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Evaluating specificity of sequential extraction for chemical forms of lead in artificially-contaminated and field-contaminated soils

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ABSTRACT

In the present study, we evaluated a commonly employed modified Bureau Communautaire de Référence (BCR test) 3-step sequential extraction procedure for its ability to distinguish forms of solid-phase Pb in soils with different sources and histories of contamination. When the modified BCR test was applied to mineral soils spiked with three forms of Pb (pyromorphite, hydrocerussite and nitrate salt), the added Pb was highly susceptible to dissolution in the operationally-defined “reducible” or “oxide” fraction regardless of form. When three different materials (mineral soil, organic soil and goethite) were spiked with soluble Pb nitrate, the BCR sequential extraction profiles revealed that soil organic matter was capable of retaining Pb in more stable and acid-resistant forms than silicate clay minerals or goethite. However, the BCR sequential extraction for field-collected soils with known and different sources of Pb contamination was not sufficiently discriminatory in the dissolution of soil Pb phases to allow soil Pb forms to be “fingerprinted” by this method.

It is concluded that standard sequential extraction procedures are probably not very useful in predicting lability and bioavailability of Pb in contaminated soils.

1. Introduction

Lead has accumulated in soils of industrial countries over several centuries through anthropogenic processes such as atmospheric deposition from leaded gasoline, coal and oil combustion, incinerator emissions and mining and smelting activities [1,2]. The speciation of Pb in the soil solid phase is likely to control the potential bioavailability and toxicity of Pb to animals and humans, so much effort has been devoted to describing Pb speciation in soils, particularly using sequential extraction techniques [3]. Solubility of Pb in non-acid soils is typically very low as the metal forms insoluble oxide, hydroxide, carbonate and phosphate minerals, and binds strongly to iron oxide minerals and soil organic matter [4,5]. It is perhaps surprising that the reactivity or “lability” of soil Pb as measured using isotopic exchangeability is quite high [3].

Given the range of chemical forms that Pb can take in soils, it is important to determine whether commonly employed sequential extraction techniques are sufficiently selective to distinguish forms of Pb with different chemical labilities and bioavailability. Some studies suggest that plant availability of Pb is correlated to particular Pb fractions extracted in the sequential extraction procedure [6]. However, previous research has shown that the bulk of Pb in contaminated soils is extracted in the “reducible” or “oxide” fraction, a strongly acidic extractant (pH near 2) of the 4-step BCR method [7,8]. Since Pb oxides, hydroxides and carbonates are common minerals of highly Pb-contaminated soils [5,9], and these minerals are quite susceptible to acid dissolution, it is very likely that the sequential extraction step, intended to dissolve Pb bound to oxides of Fe and Mn by reduction at pH 2 (the “reducible” fraction), would also dissolve a large fraction of Pb in these acid-labile minerals. In fact, the non-selectivity of reagents used in sequential extraction has been demonstrated previously [8,10]. For example, Atkinson et al. noted that non-labile Pb (as measured by isotope exchange) is extracted in the “reducible” and “carbonate” fractions as defined by sequential extraction [3]. Ahnstrom and Parker concluded that there was a poor correspondence between the size of the various fractions of Cd extracted by sequential extraction and the size of the labile Cd pool as measured using isotopic exchange [11]. In summary, there is considerable uncertainty about the value of standard sequential extraction procedures in predicting the lability and bioavailability of trace metals such as Pb and Cd in soils.

In the present study, we evaluate a commonly employed 3-step sequential extraction procedure (modified BCR test) for its ability to discriminate among several well-defined solid phases of Pb to determine whether sequential extraction can provide

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useful information on Pb speciation in soils. We further apply the BCR test to soils contaminated in the field by different sources of Pb in order to assess the ability of this sequential extraction to "fingerprint" soils by the type of Pb contamination.

2. Material and method

2.1. Preparation of minerals

In order to create solid forms of Pb with different inherent chemical labilities as models for forms of Pb found in contaminated soils, we synthesized the pure Pb minerals, pyromorphite and hydrocerussite, both of which occur in Pb-contaminated soils, but have different susceptibilities to acid dissolution. We also synthesized microcrystalline goethite (FeOOH) to serve as a model mineral for strongly chemisorbed Pb. Pyromorphite and hydrocerussite were prepared according to the procedures described by Gebeyehu and Bostrom [12] and Flis et al. [13], and goethite synthesis was done according to Atkinson et al. [14]. Following synthesis, all mineral suspensions were repeatedly washed in deionized water to remove excess salts, then air-dried to powder form. X-ray diffraction was used to confirm the identity of the prepared minerals, with degree of crystallinity estimated from diffraction linewidths using the Debye–Scherrer equation.

2.2. Preparation of Pb-spiked soils and FeOOH

A 1000 mg l^{-1} Pb stock solution was prepared by dissolving 1.599 g $Pb(NO_3)_2$ in distilled water and diluting to 1 l in a volumetric flask. A sandy loam topsoil (Arkport series) collected from a pasture (Ithaca, NY), and a peat soil sampled from a vegetable farm (Orange County, NY) were used as representative mineral soil and organic soil for Pb spiking. After air-drying the soil samples, they were pulverized and passed through a 2 mm sieve prior to further analysis. The Arkport mineral soil had a pH of 5.1, organic matter content of 31.8 g kg^{-1} and total Pb content of 4.5 mg kg^{-1} determined by a microwave-assisted hydrofluoric acid digestion technique (EPA 3052). The peat had a pH of 4.9, organic matter content of 711 g kg^{-1} and total Pb content of 9.3 mg kg^{-1} [15]. Organic matter content was determined using the Walkley–Black method.

In order to spike the soils and goethite with 1000 mg kg^{-1} Pb in soluble form, we added 1.0 ml Pb $(NO_3)_2$ to each gram of dry mineral soil, organic soil and FeOOH along with a small volume of deionized water, shook the suspensions for 24 h, then centrifuged for 30 min at 5500 rpm. The supernatants were then discarded, and the solids were washed once with deionized water before being aged (in a moist state) in the laboratory at room temperature. Spiking of the mineral soil (MS) with the Pb minerals, pyromorphite and hydrocerussite, was done to achieve a final soil concentration close to 1000 mg kg^{-1}. This involved mixing 0.0665 g pyromorphite or 0.0625 g hydrocerussite with 50 g air-dry soil. These Pb mineral-spiked soils were stored in the air-dry state.

Table 1
Pb-spiked solids prepared for aging and sequential extraction.

Spiked solid	ID	Total Pb (mg kg^{-1})
Mineral soil+pyromorphite	A+PM	1265 ± 57
Mineral soil+hydrocerussite	A+HC	1235 ± 212
Mineral soil+$Pb(NO_3)_2$	A+Pb	1146 ± 139
Peat+$Pb(NO_3)_2$	P+Pb	1124 ± 14
Goethite+$Pb(NO_3)_2$	G+Pb	1232 ± 56

2.3. Aging and sequential extraction of Pb-spiked samples

The Pb-spiked samples prepared in this experiment are listed in Table 1, which shows three materials (mineral soil, organic soil and goethite) spiked with soluble Pb nitrate, and the mineral soil spiked with insoluble Pb hydroxycarbonate and phosphate. The total Pb concentrations in these materials, reported in Table 1, were determined by digestion using aqua regia and analysis of digests for Pb by flame atomic absorption.

Once the spiked materials listed in Table 1 were prepared, they were allowed to age and subsamples were collected for sequential extraction at 1 day, 1 week, 3 weeks, and 6 weeks after spiking with Pb. The modified 3-step BCR sequential extraction procedure, with extracting solutions used for each step, is shown in Table 2 [16]. The H_2O_2 and $NH_2OH \cdot HCl$ extractants were prepared on the same day the extraction was carried out because of the possibility of reagent decomposition. The fourth step listed in Table 2, involving acid digestion of the soil residue to determine the amount of total soil Pb not extractable in the 3-step process, was not carried out. Instead, total Pb was measured in the spiked materials as a less error-prone means of estimating the fraction of total Pb extracted by the 3-step procedure.

2.4. Sequential extraction of field-contaminated soils

We collected 6 contaminated soils with different sources of Pb contamination inferred from the history of the sites. These are listed in Table 3 and include soils from an industrial site (LP44), an old apple orchard (G6), an urban backyard (AL), and 3 soils from urban community gardens in New York City (GP, GA1, GA2). GA1 and GA2, although from the same garden, had different levels of Pb contamination and organic matter, possibly reflecting soil remediation of GA1 by compost amendment.

Key properties of the contaminated soils, specifically pH, organic matter content and total Pb concentration are summarized in Table 3. Soil pH (1:2.5 soil to water, w/v) was measured using a pH meter with glass electrode. Soil organic matter content was determined using the standard Walkley–Black dichromate oxidation method. Aqua regia digestion, based on the procedure

Table 2
Modified BCR three-step sequential extraction procedure.

Extraction step	Fraction ID	Reagent used	Pb form targeted
1	A	Acetic acid CH_3COOH (0.11 mol l^{-1}). pH 2.55	Exchangeable, water and weak acid-soluble Pb (e.g. carbonates)
2	B	Hydroxyl ammonium chloride $NH_2OH \cdot HCl$ (0.5 mol l^{-1}). pH 1.54	Pb bound to reducible minerals (e.g. iron/manganese oxides)
3	C	Hydrogen peroxide H_2O_2 (8.8 mol l^{-1}). pH 2.45. Ammonium acetate CH_3COONH_4 (1mol l^{-1}). pH 2.0	Pb bound to oxidizable solids (e.g. organic matter and sulfides)
4	D	Acid digestion (to determine unextracted Pb)	Residual Pb (occluded, structural)

recommended by the International Organization for Standardization (ISO) [17], followed by flame atomic absorption spectrometry (FAAS) to measure Pb in the digests, was used to determine total soil Pb concentration. Three analytical replicates were measured for each soil sample. Quality control was assured by the use of reagent blanks, duplicates and a standard reference soil material.

Sequential extraction by the 3-step BCR method, as described above, was performed on the 6 field-contaminated soils listed in Table 3 in order to determine whether the sequential extraction method is sensitive to the type of Pb contamination in soils.

All analytical results were performed as the average of three replicates, presented as mean+standard deviation. Statistical analyses and graphics were carried out using Excel (Microsoft Inc.) software packages.

2.5. Scanning electron microscopy (SEM) with energy dispersive X-ray spectrometry (EDS)

2.5.1. Sample preparation

The field-contaminated soils investigated by SEM–EDS included samples collected at the industrial site (LP44) and urban backyard (AL). These were pulverized to pass a 1 mm sieve, with a very small amount of the soil powder mounted on aluminum holders using two-sided adhesive tape. The mounted soil powders were coated with a thin carbon layer prior to making observations by SEM (JEOL 8900 Electron Probe Microanalyzer) operating at 15.0 kV.

Table 3
Soil properties of the field-contaminated soils from the industrial site (LP44), old apple orchard (G6), urban backyard (AL), and from urban community gardens in New York City (GP, GA1, GA2).

Soil sample code	pH—H_2O	Land use	O.M. (g kg^{-1})	Total soil Pb (mg kg^{-1})
LP44	7.3	Industrial	38.5 ± 0.4	1699 ± 40
G6	5.8	Orchard	69.3 ± 0.6	1269 ± 16
AL	6.7	Urban backyard	87.5 ± 1.4	1229 ± 40
GP	6.7	Urban garden	127 ± 3	1179 ± 32
GA1	7.2	Urban garden	83.9 ± 0.1	351 ± 20
GA2	7.5	Urban garden	45.7 ± 2.9	1106 ± 24

2.5.2. Micrographs of samples

Chemical element mapping for samples LP44 and AL was done on the scanning electron microscope (SEM) equipped with energy dispersive X-ray detector (EDS). The scanning system forms images from secondary and backscattered electrons, and elemental maps from secondary X-rays. Mapping of selected elements of interest for these two soils (LP44 and AL) is displayed (see Appendix).

3. Results and discussion

3.1. Sequential extraction of model Pb-spiked soils

This experiment was designed as a direct test of the BCR sequential extraction's ability to distinguish very different forms of Pb in soil. For this purpose, we initially spiked the mineral soil with a soluble form of Pb ($Pb(NO_3)_2$) and two mineral forms with different stabilities in acid environments (hydrocerussite, pyromorphite) to achieve a soil Pb concentration of about 1000 mg kg^{-1} Pb. Sequential extraction was then conducted at 1 day, 1 week, 3 weeks, and 6 weeks after spiking to determine aging effects on extractability of added Pb. The results, shown in Fig. 1, revealed that the extractable Pb forms, as defined by the BCR method, changed little from 1 day to 6 weeks. The largest fraction of Pb (> 50%) was extracted in fraction B (see Table 2), commonly referred to as the "reducible" or "oxide" fraction. The next-largest fraction (20–30%) was residual Pb (not extracted by Steps 1–3). Consequently, the fractions generally identified as exchangeable or highly-acid labile (e.g., Pb carbonates) and oxidizable (e.g. associated with organic matter and sulfides) Pb were lower.

In comparing the sequential extraction profiles of the mineral soil spiked with three different forms of Pb (Fig. 1), the pyromorphite form resulted in the least extractable Pb as measured by Pb dissolved in fraction A, with no discernible difference in the extraction profiles of the soil spiked with Pb salt and Pb hydroxycarbonate. This pattern did not change with aging, and presumably reflects the susceptibility of hydrocerussite to dissolution at low pH. That is, Pb precipitated in this mineral soil in the form of hydroxycarbonate is indistinguishable from adsorbed Pb salts by this sequential extraction procedure.

All three forms of Pb added to the mineral soil (phosphate, hydroxycarbonate, nitrate salt) were highly susceptible to

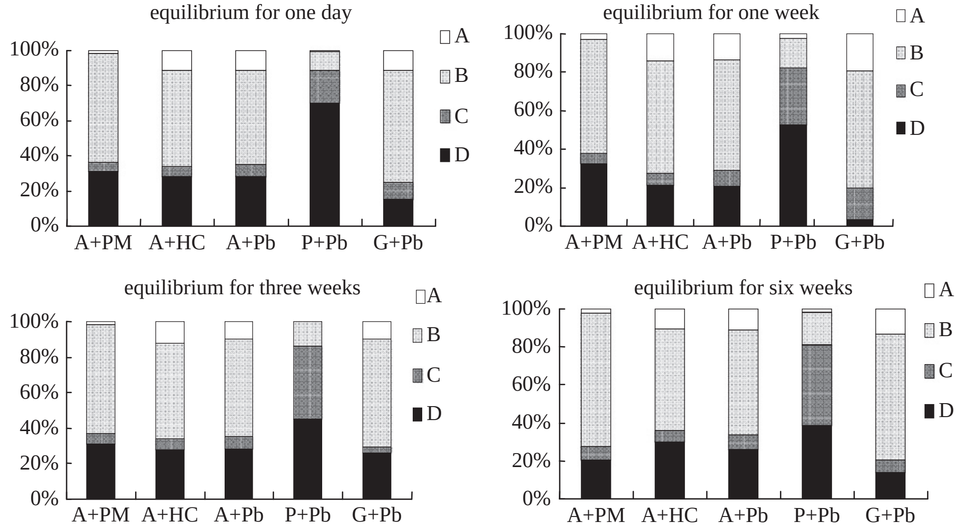

Fig. 1. Distribution of lead in different fractions in mixed sample: mineral soil+pyromorphite (A+PM), mineral soil+hydrocerussite (A+HC), mineral sSoil+$Pb(NO_3)_2$ (A+Pb), peat+$Pb(NO_3)_2$ (P+Pb) and goethite+$Pb(NO_3)_2$ (G+Pb).

dissolution in fraction B, the so-called "reducible" or "oxide" fraction. Since dissolution of Pb phosphates, oxides or carbonates is not dependent on chemical reduction, it is clear that the low pH of hydroxylamine hydrochloride rather than its reduction potential caused the release of Pb from the spiked soils into fraction B. The fact that Pb in the form of pyromorphite did not resist dissolution into fraction B any more than the more reactive and labile forms of added Pb suggests that pyromorphite formation in Pb-contaminated soils may not be the most effective way to reduce soil Pb reactivity or bioavailability.

The peat soil spiked with soluble Pb had the lowest "lability" profile of the model soil systems tested (Fig. 1), with virtually no Pb extracted in fraction A, and a much smaller fraction of total Pb extracted in fraction B. The residual (unextracted) fraction of Pb in this model system was larger than that of any other Pb-spiked material, although there was a tendency for this residual fraction to decrease with aging. Possibly, there was a slow oxidation of organic matter that released some Pb into more labile forms. Chalcophilic trace metals have been shown to bond selectively with sulfides in organic matter [18,19], but the susceptibility of organic sulfide groups to oxidation could lead to metal release into more labile forms during aging with exposure to oxygen.

The sequential extraction results are a strong indication of the stability of Pb–organic matter complexes even at the low pH of BCR sequential extraction. It appears that soil organic matter is capable of retaining Pb in more stable forms than soil minerals, Fe oxides or precipitates of phosphate and carbonate.

The Pb–goethite model system had a unique extraction profile compared to the spiked soils (Fig. 1), as a particularly large fraction of the total Pb was dissolved in fraction B, consistent with the expected reductive dissolution of FeOOH by acid hydroxylamine hydrochloride. It did, however, have a larger percentage of Pb in the most labile fraction A compared to the organic soil or pyromorphite-spiked mineral soil, and did not have as large a residual fraction as the organic soil. The results suggest that goethite is not as strong an adsorbent for Pb as organic matter.

3.2. *Sequential extraction of Pb-contaminated soils from the field*

The soils chosen for sequential extraction testing (see Table 3) were selected from the field based on their relatively high concentrations of Pb and evidence that they had been contaminated for a long time.

The sequential extraction profiles in these soils (shown in Table 4 and Fig. 2) revealed a generally quite small part of Pb in the most labile fraction A, with most of the Pb in fraction B. These results in themselves do not provide much indication of the likely forms of Pb in these soils, as the spiked soil extraction profiles (Fig. 1) similarly had a large part of Pb in fraction B and a small part in fraction A. Thus, forms of Pb adsorbed on soil surfaces or precipitated in hydroxycarbonate or phosphate minerals, are extracted minimally into fraction A and predominantly into fraction B. This lack of distinguishability of different forms of Pb in soils using sequential extraction derives from the aggressiveness of the extractants themselves, and the indication that the very low pH of extraction (of Step 2 in particular) is the main factor in solubilizing a wide range of Pb forms. An example of this is the very similar extraction profiles of field soils G6 and AL despite having quite different sources of Pb contamination (lead arsenate and lead paint, respectively).

Table 4
Different fractions ("A" represents exchangeable Pb, "B" represents reducible Pb, "C" represents oxidizable Pb, and "D" represents residual Pb) of lead (mg kg^{-1}) in polluted soil sample from the industrial site (LP44), old apple orchard (G6), urban back yard (AL), and from urban community gardens in New York City (GP, GA1, GA2).

Soil sample	A	B	C	D[a]	Sum[b]	Total	Recovery (%)[c]
LP44	142 ± 1.4	1094 ± 22	207 ± 0	256	1443	1699 ± 40	100
G6	36.5 ± 2.7	1072 ± 11	190 ± 13	0	1298	1269 ± 16	102
AL	35.6 ± 4.0	886 ± 70	323 ± 44	0	1245	1229 ± 40	101
GP	22.8 ± 0	522 ± 26	158 ± 1.6	477	703	1179 ± 32	100
GA1	18.5 ± 1.3	124 ± 6.6	26 ± 0.2	183	168	351 ± 20	100
GA2	150 ± 0.5	807 ± 19	132 ± 14	18	1088	1106 ± 24	100

[a] D as 0 when sum above total.
[b] Sum=A+B+C.
[c] Recovery (%)=A+B+C+D/Total.

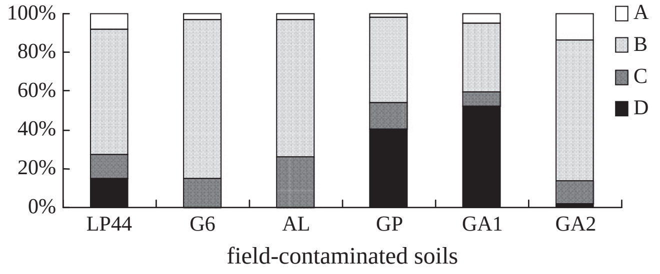

Fig. 2. Distribution of lead in different fractions ("A" represents exchangeable Pb, "B" represents reducible Pb, "C" represents oxidizable Pb, and " D" represents residual Pb) in polluted soil sample from the industrial site (LP44), old apple orchard (G6), urban backyard (AL), and from urban community gardens in New York City (GP, GA1, GA2).

Two of the urban garden soils had relatively high residual (non-extractable) fractions of Pb, which could possibly be explained by the high organic matter contents of these soils. It was shown earlier (see Fig. 1) that a high-organic matter soil, after spiking with soluble Pb, had high residual Pb similar to these garden soils.

The more labile form of Pb (fraction A) was most prominent in soils from the industrial site (LP44) and the urban community garden (GA2). It is perhaps relevant that these 2 soils had the highest pH of soils tested, and therefore may have contained Pb in the form of carbonates or hydroxycarbonates, minerals which are particularly susceptible to dissolution into fraction A. The orchard soil (G6) is known to contain Pb in the form of Pb arsenate [20], yet the extraction profile is dominated by the "oxide" fraction (B), a further indication that the acid hydroxylamine hydrochloride extractant has poor selectivity for the forms of Pb dissolved. The urban yard and garden soils are likely to have contained Pb from paint pigments (particularly "white lead" or hydrocerussite) and from leaded gasoline, but the sequential extraction data in Fig. 2 do not suggest that these different sources of Pb produce unique extraction profiles.

3.3. *SEM images of particles from soils (LP44 and AL)*

The mapping (Fig. 3) showed that the spatial distribution of phosphorus (P), chlorine (Cl) and lead (Pb) were consistent with the presence of the stable lead phosphate, pyromorphite. Under long-term aging in soil, the reaction:

$$5Pb^{2+}+3H_2PO_4^-+Cl^-=Pb_5(PO_4)_3Cl(s)+6H^+$$

would eventually convert more soluble forms of soil Pb to pyromorphite, a very insoluble mineral which is chemically and biologically stable in surface soil environments. This process may immobilize soil Pb and reduce its bioavailability [21–23]. Through this reaction, labile Pb species would be transformed to relatively insoluble forms. However, this potential stabilization of Pb was not apparent in soils LP44 and AL from the observed

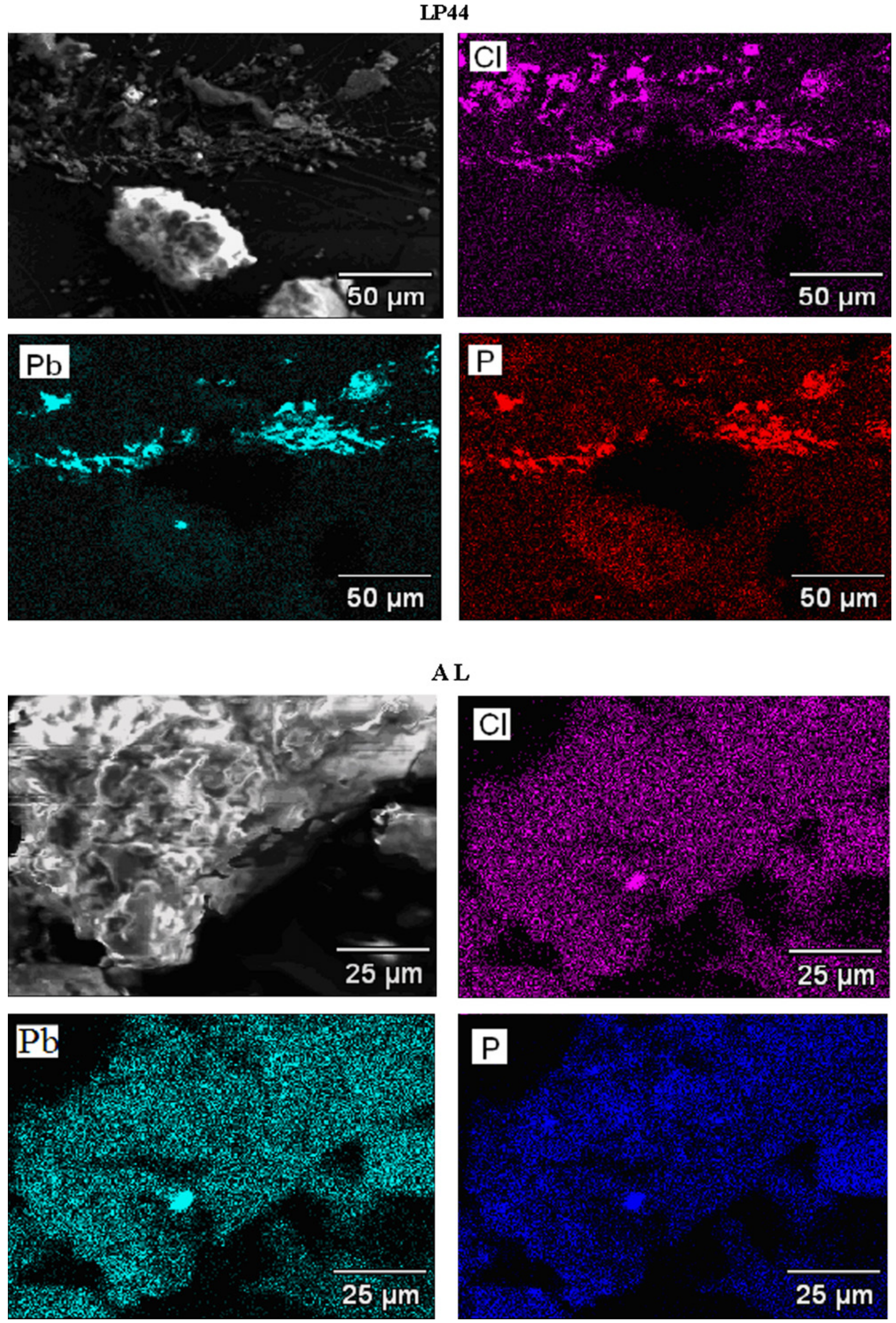

Fig. 3. Mapping of specific elements in polluted soil samples from the industrial site (LP44) and urban backyard (AL).

distribution of lead in different extractable fractions (Fig. 2) employed in the modified BCR test. We suggest that much of the Pb in these contaminated soils remains in forms more chemically labile than pyromorphite, accounting for the observed results.

4. Conclusions

The present study showed that the BCR sequential extraction for three forms of Pb added to the mineral soil (phosphate, hydroxycarbonate, nitrate salt) did not produce extraction profiles sufficiently different to distinguish different forms of Pb in soil. Furthermore, in comparing sequential extraction of mineral soil, organic soil and FeOOH spiked with Pb nitrate, the stability of Pb–organic matter complexes was demonstrated despite the low pH of BCR sequential extraction. It appears that soil organic matter is capable of retaining Pb in more stable forms than soil minerals, Fe oxides or precipitates of phosphate and carbonate.

The field soils contaminated by different sources of Pb did not show very unique sequential extraction profiles. Generally a very small part of the total Pb was in the most labile ("exchangeable") fraction, with most of the Pb extracted into the operationally-defined “reducible” fraction.

These results indicate that the BCR sequential extraction procedures, and other similar sequential extraction procedures using aggressive extracting agents, are unlikely to reliably predict the lability and bioavailability of Pb in soils with different sources or forms of Pb contamination.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.talanta.2013.01.008.

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