting low mobility [10,11].

Several ecoremediation technologies based on biological methods [12,13] have been developed for soil decontamination. Potential bioremediation agents include wild plants, known as hyperaccumulators [14,15], genetically engineered plants [16,17], fungi [18], and natural [19-21] or genetically modified microrganisms [22-24]. Such agents exhibit enhanced biochemical pathways responsible for the adsorption of heavy metals or radionuclides. The disposal of contaminated biomasses represents, however, a trouble and is a big limit to the methodology application. Other studies concerning the use of amendments able to limit the radionuclides uptake by plants have been carried out [25,26]. These systems are able to reduce fodder and vegetables contamination, but they do not reach an acceptable level of toxicity reduction.

During voluntary activities carried out as part of the agro-veterinary project by the Humanitarian Association "Smile – Un sorriso per Chernobyl", it was possible to directly observe how isotopes of elements of the first group, which form soluble salts in water, remains in the superficial soil layers, even after seventeen years from the incident at the nuclear reactor. This radioisotopes bioavailability in the uppermost soil horizon is the result of the low rate transport caused by filtration of atmospheric precipitation, transfer on the colloidal and fine-dispersed particles and migration along the plant root system. It has been also remarked how the velocity of the radioisotopes vertical migration is a soil-type depending process [10] and this is probably due to the formation of stable complexes between radioisotopes and soil clay minerals. Toso and Velasco [27] described how the vertical distribution of low solubility elements in the soil is attributed to their presence in three forms: mobile, adsorbed and bonded.

Therefore, it is a plausible working hypothesis that the radioisotopes may be displaced from complexes or adsorption sites, mobilized by water, and then immobilized in the underlying layers of the soil, exploiting the sorbent and complexing capacities of the soil. The bioavailability of radioisotopes would be decreased by their transfer from the zone of leaching to the zone of accumulation. The removal of radioisotopes from the most superficial soil layers, even just few centimetres, would render them unavailable to herbaceous plants and grasses, allowing the production of safe hay, while their subsequent stabilisation at greater depth would guarantee against their transfer into the water-bearing stratums, also been this a long-term stabilization process within periods comparable with natural decay.

The aim of this paper was to assess in laboratory assays the possibility of removing the radionuclides complexed in the superficial soil layers and fixing them into lower levels where insoluble stable compounds can be formed again.

Methods

Soil samples

Two soil samples were used in this investigation. The first sample (named Type 1) was a podzolic, coarse textured (>70% sand), sandy soil, collected from the O horizon and characterized by a 6% organic content. The collection of this sample was performed in a so called "far zone" (~150 km) from the Chernobyl Nuclear Plant. The global radioactivity measures of this soil type, performed by gamma spectrometry, are shown in Table 1. Gamma spectrometry analytical parameters are reported in Table 1 notes. Radioactivity level measured by liquid scintillation was 86,9 DPM/g (see below).

The second sample (named Type 2) was a soil of the same type of the previous one, collected from the O horizon but characterized by a 10% organic content. It was collected in a not Chernobyl disaster affected area located in the North-West of Italy. The global radioactivity measures of this soil type, performed by gamma spectrometry, are also reported in Table 1.

Each of the samples was collected on a 40 cm side square surface, to a depth of 20 cm. The soil samples were cleaned, removing grass, roots and pebbles. Each sample was then dried at 70°C, in a constant vacuum of 700 mmHg. Dried samples were kept at room temperature. Before each experiment, a fraction of samples were rehydrated at 10% w/w.

Sample treatments

Set up of leaching solutions

In order to obtain solutions with efficient radioisotope solubilizing power, various aqueous solutions containing ions normally used in fertilization (such as NO_3^-, NH_4^+, PO_4^{---} and K^+), in equal concentrations were tested. To obtain these solutions, the following reagents were used:

Table 1: Global radioactivity features of Type 1 and Type 2 soil samples performed by gamma spectrometry. Minimum and maximum radionuclides activities detected in Type 1 and Type 2 soil samples, calculated as the difference between the sample and the background values, are shown in Bq/kg. If available, in subscript, for each activity value, the ratios between the sample and the background are shown in percent. Sigma value are shown in percent. Sample mass = 0.75 kg. Gamma spectrometry analytical parameters: detector system = Germanium (ORTEC); start – stop channels = 2.42 – 1988.50 keV; peak rejection level = 30.00%; background width with average of 3 points; decay during acquisition taken in count; adsorption correction = sand (on max activity value). n.a. = not available.

	Type 1 soil			**Type 2 soil**		
	Activity [Bq/kg]		1 Sigma [%]	*Activity* [Bq/kg]		1 Sigma [%]
	Min	Max		Min	Max	
Cesium library						
CS-134	0.387 (100%)	0.464 (100%)	24.18	-	-	-
CS-137	184.052 (99%)	220.733 (100%)	1.89	1.295 (n.a.)	-	13.45
Natural library						
TH-234	9.380 (47%)	14.844 (45%)	7.024E+6	10.641 (n.a.)	18.323 (n.a.)	3.892E+6
PA-234	-	-	-	-	-	-
TH-230	-	-	-	-	-	-
RA-226	25.044 (70%)	33.297 (70%)	7.16	10.800 (n.a.)	14.340 (n.a.)	11.26
PB-214	11.699 (90%)	16.411 (100%)	4.25	1.287 (n.a.)	-	13.17
BI-214	10.620 (89%)	14.332 (100%)	4.10	1.348 (n.a.)	-	14.64
PB-210	22.634 (70%)	46.897 (71%)	1.279E+7	9.593 (n.a.)	19.419 (n.a.)	7.144E+6
U-235	-	-	-	-	-	-
TH-231	-	-	-	-	-	-
PA-231	-	-	-	-	-	-
TH-227	-	-	-	-	-	-
RA-223	-	-	-	-	-	-
RN-219	3.581 (100%)	4.590 (100%)	25.97	-	-	-
AC-228	12.882 (87%)	16.795 (100%)	4.96	1.978 (n.a.)	-	15.90
TH-228	-	-	-	-	-	-
RA-224	17.797 (100%)	23.059 (100%)	15.98	-	-	-
PB-212	13.984 (93%)	19.545 (100%)	2.51	1.088 (n.a.)	-	10.37
BI-212	16.032 (100%)	18.941 (100%)	8.85	-	-	-
TL-208	4.462 (90%)	5.960 (100%)	3.14	0.498 (n.a.)	-	13.49
K-40	357.815 (98%)	412.107 (100%)	1.80	7.892 (n.a.)	-	23.03

potassium nitrate, nitric acid, ammonium nitrate, potassium phosphate, ammonium phosphate and ammonia (Carlo Erba Reagents). These reagents were combined in different proportions.

Ten g of contaminated (Type 1) re-hydrated soil were treated in 50 ml conical test-tubes with 10 ml of the tested solution, in reverse agitation for over night. After centrifugation at 2000 rpm for 20 min, 10 ml of the supernatant fraction were collected and subjected to scintillation counting as described below. The solution that showed the best eluting capabilities in extracting radioactive compounds from soils (named *solution R*; composition given below) was used in the sample treatments in columns.

Samples treatments in columns

Two experiments in column were prepared to assess the removing effects of the chemical treatments in conditions similar to natural ones and to assess the ability of the soil to immobilise the solubilised radionuclides.

A first column, named *column A*, was set to represent the uppermost contaminated soil layers (the zone of leaching), while a second column, named *column B*, was set to represent the deeper soil layers (the zone of accumulation).

Each plastic column was 80 cm in length and 4 cm in diameter. The column was held by a stand; the bottom end was closed by a glass stopcock, filled with cotton to prevent loss of the soil sample. A 200 ml beaker was placed under the column to collect the eluates from the stopcock.

The column A was filled with 100 g of contaminated soil sample (Type 1) up to about 10 cm in height. The soil sample was first treated with 20 ml of solution R, followed by serial addition of 4 aliquots of 10 ml of demineralised water. The water was added only after most of the aqueous solution had flowed out from the stopcock.

Nine treatments were then carried out with 10 ml of solution R, each of which were followed by 4 to 7 washes with 10 ml of demineralised water. These operations were carried out in order to simulate atmospheric precipitations; the total amount of water used in this experiment corresponds approximately to 630 mm of rain, corresponding to the average yearly rainfall. The use of a double volume of solution R for the first treatment was adopted to overcome the immediate buffering power of the soil, to avoid its deactivating effect and to reduce the chemical reagents usage. Each addition of water was followed by waiting until the columns had completely emptied and then collecting each fraction in the beaker. Ten ml of these fractions were scintillation counted as described below and the values obtained, after subtracting the SRL value (see below), were corrected according of the real volume of the fraction.

The column B was filled with about 650 g of normal (Type 2) soil sample to a height of 65 cm. Fifty one ml of the solution resulting from the collection and mixing of the first fifteen treatments and washes of the column A were added. This solution was characterised by a total radioactivity of 22.55 DPM/ml (determined as described below), calculated according to the counting of 10 ml of this solution. This treatment was followed by 32 washes with an average of 20 ml of demineralised water. Similarly to the first column, each addition was followed by waiting until the column had completely emptied before collecting 10 ml aliquots that were analysed. The resulting values were corrected as described above.

At the end of the two tests, the residual radioactivity of the first column and the distribution of the radioactivity in the second one were checked. The soil sample in the column A was entirely collected. Column B was split into 6 segments of the same length and the soil contained in each one were collected. All the soil samples were vacuum dried, homogenized, and subjected to counting as described below.

Analytical procedures

Determination of radioactivity levels in solid matrices

Bulk global measures on untreated soil samples were performed by gamma spectrometry (WAVE s.n.c, Turin, Italy), but the need of a very sensible and fairly rapid method, able to operate on small soil samples, dictated the decision to measure radioactivity by liquid scintillation. Moreover, as the direct counting on the soil samples gave heavy quenching effects that compromise the reading efficiency, the counting was carried out on liquid extracts.

All solid soil matrices were subjected to extraction in order to assess the initial and the final radioactivity levels, at the end of treatments. The extraction was carried out as follows.

Each soil sample (untreated, collected from the entire volume used in the column A or collected from each of the six segments of the column B) were first vacuum dried and then accurately homogenized. Ten g of soil samples, collected from the homogenisation, were extracted twice, first with 7 and than with 5 ml of a solution containing 1 M NH_4Cl and 0.2% HNO_3 for 3 hours in reversal agitation, and centrifuged for 20 min at 2000 rpm. Ten ml of the liquid phase were collected and counted three times in liquid scintillation as described below. An average of counting values was calculated.

Determination of radioactivity levels in eluted solutions

The radioactivity of eluted solutions was directly determined by scintillation counting. Radioactivity values were adjusted using a background radioactivity level value obtained as follows.

The uncontaminated Type 2 soil had pedological features similar to those of contaminated Type 1 soil. Type 2 soil radioactivity level is mainly due to the presence of natural isotopes or ubiquitary environmental contaminants; so this level was considered as representative of the average soil standard contamination.

Five aliquots of 5 g of Type 2 soil were extracted for 3 hour in reversal agitation with 10 ml of solution R diluted 1:6; this dilution value was obtained as ratio between the total amount of solution R and the total amount of water used as washing during the column A experiment. The entire volume of each extract was submitted to counting 4 times. The average value of all results was calculated and was considered as the *Standard Radioactivity Level* (SRL) of eluates of a generic uncontaminated soil.

Liquid scintillation counting

Liquid scintillation counts were carried out with a Packard Tricarb 1900 scintillation counter, using 20 ml plastic vials in which were placed 10 ml of sample solution and 10 ml of liquid scintillation cocktail (Instagel – Packard). Instrumentation efficiencies are 60% for ^{3}H, 95% for ^{14}C, 99% for ^{137}Cs and 97% for ^{90}Sr (0–2000 keV).

The DPM counts were carried out for 60 min for each sample, considering a reading window comprising all the energies (0 – 2000 keV counting channels). This counting method was chosen considering that it was not so important to identify the radionuclides involved into the reactions, since the objective was to shift all the isotopes producing significant doses of radiation.

The transformation into DPM was done automatically by the instrument. The count conditions were as follows: quench indicator tSIE/AEC, count termination at 5%, coincidence time 18 ns. The count values of the first vials of each assay, containing only ultra pure demineralised water and the scintillation cocktail, were used as background and subtracted from all of the other samples.

Results

Leaching solution

The experimental tests were carried out at room temperature with various combinations of reagents. The best results were obtained using an aqueous solution of 0.185 M of KH2PO4 and HNO3 (2.5% KH2PO4 and 1.17% HNO3) with a pH of 1.1 (named *solution R*). Experimental tests (data not shown) have demonstrated a relationship between the efficacy of the treatment and the humidity of the soil sample used, and that the concentration of the solution used here is high enough for carrying out general tests to study the elution rates.

Removal of the radionuclides from the soil

The eluates of the uncontaminated Type 2 soil showed a radioactivity level of 4.75 DPM/ml. This value, considered as "background" and used as the Standard Radioactivity Level (SRL), was subtracted from all the scintillation counts of liquid matrices.

Ten treatments, intercalated with 4 to 7 washes with water, were carried out with the working solution in column A, containing about 10 cm of contaminated soil. The radioactivity values counted in the sequential eluates showed a sinusoidal trend related to each treatment with the reagent. This oscillation is shown in Figure 1, where the graph reports the radioactivity values of only 5 eluates following each treatment, without considering the subsequent ones because lower than or equal to SRL value.

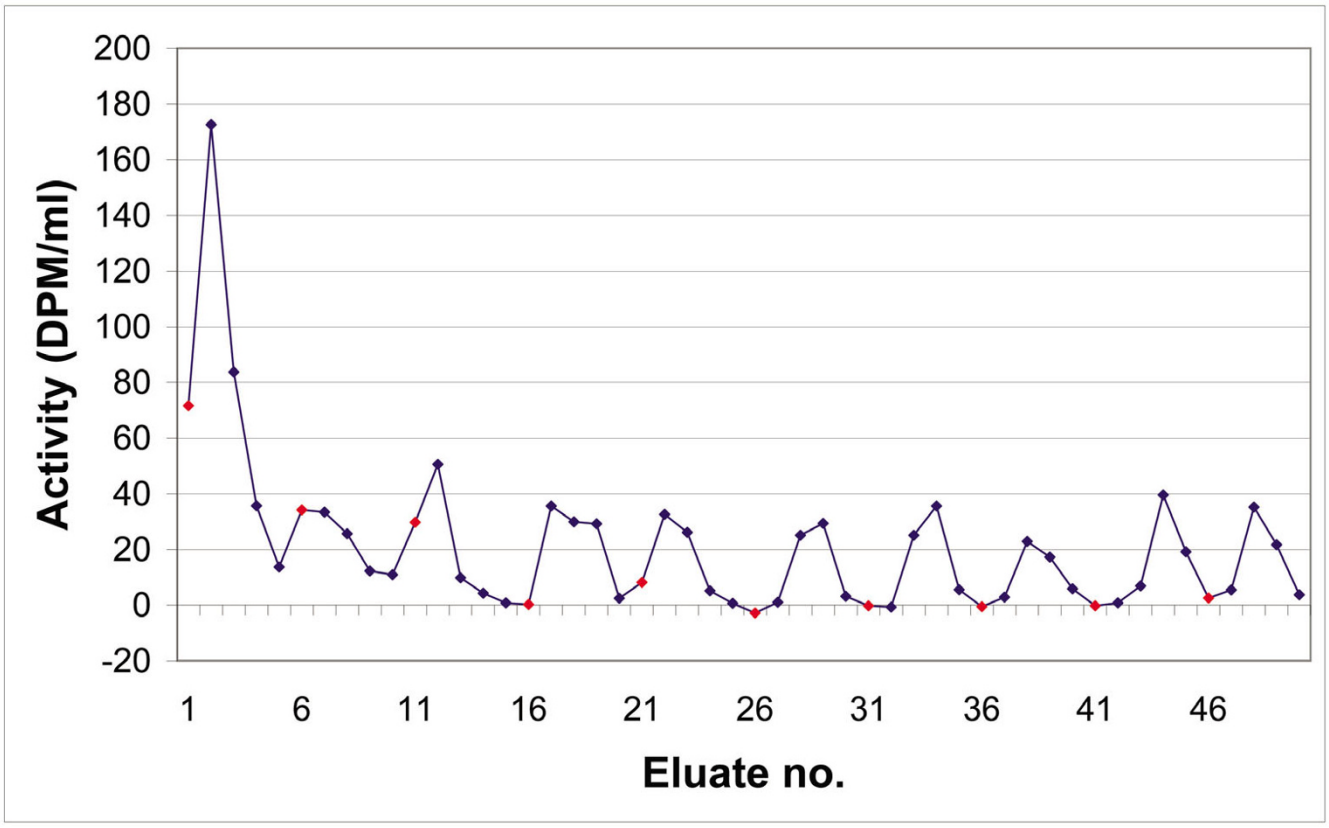

Figure 1

Radioactivity values counted in eluates from column A experiment containing contaminated soil (Type 1). Each treatment with *solution R* (see text; red points), followed by 4 washings with deionised water (black point), yields the solubilization of radionuclides which pass into the eluates from the experimental column. Each peak does not correspond to a specific isotope. Radioactivity values are in DPM/ml. With the proceeding of treatments the DPM's peaks, corresponding to the major isotopes release, are retarded with respect to the first elution after addition of solution R.

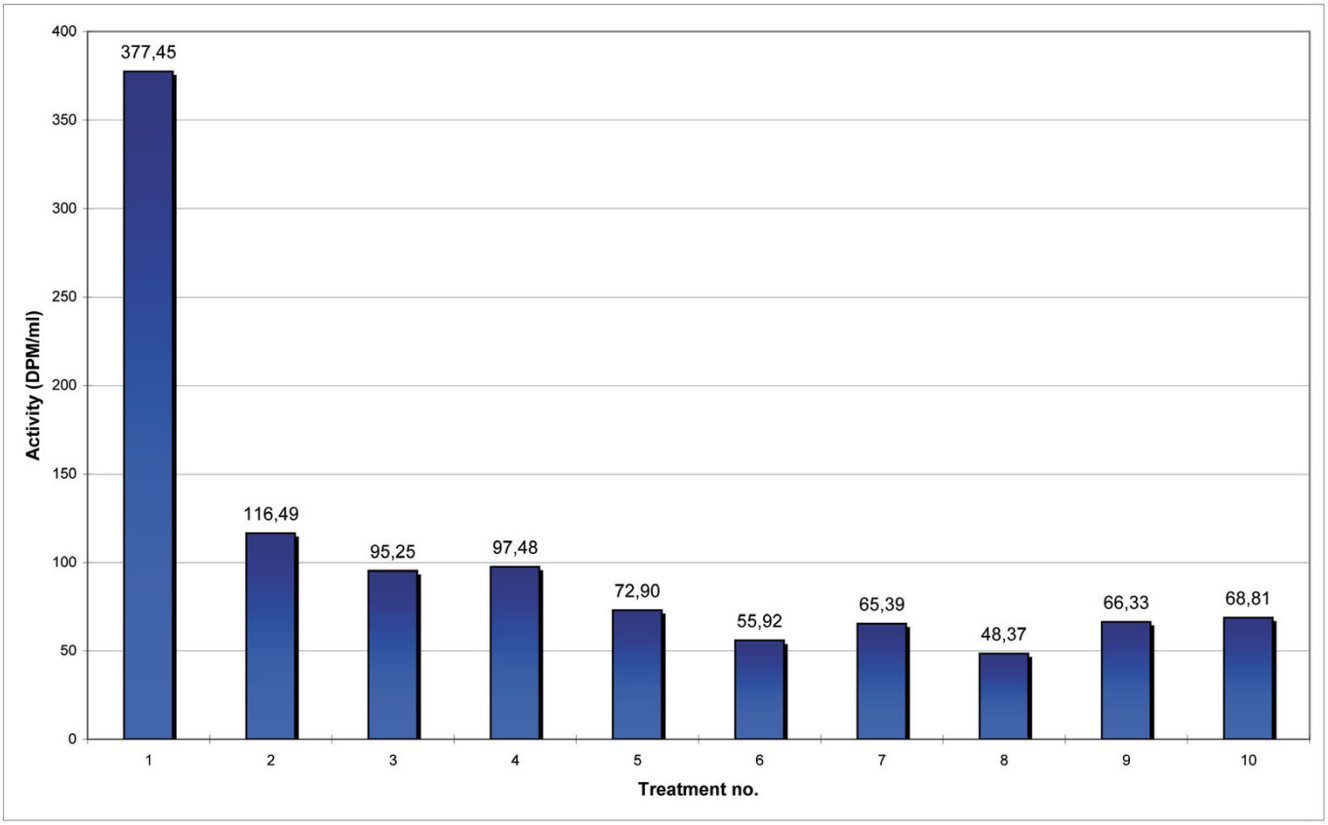

Figure 2

Total radioactivity eluted following each treatment during the column A experiment. The columns represent the sum of the radioactivity values counted for each treatment cycle. Radioactivity values are in DPM/ml. The first treatment, carried out with 20 ml of *solution R* (see text), shows a radionuclides separation power 3.5 to 9 times greater than the following ones, in which an half amount of reagent (10 ml) has been employed.

Furthermore, the total radioactivity eluted by each treatment with solution R was calculated as the sum of the count values of eluates belonging to each treatment series. The results are plotted in the graph in Figure 2. The first treatment carried out with twice the volume of reagent as the following treatments, demonstrates a 3.5 to 9 times greater capacity to remove radionuclides.

The radionuclides removing activities of the following treatments show a homogeneous trend, with a decreasing tendency line reported in Figure 3.

The count values of the extractions carried out on soil samples used in column A experiment were as follows: 86,9 DPM/g for the untreated contaminated (Type 1) soil (before the experiments), 56,0 DPM/g for the treated Type 1 soil. The difference between radioactivity values of the contaminated untreated soil and the contaminated treated soil is 30,9 DPM/g, which corresponds to the total extracted radioactivity, and represents 35.6% of the initial radioactivity.

Recomplexing capacity of the soil

Fifty one ml of the mixture of eluates from Column A experiment, containing the radioisotopes removed from that column, with a total radioactivity level of 22.55 DPM/ml, were added into column B. This addition was followed by 32 washes with water, five of which were used to saturate the column. The radioactivity values of each of the 27 eluted fractions cannot be well distinguished from "background" radioactivity level (SRL) and their trend is random (data not shown).

Column B was divided into six segments of the same length at the end of the experiment. The radioactivity levels of the soil contained in each segment were measured according to the method explained above, and the values are reported in Figure 4b. The lower radioactivity value

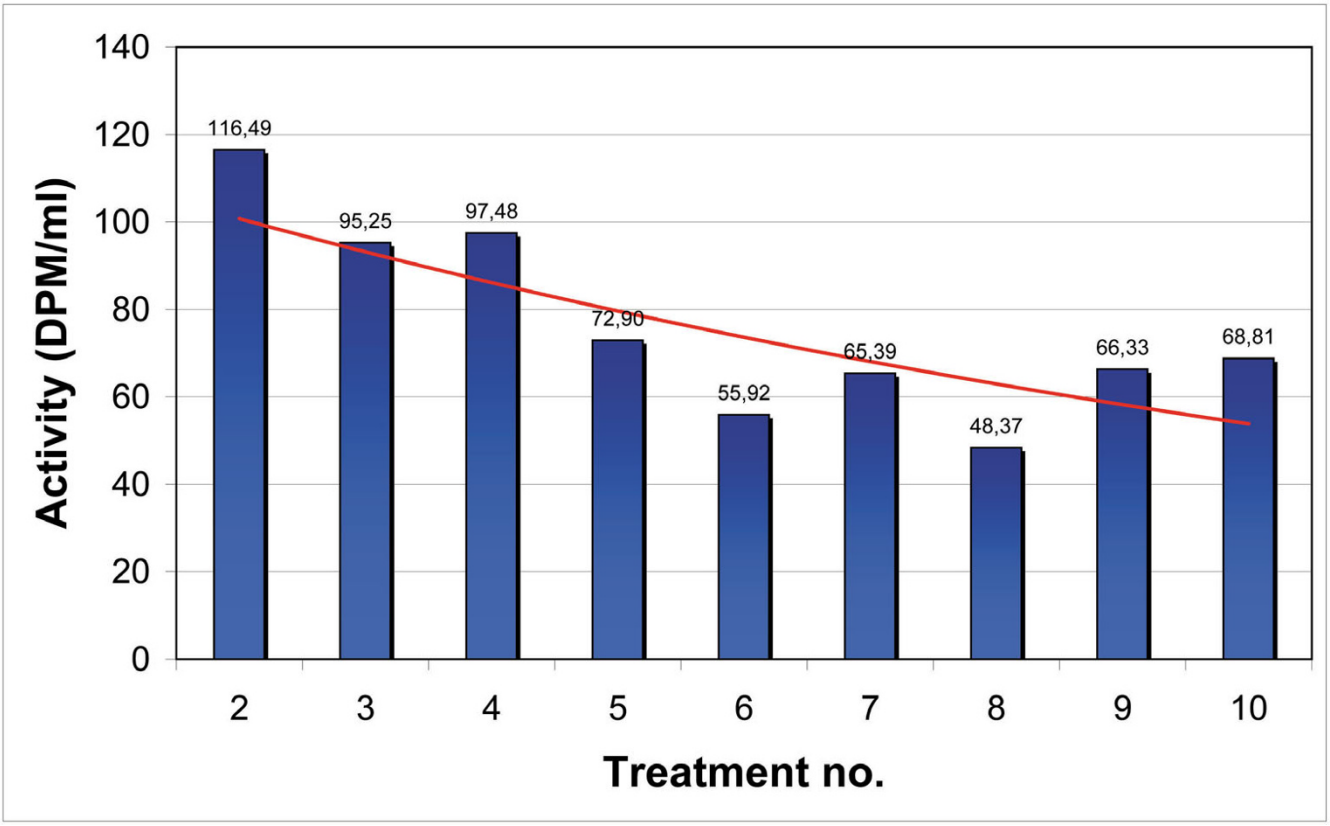

Figure 3
Total radioactivity eluted following each treatment with 10 ml of solution R during the column A experiment. The graph shows in detail the efficiency of each treatment cycles with 10 ml of *solution R*. The columns represent of the sum of the radioactivity values counted for each cycle. Radioactivity values are in DPM/ml. The tendency line gradually comes down with the proceeding of the treatments, with the decreasing of radionuclides concentration eluted from the column.

(6,57 DPM/g) recorded in the top segment is due to the isotopes solubilizing action exert by traces of reagents contained in the volume of the solution initially added into the column. The higher radioactivity values, respectively recorded in the second and the third segment from the top of the column, are due to the fixation at these layers of the isotopes introduced with the mixture of eluates and the natural isotopes mobilized from the top segments.

Discussion

This work, entirely carried out in the laboratory, is a preliminary step to field tests and only aims to investigate new approaches to the problem of polluted areas recovery.

First we identified which treatment solution simultaneously satisfied the need to remove all of the soil radioisotopes and the need to not reduce soil fertility or, at least, to guarantee the possibility of restoring fertility with a later simple treatment. Therefore, we tested the use of ions normally present in inorganic fertilisers, in a combination able to obtain a leaching effect on the soil matter-heavy element complexes without endangering the soil characteristics. The best results were obtained by mixing equal amounts of a 5% solution of KH_2PO_4 and a 2.34% solution of nitric acid (solution 0.185 M of both reactives).

Results from the column experiment on the contaminated soil gave the following indications. From the observation of trends in Figure 1, it may be noted how in the first five treatments the addition of the solution R causes an immediate removal of the radionuclides, while in subsequent treatments the highest peaks of radioactivity are delayed and do not correspond to the first eluate after the addition of solution R.

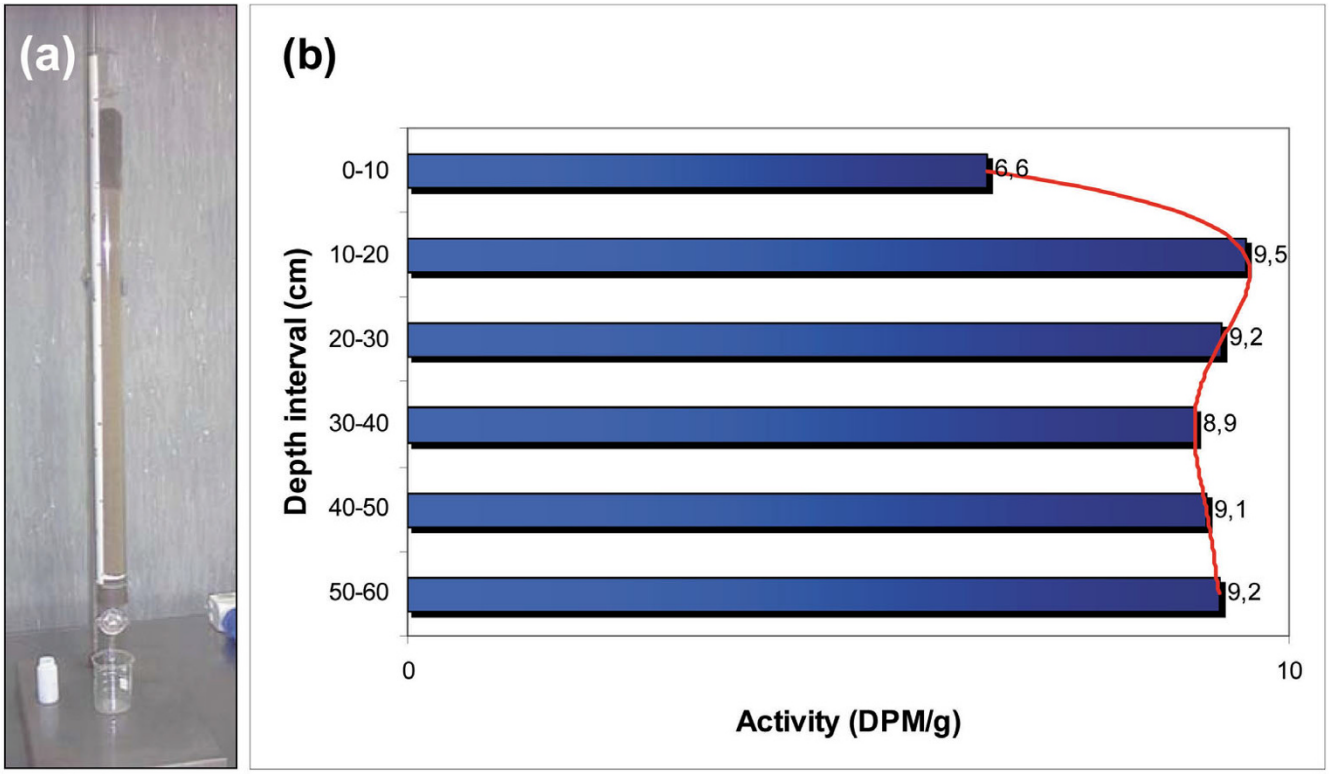

Figure 4
Radioactivity distribution in the uncontaminated soil at the end of the column B experiment. (a) The column B experiment. The column is held by a stand; the bottom end is closed by a glass stopcock, filled with cotton to prevent loss of the soil sample; a beaker is placed under the column to collect the eluates from the stopcock; (b) Radioactivity level of the six soil fractions, corresponding to each segments of the column B experiment. Activity values are in DPM/g. The highest radioactivity values are found in correspondence of the second and the third fraction, due to the complexing and buffering soil power.

The double volume of solution R used in the first treatment showed a higher eluting power; the extraction ratio was in fact higher than 1:2, the ratio that was expected by the use of a double amount of reagent, and highlighted a difference of action of the reagent as the used quantities vary. This rapid decrease in specific activity of eluates almost certainly results from the rapid flushing of the most soluble cationic radioisotopes. A radioactivity level ranging from 3.5 to 9 times higher in the first treatment, suggests a quadratic or cubic relationship between quantities of reactant and removed radionuclides, proposing the need for a more in-depth study of the phenomenon.

Evaluating the total radioactivity eluted by the 10 ml treatments, the trend of the tendency line can be noted, which falls gradually as the radionuclides are removed from the soil (Figure 3). The above observations lead us to believe that the slope of the tendency curve is a function of the total quantities of reactant used.

The problem of avoiding radionuclide transfer into the water-bearing stratums was investigated using an uncontaminated soil by checking the mobilization of isotopes following their solubilisation to see if the complexing and buffering power of the soil is able to fix radioactive substances in the space of relatively few centimetres. The results obtained from tests carried out on the column B experiment showed the following indications. All the eluates of the 27 washes with water (a total of 936 ml in a column with diameter 40 mm), corresponding to about 740 mm of rain, showed a radioactivity always similar to the background SRL value eluted from the non contaminated soil. Therefore, the entire radioactivity initially added in was held back in the mass of soil. The measured radioactivity levels of the six fractions of soil obtained from the sectioning of the column show that the radionuclides were mobilised only several cm.

Conclusions

The acquired experimental data demonstrate that the radionuclides found in contaminated soil can be removed by a nitric acid solution containing ions commonly used in agricultural fertilization. The total amount of radioisotopes removed on the quantity of reagent used. The procedures can be further improved and optimised for large scale field use.

Furthermore, the radionuclides present in the treatment solution after their removal can be rapidly recomplexed by the deeper soil layers with sufficiently strong bonds to avoid their removal even in the case of heavy flushing with water.

The possibility of using the system here described, with other methods already applied in contaminated areas, certainly opens up new prospects for intervention in order to limit the introduction of radioisotopes into the food chain following environmental pollution, as it would allow the recovery of large pasture areas for animal rearing.

The reduction of radioactivity to levels matching the international recommended standards for soil and the depth to which radionuclides have to be accumulated, together with the pedological features of the soil, certainly define the cost of a field application of this methodology, and have to be more investigated in further studies.

In this paper it is not a debatable issue if the choice to use the method here proposed is more useful than the simple land abandon for a suitable period, waiting for natural radioactive decay, or alternative methods for the environment recovery or for maintaining animal and human health.

List of abbreviations

DPM = Disintegrations Per Minute

mmHg = millimeters of mercury

Competing interests

None declared.

Authors' contributions

PC participated in sample collection, in study design and coordination, carried out experimental analyses and drafted the manuscript. AC participated in sample collection and in the study coordination. SG participated in study design, data analyses and wrote the manuscript. MCA and SA participated in the study coordination. All authors contributed to and approved the final version of the manuscript.

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