

Modelling of Heavy Metal Mobility in Delhi Soils before and after Remediation with Green Amendment Rock Phosphate using Sequential Extraction, TCLP and PBET

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Abstract: The particular behaviour and fate of trace metals like lead (Pb) are governed by a range of different physico-chemical processes and thereby the resulting different physico-chemical forms rather than by its total concentration. These dictate its availability and mobility in the soil or sediment system. In the solution phase, the chemical form of a lead determines the biological availability and chemical reactivity (sorption/desorption, precipitation/dissolution) towards other components of the system. Sequential methods primarily are intended to understand the particular environmental behaviour of lead, present in a variety of forms and in a variety of matrices. After applying cost effective green remediation technique of chemical immobilization by phosphate amendment the mobility factor of lead reduced considerably. This has been inferred from the sequential extraction studies as the lead has moved from the exchangeable sites to the residential sites. The results have been further confirmed by PBET and TCLP studies.

Key words: Heavy metals, mobility, speciation, sequential extraction, fractionation, bioavailability, soil, PBET, TCLP, immobilization.

Introduction

Total metal content consists of fractions of different solubility and availability. Metal bioavailability depends on its chemical behaviour, soil properties and the individual characteristics of the receptor (organism or plant). The concentrations of heavy metals (e.g., Pb) in soil are associated with biological and geochemical cycles and are influenced by anthropogenic activities such as agricultural practices, industrial activities, and waste disposal. Heavy metals that accumulate in soils in the vicinity of industries related to automobiles, paint and battery recycling workshops, can enter food chains and thus directly influence man's health. The risk of

occurrence of these elements is generally defined by their mobile fractions rather than their total concentration in soils (Ni meëek et al., 1998). The key to the determination of heavy metal mobility and thus the bioavailability is a precise definition of their distribution into separate soil fractions. Heavy metals bind to the different components of the soil in various forms and sequential extraction provides this information, thereby enabling identification and quantitative determination of various forms of the same chemical element. This extraction procedure takes advantage of the solubility mechanisms in water, ionic exchange, oxidation and reduction processes, as well as complexation and digestion of mineral and organic soil components. Separated fractions are defined in a

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conventional and operational manner, and as such present a certain approximation in describing different forms of metals found.

Soils consist of heterogeneous mixtures of organic and inorganic substances and the binding mechanisms for metals vary with the composition of the soil. The ecological effects of heavy metals in soil are closely related to the distribution of species in the solid and liquid phases of the soil. Depending on their origin, trace elements exist in different mineral forms and chemical compounds, and in different combinations with mineral and organic components of soil and sediments which may vary according to various conditions: for example, pH has an influence on the trace-metal forms, and other parameters affecting their concentration levels, mobility and transformation and accumulation processes in the ecosystem are redox conditions, oxidation states, temperature, the presence of organic matter, and microbiological activity. All these factors strongly influence the biogeochemical cycles of elements in our environment. In acidic soils, simple cations and complexes of chlorides and sulphates usually exist, while in neutral and slightly alkaline conditions carbonate complexes dominate (Kotand, 2000)

In a sequential extraction procedure (SEP), a sample is treated with a series of progressively harsher reagents to dissolve increasingly refractory forms. Ideally, the reagents are chosen to selectively attack a specific soil compartment with minimal dissolution of nontargeted phases. The sequential extraction by Tessier allows separating heavy metals into the following fractions: (i) exchangeable, (ii) bound to carbonates, (iii) occluded in Fe and Mn oxides and hydroxides, (iv) forming organo-metallic complexes and sulfides, (v) strongly bound to silicates (Tessier et al., 1979). Heavy metal mobility is determined by the amount of the metal present in fractions (i) and (ii). Kabala and Singh (2001) highlight the fact that heavy metals in these fractions are mobile or easily mobilizable in natural soil conditions and therefore represent the highest environmental risk. The same conclusion was reached by Zhang et al. (2003).

The potential mobility of metal contaminants stabilized on rock phosphate is assessed with use of the EPA Toxicity Characteristic Leaching Procedure (TCLP) Test. The potential bioavailability of metal contaminants stabilized on apatite phase is evaluated by the *in vitro* bioaccessibility extraction procedure developed by Ruby and co-workers' (SOP 110499), using their well-tested synthetic gastric juice cocktail approved by Environmental Protection Agency (EPA).

The goal of our study was to evaluate the status of, and indicate factors governing, metal bioavailability in

long term contaminated soils of the industrial area in Delhi. The study was performed on a representative group of soils with a wide range of metal content. Various parameters for the characterization of metal bioavailability were used, e.g., sequential extraction and *in vitro* test. The levels of Pb contamination and mobility in unamended and amended soils in the exchangeable and resident fractions of the soil were determined. The soils have been amended with rock phosphate (RP). Mobility has been determined for various soil samples collected from an industrial site in the National Capital Region (NCR) of Delhi.

The systems that were examined in the absence of phosphate behaved, for the most part, adequately according to the operational definitions of the extraction procedure.

However, when the samples were amended with phosphate, results were drastically changed with a significant shift of extractable Pb to the residual phase. This redistribution was due to pyromorphite formation during the extraction procedure. The objective of determining the effectiveness of remediation of Pb by its immobilization in a soil using a mixture of PR and PA as P source was accomplished by these extraction studies. The concentration of lead in the exchangeable fraction considerably reduced. Rock phosphate has been shown to effectively immobilize Pb in contaminated soils (Basta et al., 2001; Boisson et al., 1999; Cotter-Howells et al., 1996; Hettiarachchi et al., 2000; Ma et al., 1995; Ryan et al., 2001; Yang et al., 2001). Its effectiveness is based on P-induced conversion of reactive Pb into less labile lead pyromorphite. In the presence of adequate P, lead-phosphates are at least 44 orders of magnitude less soluble than galena (PbS), anglesite (PbSO₄), cerussite (PbCO₃), and litharge (PbO), which are common Pb minerals in contaminated soils (Laperche et al., 1996; Ruby et al., 1994). Natural lead-phosphate minerals have been identified in contaminated soils (Ruby et al., 1994; Davis et al., 1998; Cotter-Howells et al., 1996). In light of their intrinsically low solubilities, efforts have been made to form lead phosphates in lead-contaminated soils through P addition. Due to rapid kinetic formation of pyromorphite, the extraction method cannot prove that pyromorphite is not formed during the extraction procedure (Scheckel et al., 2003, 2004).

Materials and Method

Site Characterization

The soil samples used for this study were collected from a Pb contaminated site in an industrial area of Naraina and Mayapuri in the NCT of Delhi. The soil samples (0–

30 cm) were collected using a stainless steel auger. Industrial activities, which included a recycling operation for lead batteries, industrial activities, which included paints and recycling operation for lead batteries, have all contributed to the contamination of this site. The total Pb concentrations in the soil ranged from 1875 to 6625 mg kg⁻¹. Lead concentration decreased with soil depth, with the majority of the Pb present near the soil surface (0–20 cm).

Soil Analysis

Sample preparation

All soil samples were air dried to constant weight, sieved through a stainless sieve (diameter 2 mm) and homogenized. An aliquot part of 30 g obtained by quartering was finely ground in a planetary agate mortar (Fritsch, Germany) and used for analytical procedures (total concentrations of Pb). The fraction < 2 mm was used for sequential extractions in order to preserve in site soil composition.

Metal concentration

They were then digested using the microwave digestion procedure (USEPA Method 3051) for total Pb concentration using scientific microwave *Anton Paar Multiwave 3000*. The clean soil sample was collected from Himalayan region. Concentrations of Pb in the extracts were analysed with AAS (acetylene air flame) (*Perking Elmer A Analyst -100*) with addition calibration.

Electrochemical Properties

Electric conductivity (EC) and pH were measured in water suspensions and in 0.01 M CaCl₂. (*Elico CM 180* and *Elico LI 127*)

The Pb contents and pH in the contaminated soils varied in range 2400–6625 mg kg⁻¹, and 8.11, respectively.

Clay Content

The clay content was determined by sedimentation analysis using a hydrometer after dispersion in sodium polyphosphate.

All these properties for the given soil have been presented in Table 2.

Heavy Metal Mobility

In order to determine Pb mobility, the classical sequential extraction by Tessier et al. (1979) was used. 1 g of dried sample was placed into 100 ml PE centrifuge tubes (c.f. Table 1). The extraction procedure was as follows:

- (A) **Exchangeable fraction.** Samples were extracted with 8 ml of 1M MgCl₂ (pH = 7.0) and continually shaken for a period of 1 hour (at 20°C).
- (B) **Fraction bound to carbonates.** The residue left after fraction A was extracted with 8 ml of 1M NaOAc (pH = 5.0) and continually shaken for a period of 5 hours (at 20°C).
- (C) **Fraction bound to Fe and Mn oxides and hydroxides.** The residue left after fraction B was extracted with 20 ml of 0.04M NH₂OH.HCl in 25% acetic acid (CH₃COOH) and occasionally shaken for a period of 6 hours (at 96 ± 3°C).
- (D) **Fraction bound to sulfides and to organic matter.** 3 ml of 0.02M HNO₃ and 5 ml of 30% H₂O₂ were added to the residue left after fraction C. A pH of 2.0 was achieved using HNO₃. This mixture was kept at 85 ± 2°C for a period of 5 hours and occasionally shaken. 5 ml of 30% H₂O₂ were added after 1.5 and 3 hours. 5 ml of 3.2M NH₄OAc in 20% HNO₃ were added into the cooled mixture.
- (E) **Residual fraction.** 15 ml of HNO₃ and 2 ml of H₂O₂ was added to the residue of fraction D and digested using scientific microwave. Samples were analysed using ICP.

Table 1: Sequential extraction scheme

Extraction step	Extracted metal species	Extracting solution	Extraction condition
1	Exchangeable	1 M MgCl ₂ , pH 7	1 h, 20°C
2	Weakly complexed and bound to carbonate	1 M NaCH ₃ CO ₂ , pH 5	5 h, 20°C
3	Bound to Fe oxides of low crystallinity and Mn oxides	0.04 M NH ₂ OH—HCl in 25% (v/v) CH ₂ CO ₂ H	6 h, 96°C
4	Bound to organic matter and sulphides	30% H ₂ O ₂ + HNO ₃ (pH 2), 3.2 M CH ₃ CO ₂ NH ₄ in 20% (v/v) HNO ₃	5 h, 85°C
5	Residual	15 ml HNO ₃ + 2 ml H ₂ O ₂	Microwave digestion

The Toxicity Characteristics Leaching Procedure (TCLP) was used to evaluate the efficiency of P amendments on lead toxicity (USEPA, 1992; 1995).

PBET Procedure: Bioavailable Pb in the treated and treated soil samples was determined by a modified PBET procedure (Ruby et al., 1996).

Results and Discussion

The extraction results showed high variability between soils. Sequential extraction procedures provide useful information for risk assessment, since the amount of metals mobilized under different environmental conditions can be estimated. The mobility and bio-availability of the metals decrease approximately in the order of the extraction sequence.

Trace elements undergo a wide variety of chemical reactions in soil and soil-residual systems. Sorption is an important chemical process that regulates partitioning of trace elements between solution and solid phases in soils. Soils with a large proportion of Pb in water-soluble and exchangeable fractions are potentially of greater hazard when compared to soils where Pb primarily resides in the residual fractions.

All treatments including control were done in duplicate. Agreement between the duplicate treatments was good for the total extractable lead and the various fractions.

Lead in Different Fractions before Remediation

The Pb fractions expressed as percentages of the sum of individual chemical fractions are presented in Table 3. For Pb, the percentage of Pb fractions follows the order: residual > amorphous Fe oxides > exchangeable > organic > carbonate. The highest percentage of Pb was found to be in the residual fractions. The chemical nature of the different fractions is very complex. The various fractions and the chemistry involved are as under.

Exchangeable and Carbonate Bound Fraction

The acid-soluble fraction contains the metals that are precipitated or co precipitated with carbonate. The carbonate form is a loosely bound phase and is liable to change with environmental conditions, so this phase is

susceptible to changes in pH, and is generally obtained using a mild acid. The acid-soluble fraction generally contains a relatively small percentage of the total metal content. This reagent is capable of dissolving carbonates without attacking organic matter, Fe and Mn oxides and aluminosilicates. The exchangeable fraction may indicate which metals are more mobile. The exchangeable Pb in the surface layers was high (15.7% and 6%). This relatively large pool of Pb in the exchangeable fraction suggests that the soil presents a potential hazard to human health and the environment through direct soil ingestion and leaching to groundwater.

Fraction Bound to Fe Oxides of Low Crystallinity and Mn Oxides

Iron and manganese oxide soil minerals are important sinks for trace elements in soil (McBride, 1994; Sposito, 1986) and residual-amended soils (Essington and Mattigod, 1991; Hettiarachchi et al., 2003; Lombi et al., 2002; Scheckel and Ryan, 2004). Trace element sorption by the oxide surface is a highly pH-dependent process described by the following chemical reactions (Figure 1).

cation adsorption: $\text{Fe—OH} + M^+ \longrightarrow \text{Fe—OM} + \text{H}^+$

where Fe—OH is an iron oxide surface, *M* is a trace element cation.

This fraction represents the content metals bound to iron and manganese oxides that are released when the matrix is subjected to reducing conditions (Wong et al., 2002). These oxides are found in large amounts in soil and sediment, but are less abundant in sewage sludge. The toxic heavy metal lead is present in a considerable amount (19.18%). Studies have indicated that adsorbed metal is strongly bonded and not readily desorbed from these oxide surfaces. A recent review of trace element sorption by oxides (Brown and Parks, 2001) shows that Fe and Mn oxides have a much greater adsorption capacity (e.g., high capacity scavengers) compared with Al oxides and other clay minerals. Molecular scale X-ray spectroscopic studies show that the strong bonding of Pb, Cu, Co, Cr, Mn Ni, and Zn to these oxide surfaces is due to formation of inner-sphere metal surface complexes and formation of metal hydroxide precipitate

Table 2: Selected physico-chemical properties of the contaminated and control soil samples*

Reference soil	pH ^a	Conductance (μS)	OM ^b	Sand	Silt	Clay	Pb _{Tc} (mg kg^{-1})
Contaminated sample	8.11	0.39	2.63	77.5	5	17.5	6625

*Data represents an average of twelve replicates with a standard deviation. ^apH was determined with a 1:1 ratio of soil/water. ^bOrganic matter. ^cTotal concentration.

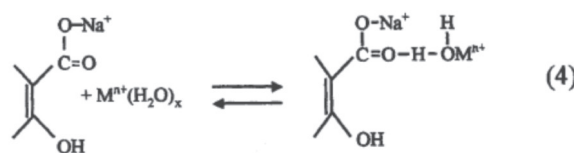
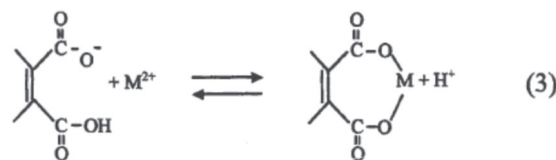
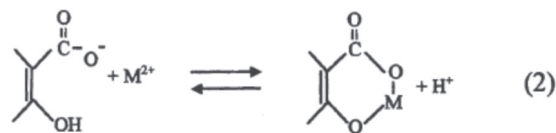
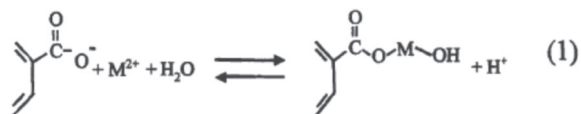


Figure 1: Mechanisms of complexation of heavy metal cations with natural organic matter (NOM)
(extracted from Senesi, 1992).

phases (Brown and Parks, 2001). New solids found after metal ion reactions with soil materials, including metal silicates and mixed double hydroxides with Al, can substantially reduce metal solubility and availability (Scheckel and Sparks, 2001). Sorption by metal oxides is a major mechanism for removal of trace element cations (i.e., Pb, Cu, Zn, Cd, Cr, Cu, Pb, Hg, Ni, and Zn) and trace element (Sparks, 2003).

Fraction bound to organic matter and sulphide

The organic fraction released under oxidizing conditions is not considered mobile and bioavailable, metals are incorporated into stable high molecular weight humic substances, which release small amounts of metals over long time periods. The toxic metals bound to organic matter and sulphides can easily be released under oxidizing conditions, so an oxidation process was used

to leach the metals associated with this phase. In the unamended soil, lead was present as 13.22% of the total metal detected.

Residual Fraction

The residual or nonextractable metals are retained within the crystal lattices of minerals and inside crystallized oxides (Singh et al., 1998). The highest percentages of all the heavy metals were found to be in the residual fractions. This trend may be explained by residence time effect which may reduce metal mobility and bioavailability due to complexation, adsorption and precipitation of metal ions in the soil particle surface. Lu et al. (2005) investigated the time effect on the fractionation of heavy metals in soils and found that soluble metals added were transformed from easily extractable fractions to more stable fractions. In the unamended soil lead was present as 45.74% of the total metal extracted in the top soil.

Lead in Different Fractions after Remediation

Lead is present in various oxidation states in soil. The solubility of the Pb compounds (lead acetate > PbSO₄ > PbCO₃ > PbS ~ pyromorphite) followed trends based on known solubility data. Likewise, we anticipated the steps (fractions) of the sequential extraction procedure to variably induce solid-phase solubility as exchangeable (F1) < carbonate (F2) < iron/manganese oxide (F3) < organic matter (F4) < residual (F5). These assumptions were confirmed from our findings. Results for the sequential extraction of the Pb contaminated soil samples with and without commercially available rock phosphate have been summarized in Table 3 and represented in Figures 2–4. The majority of geochemically stable and less bio-available pyromorphite is expected to get formed in the residual fraction. After applying rock phosphate as a green amendment, substantial amount of metal was still associated with the Fe and Mn oxides and hydroxides of the soil. This can be explained on the basis of sorption by metal oxides. This is a major mechanism for removal

Table 3: Distribution of lead (Pb) among the individual fractions from the five step sequential extraction for contaminated soil sample with and without remediation

Soil	Exchangeable fraction (%)	Carbonate bound fraction (%)	Fe-Mn oxide bound fraction (%)	Organic bound fraction (%)	Residual fraction (%)
Control	15.7	6	19.18	13.22	45.74
Soil + Rock Phosphate (1 wk)	10.8	5.2	17.08	14.8	51.99
Soil + Rock Phosphate (4 wk)	4.6	4.1	25.4	27.9	37.8

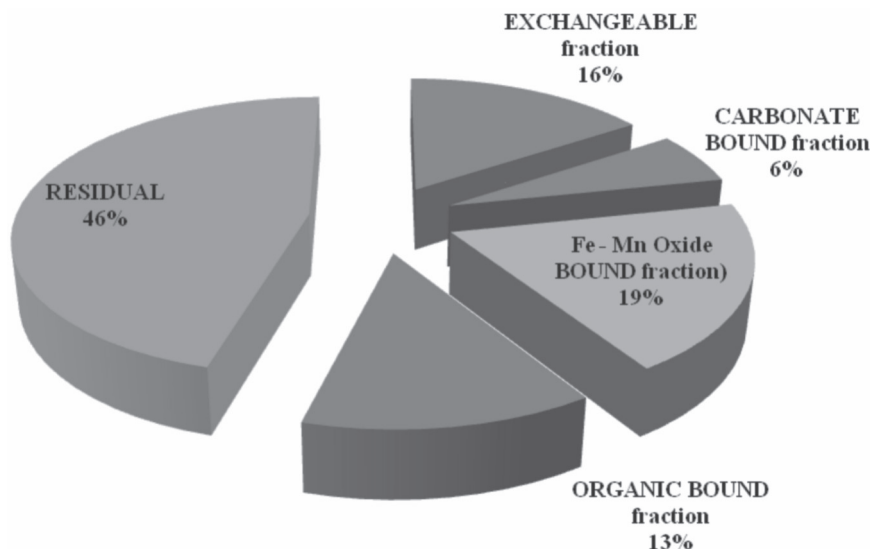


Figure 2: Distribution of lead (Pb) among the individual fractions from the five step sequential extraction for control soil sample.

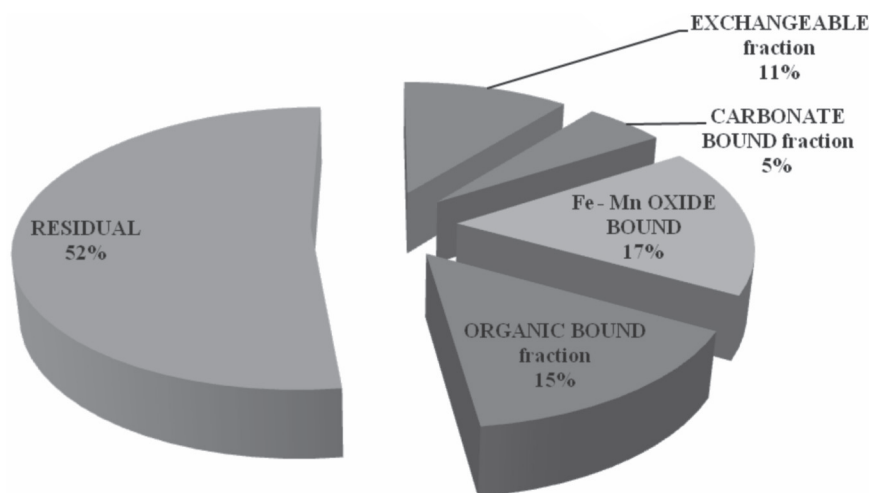


Figure 3: Distribution of lead (Pb) among the individual fractions from the five step sequential extraction for soil sample with phosphate amendment (after 1 week).

of trace element cations (i.e., Pb, Cu, Zn, Cd, Cr, Cu, Pb, Hg, Ni, and Zn) and trace element (Sparks, 2003).

At the end of fourth week the residual fractions decreased while the organic and Fe–Mn forms of Pb fractions increased, and the water soluble and exchangeable fractions of Pb was reduced by the treatment as well. This is in agreement to the fact that adsorbed metal is strongly bonded and not readily desorbed from these oxide surfaces. These oxides are promising remediation materials (Brown and Parks, 2001). Zhang et al. (2003) found out that water soluble and exchangeable Pb were more leachable or bioavailable; exchangeable and

carbonate fractions of Pb were responsible for Pb phytoavailability (Samaras and Tsadilas, 1997).

This result greatly supported the results of Pb leachability (TCLP) as presented in Table 4 and Figure 5, where it was observed that leachable Pb was significantly reduced by the treatments in the contaminated soil. Also, the results provided a good stand for the in vitro availability (PBET) results in the figure. It was obvious that the reduction of water soluble and exchangeable soil Pb fractions could be responsible for the reductions of the in vitro availability of Pb in the urban soil.

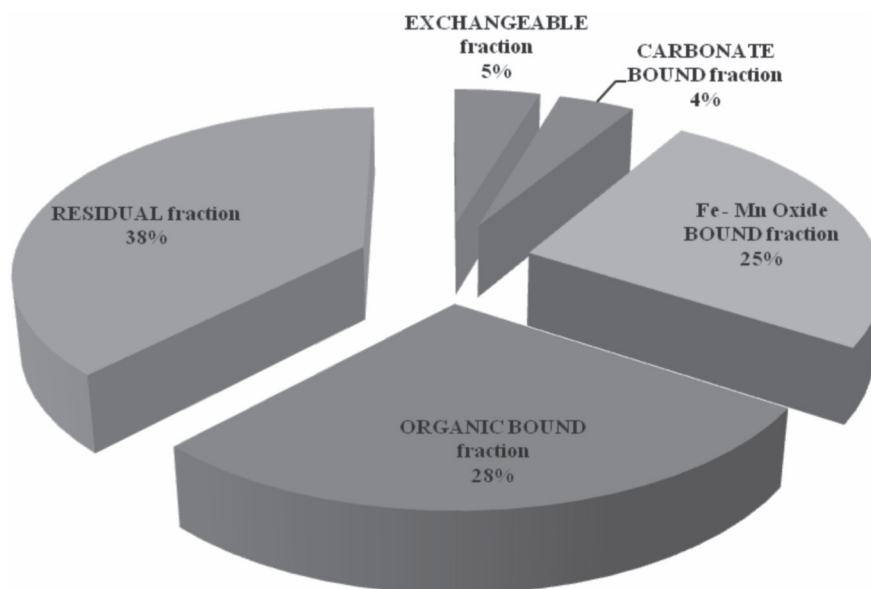


Figure 4: Distribution of lead (Pb) among the individual fractions from the five step sequential extraction for soil sample with phosphate amendment (after 4 weeks).

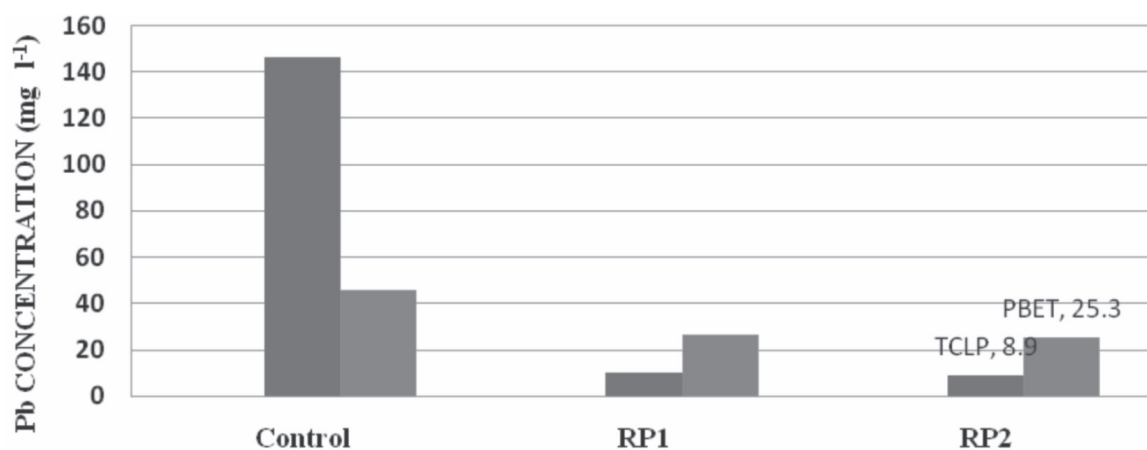


Figure 5: Lead (Pb) concentration (mg l⁻¹) in TCLP solution and PBET of soil with and without treatment.

Table 4: Lead concentration (mg l⁻¹) in TCLP solution and PBET of soil with and without treatment

Contaminated soil	Control	RP1	RP2
TCLP	145.7	9.8	8.9
PBET	45.8	26.5	25.3

Control – without remediation

RP1 – phosphoric acid (PA) + LAB GRADE Ca(H₂PO₄)₂

RP2 – phosphoric acid + COMMERCIAL rock phosphate (RP)

TCLP Lead in Soil Profiles

The concentration of Pb from phosphorus amended soils decreased sharply and remained below their regulatory levels in drinking water. Without P treatments, TCLP-extractable Pb concentrations in the surface soils far exceeded 5 mg⁻¹ critical level of hazardous waste (USEPA, 1995). This is possibly because substantial amount of the Pb is in the exchangeable and carbonate fraction, which would readily dissolve in the acidic TCLP solution. Phosphate amendment was effective in reducing the TCLP Pb to below the critical level in the surface soil samples. These results are of great significance with respect to the disposal of the soil, because they show that P amendments can change the soil to a material that would be considered non-hazardous (Table 4).

Lead Bioavailability

Incidental ingestion of Pb-contaminated soil has been reported as a primary exposure pathway to humans for elevated blood Pb levels. A physiologically-based extraction test has been used to estimate Pb bioavailability (in vivo), which simulates Pb dissolution under gastrointestinal conditions using a chemical extraction (Yang et al., 2001). Lead bioavailability in contaminated soils has been shown to vary with its mineralogical forms (Davis et al., 1993). In vivo and in vitro assays have indicated that the mammalian gastrointestinal availability of Pb is controlled by the form and relative solubility of Pb solids (Ruby et al., 1996). The PBET has been used to assess the Pb bioavailability in a contaminated soil after receiving various amounts and sources of P (Hettiarachchi et al., 2000). The bioavailability of soil Pb is associated with its solubility and dissolution rate in the gastrointestinal tract. Bioavailable Pb in the contaminated soil based on PBET was reduced after P application. The control soil showed 45–49 mg kg⁻¹ of bioavailable Pb while P-treated soils showed reduction of PBET-Pb by 25–42%, which was similar to the 25–35% reduction reported by Hettiarachchi et al. (2000) and 39% by Yang et al. (2001) (Table 4).

Conclusions

Our results strongly demonstrate that rock phosphate was effective in reducing Pb solubility and bioavailability through dissolution of phosphate rocks and precipitation of pyromorphite. The effective and rapid Pb²⁺ immobilization from solution and contaminated soils by phosphate rock, the limited effects from other minerals,

anions, and cations, the apparent environmental stability of the reaction products, along with the ready availability and low cost of hydroxyapatite or phosphate rock suggest that this approach might have great merit for cost-effective in situ immobilization of Pb contaminated water, soils and wastes.

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Asian Journal of Water, Environment and Pollution



Aims and Scope

Asia, as a whole region, faces severe stress on water availability, primarily due to high population density. Many regions of the continent face severe problems of water pollution on local as well as regional scale and these have to be tackled with a pan-Asian approach. However, the available literature on the subject is generally based on research done in Europe and North America. Therefore, there is an urgent and strong need for an Asian journal with its focus on the region and wherein the region specific problems are addressed in an intelligent manner. In Asia, besides water, there are several other issues related to environment, such as; global warming and its impact; intense land/use and shifting pattern of agriculture; issues related to fertilizer applications and pesticide residues in soil and water; and solid and liquid waste management particularly in industrial and urban areas.

Asia is also a region with intense mining activities whereby serious environmental problems related to land/use, loss of top soil, water pollution and acid mine drainage are faced by various communities.

Essentially, Asians are confronted with environmental problems on many fronts. Many pressing issues in the region interlink various aspects of environmental problems faced by population in this densely habited region in the world. Pollution is one such serious issue for many countries since there are many transnational water bodies that spread the pollutants across the entire region. Water, environment and pollution together constitute a three axial problem that all concerned people in the region would like to focus on.

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