



Article

Soil Lead Concentration and Speciation in Community Farms of Newark, New Jersey, USA

Omanjana Goswami and Ashaki A. Rouff *

Department of Earth & Environmental Sciences, Rutgers University, Newark, NJ 07102, USA;
omanjana.goswami@rutgers.edu

* Correspondence: ashaki.rouff@rutgers.edu; Tel.: +1-973-353-2511

Abstract: Farmed urban soils often bear legacies of historic contamination from anthropogenic and industrial sources. Soils from seven community farms in Newark, New Jersey (NJ), USA, were analyzed to determine the concentration and speciation of lead (Pb) depending on garden location and cultivation status. Samples were evaluated using single-step 1 M nitric acid (HNO₃) and Tessier sequential extractions in combination with X-ray absorption fine structure spectroscopy (XAFS) analysis. Single-step extractable Pb concentration ranged from 22 to 830 mg kg⁻¹, with 21% of samples reporting concentrations of Pb > 400 mg kg⁻¹, which is the NJ Department of Environmental Protection (NJDEP) limit for residential soils. Sequential extractions indicated lowest Pb concentrations in the exchangeable fraction (0–211 mg kg⁻¹), with highest concentrations (0–3002 mg kg⁻¹) in the oxidizable and reducible fractions. For samples with Pb > 400 mg kg⁻¹, Pb distribution was mostly uniform in particle size fractions of <0.125–1 mm, with slightly higher Pb concentrations in the <0.125 mm fraction. XAFS analysis confirmed that Pb was predominantly associated with pyromorphite, iron–manganese oxides and organic matter. Overall results showed that lowest concentrations of Pb are detected in raised beds, whereas uncultivated native soil and parking lot samples had highest values of Pb. As most of the Pb is associated with reducible and oxidizable soil fractions, there is a lower risk of mobility and bioavailability. However, Pb exposure through ingestion and inhalation pathways is still of concern when directly handling the soil. With increasing interest in urban farming in cities across the USA, this study highlights the need for awareness of soil contaminants and the utility of coupled macroscopic and molecular-scale geochemical techniques to understand the distribution and speciation of soil Pb.

Keywords: community farms; urban soil; Pb contamination; metal sorption; spectroscopy; XAFS; Newark, NJ



Citation: Goswami, O.; Rouff, A.A. Soil Lead Concentration and Speciation in Community Farms of Newark, New Jersey, USA. *Soil Syst.* **2021**, *5*, 2. <https://doi.org/10.3390/soilsystems5010002>

Received: 3 November 2020

Accepted: 22 December 2020

Published: 29 December 2020

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2020 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Communities across the world are adopting urban farming to increase access to healthy, low cost, locally grown produce, and reclaim abandoned or unused plots of land. Community farms add a social and cooperative element to urban areas, create green spaces, and increase the overall aesthetic value of their surroundings [1–3]. In the United States (U.S.), urban farming has seen a growth of over 30% in the last 30 years, enhancing local food security, especially in disenfranchised communities [2–4]. Household food gardening in the U.S. increased from 36 to 42 million households from 2008 to 2013, recording a growth of 17% in 5 years, with the largest increase observed in urban areas [5]. While urban farming practices are on the rise and are expected to keep growing, the challenges of farming in urban soils includes access to land and water, and the environmental health of the soil, including the prevalence of historic contaminants such as heavy metals.

Lead (Pb) is one of the most common legacy heavy metal contaminants in urban soils [6], originating primarily from anthropogenic sources. Approximately 5–6 million metric tons of Pb was used as a gasoline additive since the 1920s until it was banned in 1996, and ~75% of this Pb was released into the atmosphere [7,8]. Millions of tons

of Pb deposited from the atmosphere, and from sources such as Pb-based paint, persist in soils. The average concentration of Pb in uncontaminated surface soils in the U.S. is $\sim 22 \text{ mg kg}^{-1}$ [9] and increases to $>150 \text{ mg kg}^{-1}$ in urban areas, with some soils reporting totals $>100,000 \text{ mg kg}^{-1}$ [10–14]. Evaluation of Pb soil concentrations in several U.S. cities detected $175\text{--}7935 \text{ mg kg}^{-1}$ Pb in residential soils with a wider range of $1\text{--}9540 \text{ mg kg}^{-1}$ Pb observed for urban farmed soils [15–28]. Results from select studies showed that Pb was distributed in the organic matter fraction or in association with iron (Fe)-manganese (Mn) oxides [18], and increased Pb concentration was observed in inner city/downtown areas [19,29], around driplines of buildings [19], and was directly proportional to traffic volume and proximity to major roadways [17]. For soils with detected Pb, direct human exposure pathways include unintended ingestion or inhalation of contaminated soil, with additional indirect exposure through consumption of food grown in farmed soils [29]. Outdoor exposure through ingestion of contaminated soil because of hand-to-mouth activity can be potentially harmful, especially for young children [30]. A proven neurotoxin, Pb has been shown to impede development in infants and children, resulting in decreased neural function and impairing overall growth and brain development [31].

In soils, metal availability, toxicity, and mobility are a function of the bonding environment and speciation with corresponding soil minerals [32]. Studies of Pb and heavy metals in urban soils, as described above, commonly use sequential extraction techniques to determine metal concentration and macroscopic-scale speciation [32–34]. Spectroscopic techniques, such as X-ray absorption fine structure spectroscopy (XAFS), are less frequently used, but can elucidate molecular-scale chemical speciation of Pb in soils [35]. XAFS analyses of contaminated soil samples have shown that Pb is usually insolubilized as phosphate minerals in the low-mobility, non-bioavailable residual fractions [35,36], and XAFS can be used to investigate and confirm the dominant species and sinks of soil Pb in contaminated heterogeneous soil samples, ultimately predicting the risk of remobilization [34,35,37]. Though sequential extractions and XAFS analysis provide complementary information, limited studies have implemented both techniques to characterize Pb in soils [34,38,39], with fewer studies applying this approach to urban farmed soils in the Northeastern U.S. [23,28].

This study investigates the occurrence and speciation of Pb in soils used for urban farming in the U.S. city of Newark, the most populated city in New Jersey (NJ). Situated at the epicenter of one of the nation's busiest transportation hubs, Newark is home to the largest port in the Eastern U.S., the busiest airport of the greater New York City (NYC) area, and the largest truck terminal in the country. Like in numerous other post-industrial U.S. cities, there has been a recent increase in urban farming in Newark. As of 2014, 74 plots had been leased out through an Adopt-a-Lot program managed by the City of Newark's Office of Sustainability. There are an estimated 100 farmers in Newark [40], operating farms ranging in size from ~ 200 to $12,000 \text{ m}^2$, including many established in school backyards and maintained by students. While urban farming practices to revitalize abandoned and vacant lots in Newark are increasing, there is no central regulation on testing soils before establishing a community farm to determine the soil quality and health. In this study, the occurrence, prevalence, and distribution of heavy metals, with emphasis on Pb, in Newark's farmed soils is addressed. Sampled soils were subject to sequential extraction and XAFS analysis to determine the concentration and speciation of Pb, and to infer mobility and potential bioavailability. This comprehensive geochemical understanding can be used to address the short and long-term risks of handling and farming in urban soils. This is of paramount importance when considering health risk assessment and in formulating risk management plans, ultimately contributing towards adoption of improved urban farming policies.

2. Materials and Methods

2.1. Site Description

Seven community farms (Sites 1–7) located in four of five wards of the city of Newark, NJ were selected for sampling (Figure 1). Based on Newark zoning maps [41], all farms are in primarily residential zones, with Sites 1–4 in proximity to community and regional commercial zones, and Sites 5–7 in proximity to heavy industrial zones. Farms ranged in size from 113 to 5801 m², with 6 farms practicing cultivation in raised beds, and 4 farms cultivating on native soil. Three farms had on-site composting facilities (Table 1).



Figure 1. Community farm research sites in Newark, NJ, USA represented by (a) ward; (b) sampling scheme and sample category.

Table 1. Select characteristics of Newark, NJ, USA community farms selected for sampling, including types of areas cultivated and availability of on-site composting.

Site	Area (m ²)	Type of Location	Raised Beds	Cultivation in Native Soil	Composting
1	474.1	Community and regional commercial zone	✓	✓	×
2	285.6		×	✓	✓
3	112.9		✓	×	✓
4	242.3		✓	×	×
5	5801.3	Heavy industrial zone	✓	✓	×
6	156.5		✓	×	✓
7	208.1		✓	✓	×

2.2. Soil Sampling

Sampling was conducted from 2016 to 2018 in the months of June and July to reflect periods of maximal use and plant growth. Samples were collected from raised beds (RB), cultivated native soil (CN), uncultivated soil (UN) near houses or buildings adjacent to the periphery/boundary of the farm, areas adjacent to parking lots (PL) (if present), and compost (C) (where available). Soils were sampled to a depth of 15–20 cm, up to the effective depth of crop roots [42]. Composite samples were created by mixing equal amounts of four randomly collected sub-samples from each representative area [43]. Samples were labeled according to Site (1–7) and category of sampling (RB, CN, UN, PL, C, Figure 1b, Table S1). A second sampling was conducted at Site 1 in July 2018 to identify the location of Pb hotspots (HS). Samples were collected from the same locations used to create composite sample 1-UN, but each sample was analyzed separately. All samples were brought back to the laboratory and air-dried at room temperature for 72–96 h. Samples were then sieved to <2 mm to remove large unwanted objects, plant roots, and gravel. The samples were oven-dried at 104 °C for 24 h to desorb excess moisture, gravimetric water, and volatile organic matter and stored for further analysis.

2.3. Metal Extractions

A single-step 1M nitric acid (HNO₃) extraction was performed to identify heavy metals present in the soil and to estimate extractable concentrations [44,45]. A volume of 10 mL 1 M HNO₃ and 1 g of oven-dried soil from the <2 mm fraction were added to a 30 mL HDPE bottle and placed on a reciprocal shaker set at 250 rpm [44]. After a 2 h reaction time, the extractant was recovered and filtered using 0.45 µm filter paper, and the metal content was analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES, Agilent 5110 SVDV).

Based on metal content and concentrations (Table S2) subsequent extractions and analyses focused on Pb. Sample concentrations were compared to the New Jersey Department of Environmental Protection (NJDEP) Residential Direct Contact Soil Remediation Standard (RDCSRS) of 400 mg kg⁻¹ for Pb [46]. The effect of particle size on Pb concentration was evaluated for samples with Pb > 400 mg kg⁻¹ from Sites 1 and 2. Approximately 20 g of the <2 mm fraction was dry sieved to <0.125 mm, 0.25 mm, 0.5 mm, and 1 mm using a field sieve, extracted using 1 M HNO₃ as described above and analyzed for Pb by ICP-OES.

Sequential extractions following the method by Tessier et al. (1979) [47] were performed for all samples. The extractions targeted chemically associated Pb in the exchangeable, acid-soluble, reducible and oxidizable soil fractions (Table 2). The final extraction step in the method targets the residual fraction and was omitted as the metal is sequestered in low-mobility, insoluble phases, and therefore is unlikely to be readily released and available to plants under normal environmental conditions [48]. A mass of 1 g of soil was weighed into a 30 mL HDPE bottle with the requisite reagents (Table 2) and placed on a reciprocal shaker set at 250 rpm. For extractions requiring elevated temperature (85–96 °C), a thermostat water bath with a reciprocal shaker was used. After each extraction, the soil and extractant mixture were allowed to sit for 30–45 min until a clear supernatant was observed. The supernatant was collected and filtered using 0.45 µm filters, then acidified with 5N HNO₃. The residual solid remaining after each extraction step was washed with 10 mL deionized water and dried overnight before proceeding to the successive extraction step. Extractants were acidified for preservation and analyzed for Pb by ICP-OES. All single-step and sequential extractions were performed in duplicate.

Table 2. The Tessier method for sequential extraction of soils, including the targeted soil fraction, extraction reagents and the predicted metal speciation and mobility.

Fraction	Extraction	Metal Speciation	Factors Influencing Metal Mobility
Exchangeable	1 M MgCl ₂ pH 7.0 1 h at 25 °C	Dissolved cations	Changes in cation composition, sorption chemistry and ion exchange; high mobility
Acid-soluble	1 M NaOAc pH 5 5 h at 25 °C	Bound to carbonates	Changes in soil pH; medium mobility
Reducible	0.04 M NH ₂ OH–HCl 25% HOAc 6 h at 96 °C	Bound to iron and manganese oxides	Changes in redox chemistry; medium mobility
Oxidizable	0.02 M HNO ₃ 30% H ₂ O ₂ 2 h at 85 °C 30% H ₂ O ₂ 3h at 85 °C 3.2 M NH ₄ OAc 30 min at 25 °C	Bound to soil organic matter	Decomposition of organic matter; medium mobility
* Residual	5:1 HF–HClO ₄	Within mineral crystal structures	Weathering and mineral decomposition; low mobility

* Extraction of the residual fraction was omitted.

2.4. X-ray Absorption Fine Structure Spectroscopy (XAFS)

Select soil samples with $>400 \text{ mg kg}^{-1}$ Pb were analyzed by XAFS to determine the molecular-scale speciation. Data were collected at Beamline 6-BMM at the National Synchrotron Light Source-II (NSLS-II), Brookhaven National Laboratory (BNL), Upton, New York. All scans were performed at the Pb LIII edge (13.035 keV) from 12.8 to 13.6 keV. The spectra were collected at room temperature at 298 K (25 °C). Data were collected in fluorescence mode using a Si (111) monochromator and a 4-element vortex silicon-drift detector with metal filters to reduce the impact of elastic scattering. Multiple scans (7–20) were recorded per sample to improve the signal-to-noise ratio. The IFEFFIT software package was used to perform data reduction, analysis and fitting [49].

3. Results and Discussion

3.1. Single-Step Extraction

The 1M HNO₃ single-step extraction is a rapid method for screening and analysis of heavy metals in soil samples and is a viable alternative to the standard EPA Methods 3050, 3051 and 3052 [10,50,51]. Using this extraction, the most commonly detected metals in the community farm soils were Pb, Cd, Cu and Zn (Table S2, Figure S1). Given the absence of health-based soil standards for community farms, the metal concentrations were compared to the NJDEP RDCSRS (Table S2) [46]. The NJDEP RDCSRS is calculated to protect human health assuming metal exposure at residential sites, and is defined as a soil remediation standard for the ingestion–dermal and inhalation exposure pathways at locations such as homes, schools (pre-K-12) and childcare centers [46]. Concentrations exceeding the standard were considered to be elevated with respect to the metal. Of the detected metals, only Pb exceeded the NJDEP RDCSRS in multiple samples (Table S2). The 1 M HNO₃ extractable Pb concentrations likely underestimate total Pb concentrations by ~33%, and therefore represent a minimum concentration for comparison with the NJDEP RDCSRS value (Table S2).

Results from single-step 1 M HNO₃ extractions are reported in Figure 2a. For all community farms, samples collected from raised beds (RB) reported the lowest Pb concentrations, ranging from 22 to 174 mg kg⁻¹. This is expected as raised beds contain clean topsoil purchased annually or every few years to avoid high levels of contaminants. In comparison, cultivated (CN) and uncultivated (UN) native soils had higher Pb concentrations ranging from 24 to 540 mg kg⁻¹, and 127 to 830 mg kg⁻¹, respectively, with three samples exceeding the NJDEP RDCSRS. Of the two sites located adjacent to a parking lot (PL), only the 3-PL sample reported elevated Pb at 438 mg kg⁻¹, suggesting historic accumulation from leaded gasoline vehicular exhaust [52]. Compost samples (2-C, 3-C, 6-C) had Pb concentrations of 72–369 mg kg⁻¹. The exact source of Pb in compost is difficult to identify, as farms usually receive biodegradable waste in large quantities from multiple commercial and non-commercial sources, that are mixed together prior to composting. Moreover, the scale and volume of composting operations is different with Site 2 having a well-established bin-composting and vermicomposting operation, whereas Sites 3 and 6 have smaller-scale operations. However, some probable sources of Pb identified in compost include plastic, paper and cardboard, and Pb-bearing food waste [53].

To estimate the extent of contamination compared to uncontaminated background levels [54,55] a contamination factor (CF) was calculated:

$$CF = C_n / B_n \quad (1)$$

where C_n is the measured concentration of the element in soil and B_n is the background value of 22 mg kg⁻¹ for Pb in uncontaminated NJ soils [9]. To determine the extent of anthropogenic Pb contamination in soil samples, the geoaccumulation index (I_{geo}) was calculated using the formula by Mueller [56]:

$$I_{geo} = \log_2[C_n / 1.5 \times B_n] \quad (2)$$

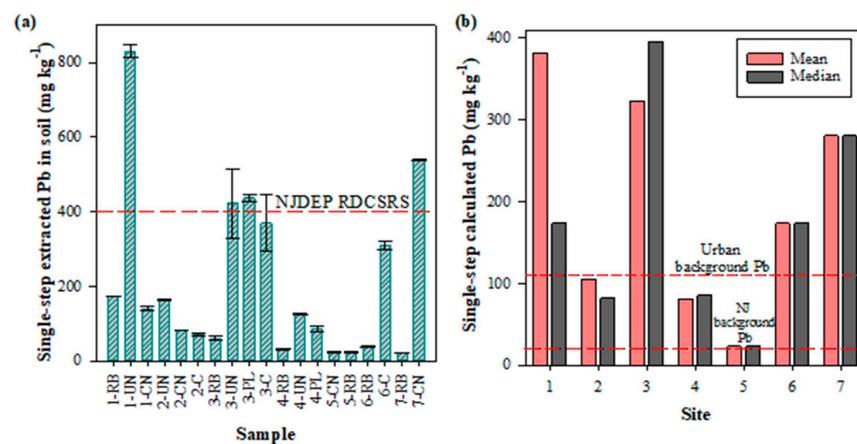


Figure 2. Single-step 1 M HNO₃ extracted Pb for (a) all samples referenced to the New Jersey Department of Environmental Protection (NJDEP) Residential Direct Contact Soil Remediation Standard (RDCSRS) (400 mg kg⁻¹); (b) the calculated mean and median of all categories for each site referenced to the background Pb in New Jersey (22 mg kg⁻¹) and urban background Pb (111 mg kg⁻¹).

The calculated CF and I_{geo} values were averaged and reported by sample category (Table 3, Table S3). Amongst all categories, RB (CF 2.7, I_{geo} 0.4) and CN (CF 8.9, I_{geo} 1.8) reported the lowest computed values for both CF and I_{geo} . While the category average I_{geo} of RB classified it as uncontaminated, according to its CF value it is still moderately contaminated. The PL, C and UN categories had a very high extent of contamination according to their CF values, corroborating a moderate to strongly contaminated classification based on their average I_{geo} . Overall, soils which are actively farmed showed slightly lower contamination as expressed by the CF and I_{geo} values of CN and RB categories, with the severity of contamination increasing in non-farmed categories known to have anthropogenic Pb inputs.

Table 3. Contamination factor (CF) and geoaccumulation index (I_{geo}) of samples.

Sample	CF	CF ^{Avg}	CF ^{Class}	I_{geo}	I_{geo} ^{Avg}	I_{geo} ^{Class}
2-C	3.3			1.1		
3-C	16.8	11.4	Very high	3.5	2.6	Moderate to strongly contaminated
6-C	14.1			3.2		
1-CN	6.4			2.1		
2-CN	3.7	8.9	Very high	1.3	1.8	Moderately contaminated
5-CN	1.1			-0.5		
7-CN	24.5			4		
3-PL	19.9	11.9	Very high	3.7	2.6	Moderate to strongly contaminated
4-PL	3.9			1.4		
1-RB	7.9			2.4		
3-RB	2.8			0.9		
4-RB	1.4	2.7	Moderate	-0.1	0.4	Uncontaminated
5-RB	1.1			-0.5		
6-RB	1.7			0.2		
7-RB	1			-0.6		
1-UN	37.7			4.7		
2-UN	7.4	17.5	Very high	2.3	3.2	Strongly contaminated
3-UN	9.2			3.7		
4-UN	5.8			1.9		

To facilitate comparison of Pb distribution amongst soil categories and individual farms, the mean and median concentration of Pb was calculated (Figure 2b, Table S4). The geogenic soil Pb concentration in the U.S. is 22 mg kg^{-1} [9] with NJ urban soils reporting a higher median Pb concentration of 111 mg kg^{-1} [57]. While the median concentration of raised bed samples at 34 mg kg^{-1} was in exceedance of naturally occurring Pb levels in NJ, the median concentration of non-raised beds (comprising UN, CN, PL and C) ranged from 112 to 310 mg kg^{-1} , exceeding both urban background and geogenic Pb level in NJ. The median concentration of Pb is lowest in Site 5 at 24 mg kg^{-1} , and highest in Site 3 at 396 mg kg^{-1} . The median Pb concentration of four of the sites exceed the background Pb value for urban soils at 111 mg kg^{-1} , implying higher Pb accumulation as a result of anthropogenic influences. While trace levels of Pb occur in soils naturally, higher concentrations are suggestive of Pb accumulation in urban soils originating primarily from historic inputs from a variety of anthropogenic sources. While soil samples with $\text{Pb} \leq 400 \text{ mg kg}^{-1}$ have been deemed safe for farming by the U.S. Environmental Protection Agency (USEPA), good farming practices established at these farms ensures that edible produce is not directly cultivated in these soils. However, physical interaction with soils with any detectable Pb might still be problematic, especially when considering Pb exposure through ingestion, inhalation, or dermal routes. The major pathway of exposure to environmental Pb is through ingestion of Pb-bearing fine particles causing elevated blood lead levels [58], followed by inhalation as the second major pathway of exposure, enabling Pb to enter the human body through the pulmonary route [30,31,45,59].

3.2. Sequential Extraction

Results from the Tessier scheme of sequential extraction of select samples are shown in Figure 3a, and all samples are reported in Table S5. To facilitate comparison of sequential extractions between samples, the soil fractions were normalized to the sum of extracted Pb and represented as a percentage of the total value (Figure 3a). The exchangeable and acid-soluble fractions represent the most mobile and potentially bioavailable concentration of metal. The concentration of Pb in the exchangeable fraction is 0 – 211 mg kg^{-1} . The exchangeable fraction consists of weakly adsorbed and easily soluble Pb and represents the amount of metal that has the tendency to be readily available for uptake by plants [48,60,61]. Lead in the acid-soluble fraction ranges from 0 to 948 mg kg^{-1} (Table S5) with 42% of samples above the NJDEP RDCSRS. Acid-soluble Pb is associated with carbonate minerals, exhibiting a tendency to mobilize upon changes in soil chemistry and lowering of pH, and therefore in acidic soils this fraction may contribute to bioavailability [61]. The reducible and oxidizable soil fractions have the largest cumulative Pb contribution in a majority of the samples with Pb concentrations ranging from 0 to 3002 mg kg^{-1} and 0 to 2227 mg kg^{-1} respectively, indicating that Fe-Mn minerals and soil organic matter are the major repositories of Pb in the soil. This is because the minerals and phases in these low-mobility fractions have high binding capacities and therefore the potential to sequester a large concentration of cations [62]. Averaging over the percentage distribution of Pb across all samples, Pb is partitioned to an extent of 32.5% with Fe-Mn minerals and 33% with soil organic matter. This is a somewhat typical representation of Pb distribution in urban and roadside soils, and these results agree with other studies where the majority of extractable Pb is distributed amongst low-mobility oxidizable and reducible fractions [63–65]. In general, organic soil components exhibit a higher affinity for metal sorption, and results suggest that soil organic material is an important sink for Pb in community farm soils.

In terms of spatial distribution, Pb concentration in samples collected beneath building driplines (area directly located underneath the roof overhang or exterior of a building), and samples collected adjacent to parking lots were high. Samples collected adjacent to buildings were found to contain higher Pb than those of raised beds, as most buildings adjacent to these plots are older, and historic use of Pb-based exterior paints is the likely source of this Pb. For example, analysis of exterior house paint collected from buildings next to sites reporting high soil Pb in downtown Washington DC reported Pb ranging from

320,000 to 380,000 $\mu\text{g g}^{-1}$ [66]. Another likely source of soil Pb contributing to elevated concentration is automobile exhaust and this may be the case for at least one sample collected from Site 3 adjacent to a parking lot (3-PL). While the exchangeable fraction concentration of sample 3-PL is <1%, the majority of Pb is partitioned into less mobile and non-bioavailable fractions, with 60% and 25% of the Pb partitioned into reducible and oxidizable fractions, respectively.

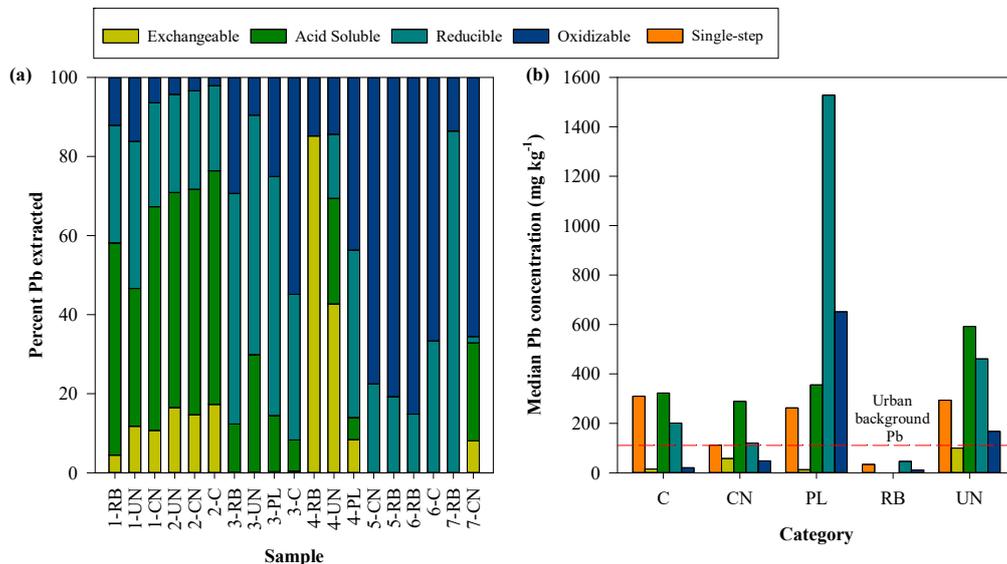


Figure 3. Tessier sequential extraction (a) normalized to the sum of the total Pb extracted from each fraction; (b) median Pb concentration for each step of the sequential extraction and the single-step extraction by sample category referenced to the urban background Pb concentration (111 mg kg^{-1}).

The median Pb concentration calculated for each sample category is represented in Figure 3b and Table S6. Raised beds and native soils constitute the majority of growing space in community farms in Newark, and samples collected from raised beds reported the lowest median concentration ranging from 0 to 46 mg kg^{-1} of Pb across all fractions (Figure 3b, Table S6). The occurrence of low amounts of Pb in raised beds is an indication that farmers follow safe cultivation practices, growing produce in elevated structures with commercially purchased clean soil thereby ensuring minimal interaction with the native soil. The median concentration of Pb is higher for sample categories which have the tendency to be influenced by anthropogenic Pb inputs like both uncultivated native soil (UN), and soils adjacent to parking lots (PL). The median distribution of Pb in these sample categories is lowest in the exchangeable fraction, with typically more accumulation in either fractions associated with carbonate minerals or Fe-Mn oxides. For the parking lot category of samples, Pb distribution increases with decreasing fractional mobility indicating enhanced Pb sequestration by Fe-Mn oxides and soil organic matter. Overall, Pb extracted from low-mobility reducible and oxidizable fractions dominate the majority of samples analyzed in this study. The prevalence of substantial Pb associated with these fractions indicates lower mobility and thus reduced bioavailability. For urban farming, this Pb is not of concern for plant uptake, however human exposure through inhalation and ingestion cannot be ignored, especially if physical handling of the soil is considered.

Based on median values, Pb concentrations determined using the Tessier scheme are generally higher than values obtained by single-step extraction. This is because of the higher extractive efficiency of the reagents used in the Tessier scheme, and increased capacity to solubilize Pb associated with stable mineral forms. Though 1 M HNO_3 extraction is a useful predictor of total soil Pb, it is more effective at extracting anthropogenically associated Pb in mobile and bioavailable fractions [50]. Measurement of metal speciation by sequential extraction has limitations [67], but remains a valuable tool in estimating potential

availability of metal in soil and sensitivity to redox and pH conditions, thereby facilitating prediction of metal cycling and availability with changing environmental conditions [68].

3.3. Metal Hotspot and Pb in Particle Size Fractions

Results from single-step and sequential extractions indicated that Site 1 had the highest overall Pb concentration amongst all community farms, with composite sample 1-UN exceeding the NJDEP RDCSRS. To determine the area(s) associated with elevated Pb, samples were taken from each location used to generate composite sample 1-UN and analyzed separately (Figure S2). The 1 M HNO₃ extractable Pb concentration of the hotspot (HS) samples ranged from 73 to 3405 mg kg⁻¹ (Figure 4a, Table S7). Site 1 is surrounded by three buildings and has four locations with possible anthropogenic inputs of Pb to the soil (Figure S2). The HS-2 sample collected adjacent to the residential building reported the highest concentration of single-step extracted Pb at 3405 mg kg⁻¹ and was identified as the Pb hotspot. High Pb concentration, especially in the mobile fractions is an indication of anthropogenic influences [45,66]. The Pb in HS-2 is likely due to the accumulation from Pb-based paints from the exterior of the adjoining building. Results of the sequential extraction showed that while all samples reported low concentrations of Pb in the exchangeable fraction, HS-2 exceeded 400 mg kg⁻¹ in acid-soluble, reducible and oxidizable fractions (Figure 4a). The majority of measured Pb in HS-2 is associated with the reducible fraction dominated by Fe-Mn oxides, and this Pb therefore has low mobility. However, handling this soil physically and without protective gear can be potentially problematic when considering exposure to Pb through ingestion, inhalation, or absorption through the skin barrier.

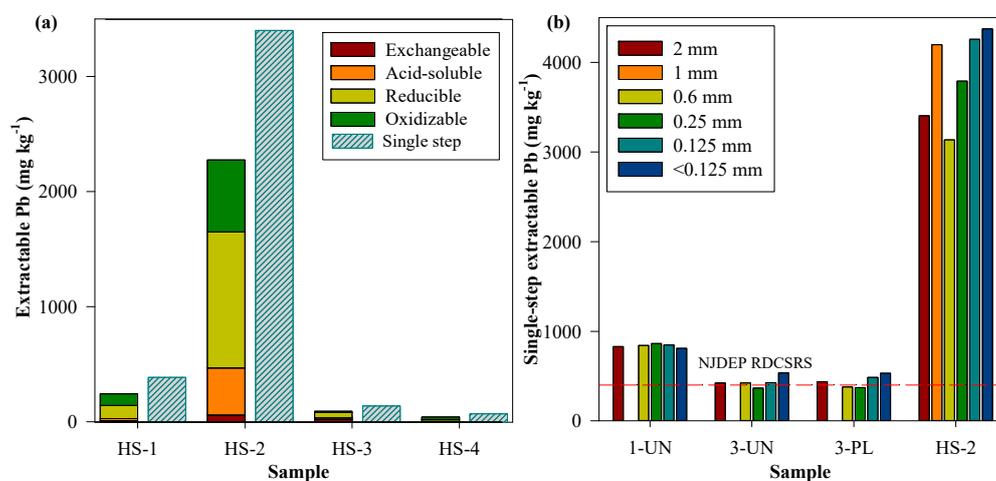


Figure 4. Pb concentration in (a) Tessier sequential and 1 M HNO₃ single-step extracted hotspot (HS) samples; (b) 1 M HNO₃ single-step extracted particle size fractions of HS-2 compared to uncultivated soil from site 1 (1-UN) and uncultivated soil and a parking lot sample from site 3 (3-UN, 3-PL) also subject to particle size analysis.

Particle size distribution is important when studying urban soils as soil components and particle aggregates may control the mobility of heavy metals, with Pb usually associated with micro-aggregates and smaller sized particles [69]. Particle size distribution of Pb was determined by a single-step extraction for samples 1-UN, 3-UN, 3-PL, and HS-2 from Sites 1 and 2 that reported extractable Pb in exceedance of the NJDEP RDCSRS (Figure 4b, Table S8). Results indicate that for sample 1-UN, Pb was almost evenly distributed across all particle size fractions, whereas for samples 3-UN, 3-PL, and HS-2, Pb concentration is inversely correlated with particle size, i.e., more Pb is associated with finer sized particles (Table S8). For HS-2 which reported the highest extractable Pb in this study, most Pb is associated with finer sized particles, with the <0.125 mm fraction containing 4375 mg kg⁻¹ of extractable Pb (Figure 4b, Table S8). The high concentration of Pb in particle size fractions (<0.125–2 mm) of HS-2 is a sign of its pervasiveness in all fractions and an indication of

historic accumulation from anthropogenic sources. A possible explanation for uniform Pb distribution in particle size fractions of 1-UN is similarities in organic matter content (that sequesters considerable soil Pb) across different particle sizes [70]. For samples HS-2, 3-UN and 3-PL the Pb concentration in the <2 mm fraction exceeds Pb in 0.6 and 0.25 mm fractions, due to possible aggregation of Pb containing particles or the presence of Pb-clusters originating from chipping, peeling, or deteriorating Pb-containing paint. Enrichment of Pb in smaller sized particles is potentially problematic due to ease of Pb remobilization and transport [71]. Transportation of finer particles can pose an airborne Pb hazard, therefore it is necessary to stabilize this fraction using immobilization techniques or other treatments [72].

3.4. X-ray Absorption Fine Structure Spectroscopy (XAFS)

To determine the speciation and coordination environment of Pb in soils with elevated concentrations, selected samples collected after each step of sequential extractions and single-step extraction of particle size fractions were analyzed by XAFS. Due to well defined Pb-O distances for different Pb sorption configurations and phases [35,73–75], and the complexity of environmental soil samples, primarily the first-shell distance was used as an indicator of the Pb sorption environment. Structural parameters and fit results (Table S9, Figure S3) were compared to structural data for Pb sorbed on a range of mineral surfaces as reported in the literature (Figure 5, Table S10, [35,73–75]).

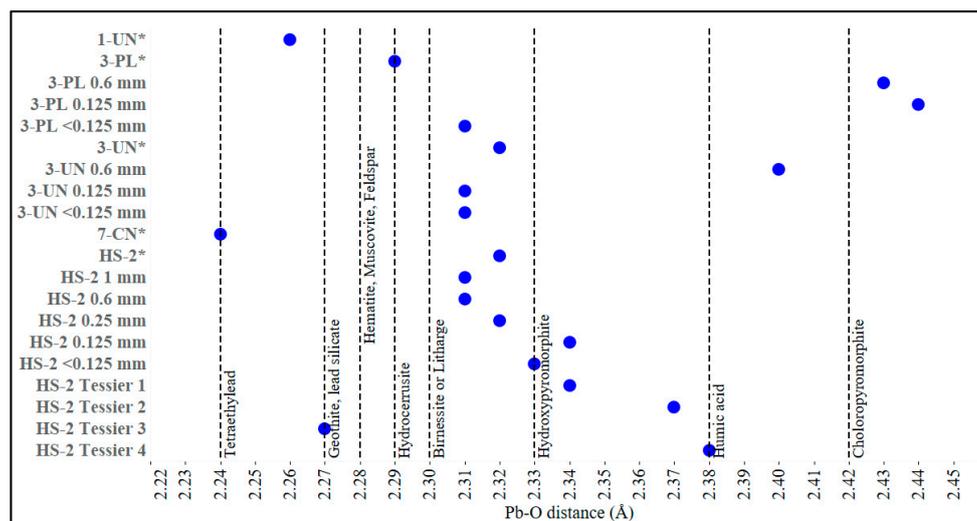


Figure 5. First-shell Pb-O distances of samples as determined by X-ray absorption fine structure spectroscopy (XAFS) analysis (y-axis, blue circle) plotted against Pb-O distances of Pb minerals and Pb-mineral sorption complexes (x-axis) as reported in the literature Table S9. Samples marked with an asterisk (*) are the <2 mm sieved particle size fraction prior to Tessier sequential extraction and sieving to 1 to <0.125 mm particle size.

Composite samples (2 mm fraction) exhibited Pb-O bond distances indicative of the dominance of Fe-Mn oxides, phosphate, and carbonate minerals. Samples HS-2 and 3-UN had Pb-O bond distances of 2.32 Å, which is in between Pb-O distances for birnessite (2.30 Å) and hydroxypyromorphite (2.33 Å), and 1-UN had a Pb-O distance of 2.26 Å consistent with goethite (2.27 Å). This is in agreement with results of the sequential extraction for these samples, which indicates most Pb was present in the reducible fraction (Figure 3a, Table S5), which in addition to Fe-Mn oxides, includes phosphate minerals [37]. For samples 1-UN and 3-UN, the Pb-O bond length is 2.32 Å, which corresponds to a model of Pb sorbed on birnessite and hydroxypyromorphite. For 3-PL, the observed bond length of 2.29 Å is consistent with hydrocerussite (2.29 Å), indicating that carbonate minerals play an important role in sequestering Pb in this sample. In sample 7-CN, the Pb-O distance is

2.24 Å, which corresponds to organometallic or tetraethyl Pb (2.24 Å), which is the form in which Pb was added to gasoline [76].

The samples recovered from sequential extraction of HS-2 were also analyzed. The speciation of residual Pb changes after each subsequent extraction. The residual Pb speciation after extractions yield Pb-O distances of 2.34 Å (HS-2 Tessier 1), 2.37 Å (HS-2 Tessier 2), 2.27 Å (HS-2 Tessier 3), and 2.38 Å (HS-2 Tessier 4); these distances indicate the dominance of hydroxypyromorphite (2.34 Å), humic acid (2.37 Å), goethite (2.27 Å) and humic acid (2.38 Å), respectively in these samples. These observations indicate the association of Pb with phosphate, iron oxide and organic matter, and is consistent with the residual phases that should be present in the sample after the relevant Tessier extraction.

The Pb speciation with particle-size fractions was assessed for HS-2, 3-UN and 3-PL samples. For HS-2 (2 mm), the Pb-O bond length is consistent with mixed sorption with birnessite and hydroxypyromorphite (2.32 Å). For particles <2mm birnessite is detected in the larger particle size fractions (0.6, and 1 mm; Pb-O 2.31 and 2.32 Å), both birnessite and hydroxypyromorphite in the intermediate fraction (0.25 mm, Pb-O 2.32 Å), followed by a transition to hydroxypyromorphite in the smaller particle size fractions (<0.125, and 0.125 mm; Pb-O 2.33 and 2.34 Å). For sample 3-UN, the average Pb-O bond distance was 2.32 Å indicating Pb is associated with birnessite and hydroxypyromorphite. The Pb-O distance increases to 2.40 Å in the 0.6 mm fraction indicating the presence of Pb sorbed on humic acid and chloropyromorphite. In the finer particle size fractions (≤ 0.125 mm), the Pb-O bond distance is shortened to 2.31 Å indicating Pb sorbed on birnessite. Similarly, 3-PL speciation changes with particle size. Higher particle size fractions (0.6, and 0.125 mm, Pb-O 2.43 and 2.44 Å) show presence of Pb sorbed on humic acid and chloropyromorphite, whereas smaller particles (<0.125 mm) are associated with birnessite (Pb-O 2.31 Å). These results indicate that in general, Pb has a tendency to be associated with Fe-Mn oxides, phosphates and organic matter in the soil, with the exact speciation contingent upon the particle size.

Beyond confirming the geochemical phases and dominant Pb species, XAFS can provide additional insights on the origin and mobility of the soil Pb. The presence of Pb tetraethyl compounds in sample 7-CN is confirmation of the anthropogenic origin of Pb from automobile emissions [76]. Similarly, Pb speciated with phosphate minerals indicates that soil Pb is insolubilized in the stable, low mobility and low bioavailable residual fraction [37,77]. However, with numerous biogeochemical reactions and soil environmental conditions, transformation of Pb to more stable forms over time [78] may have occurred. Therefore, the direct evaluation of specific sources of environmental, and especially legacy Pb, can be challenging.

4. Conclusions

This is the first systematic study conducted to understand the extent, spatial distribution, and speciation of Pb in community farm soils of Newark, NJ, a city dominated by intensive industrialization and urbanization. The median concentration of 1 M HNO₃ extractable Pb for samples from Sites 1–7 (excluding HS samples) was 127 mg kg⁻¹ (Table S4). Of these samples, 21% were in exceedance of the 400 mg kg⁻¹ NJDEP RDCSRS, representing a relatively low percentage of the soils analyzed. However, more than half of the samples, at 52%, reported concentrations higher than the urban background Pb value of 111 mg kg⁻¹ [57]. Sequential extractions using the Tessier method indicated low concentrations of Pb in the exchangeable fraction, and higher concentrations in acid, soluble, reducible and oxidizable fractions dominated by Pb in carbonates, Fe-Mn oxides and soil organic matter. The median concentration of Pb in the four fractions collected across all samples is 11 mg kg⁻¹ exchangeable, 89 mg kg⁻¹ acid-soluble, 201 mg kg⁻¹ reducible, and 44 mg kg⁻¹ oxidizable (Table S6). Results of sequential extractions correlated with first-shell XAFS analysis of select samples which revealed Pb-O distances consistent with Pb sorption with birnessite in 8 samples, pyromorphite (hydroxy/chloro) in 12 samples, humic acid in 5 samples, and goethite in 1 sample. This is an indication that the low-mobility

fractions of the soil have an increased tendency to sequester Pb, and ultimately reduce its bioavailability. Furthermore, Pb speciation was found to change with particle size, with birnessite, pyromorphite (hydroxy and chloro), and humic acid substrates dominating in the smaller particles. The XAFS analysis combined with results of sequential extraction provide evidence that multiple Pb species are present in Newark's urban soils, and multiple soil minerals serve as sinks for sequestering environmental Pb.

The majority of soil Pb in urban areas is anthropogenic in origin. Inaction in terms of remediation efforts of contaminated soil has the potential to transform soils from sinks to sources. Studies have demonstrated that Pb-contaminated soil has the potential to be air-borne as particulate Pb, thereby enhancing atmospheric Pb levels and increasing overall exposure to the contaminant [79,80]. The presence of Pb in Newark's urban community farms is a sign of its pervasiveness in urban soils, and levels of accumulation reflect that Pb emissions could have occurred from both point and non-point sources [81]. This makes it challenging to identify an exact source of the Pb in the soil and trace its origin. The Pb in Newark community farm soils is clearly anthropogenic, with potential sources including Pb-based paints, gasoline, and industrial fallout. The presence of New Jersey's largest waste incinerator in the east ward of Newark places an additional environmental burden on the community [82]. With the capacity to burn 2800 tons of waste per day [83], Pb fallout from exhaust has been speculated as an additional source of environmental Pb [82,84]. Newark has a long history of exposure to elevated Pb through drinking water in homes, schools and commercial buildings. This came to the forefront in Newark's 2019 water crisis where Pb concentrations up to four times above EPA limits of $15 \mu\text{g L}^{-1}$ were detected in drinking water [85,86]. The use of this Pb-rich tap water for watering crops may be an additional source of Pb contamination to soils, amplifying the problem of Pb exposure in the community.

Urban agriculture has numerous advantages for communities such as generating local food sources (especially in low-income communities) to reduce dependency on food imports and creating localized supply chains, shortening the farm-to-table journey thereby cutting emissions from food transportation and reducing the carbon footprint, and usage of vacant land-parcels generating alternative land use options [87]. Newark's long industrial history and environmental injustice has prepared the urban farmers to establish good farming practices like using raised beds, buying clean topsoil, and avoiding native soil for cultivation. While the urban farming community in Newark remains small, the overall Pb exposure from community farm soils also remains low. However, personal gardening practices are expanding in Newark, with a growing interest in farming in backyard soils. This can be problematic especially if growers are unaware of contaminants present in their soils and are not knowledgeable about safe farming practices to avoid urban soil contaminants. Limited by the area of their backyards, residents often farm along building driplines or in native soils, which can be concerning, especially for houses with historic use of Pb-based paints or other anthropogenic footprints. To address this gap in public awareness regarding soil health and quality, there is a need to establish safe farming workshops in conjunction with local farming organizations, expand screening of soils for contaminants, and establish citizen education and awareness programs in the greater Newark area.

Supplementary Materials: The following are available online at <https://www.mdpi.com/2571-8789/5/1/2/s1>. Figure S1: Single-step (1 M HNO₃) extractable concentration of (a) Cd, (b) Cu, and (c) Zn in samples. Figure S2: Sampling scheme implemented in Site 1 to generate samples (HS 1–4) for identifying Pb hotspot. Figure S3: Raw (solid) and corresponding fitted (dashed) (a) k^3 -weighted χ spectra and (b) radial structure functions obtained via Fourier transforming the raw k^3 -weighted spectra for samples (*marked samples denote composite 2 mm fractions). Table S1: Sample description and nomenclature used in the manuscript. Table S2: Concentration of 1M HNO₃ extracted metals for soil samples collected from Sites 1–7 and comparison to New Jersey Department of Environmental Protection (NJDEP) Residential Direct Contact Soil Remediation Standard (RDCSRS) values. Table S3: Classification based on Contamination factor (CF) and geoaccumulation index

(I_{geo}). Table S4: Calculated mean and median concentration of Pb (in mg kg^{-1}) across sites and categories for single-step extractions. The mean and median value for all samples is indicated in italics. Table S5: Average Pb concentration in soil fractions for all samples collected in the study. Table S6: Calculated median concentration of Pb (in mg kg^{-1}) across categories for sequential extractions. The median value for all samples in each fraction is indicated in italics. Table S7: Extractable Pb concentration, and Pb associated with soil fractions collected for hotspot (HS) identification. Table S8: Extractable Pb concentration in various particle size fractions of selected samples. Table S9: The structural parameters derived from EXAFS analysis of samples including error reported in analysis (shown in italics). Table S10: EXAFS parameters for Pb compounds from the literature and analyzed soil samples.

Author Contributions: Conceptualization, O.G. and A.A.R.; methodology, O.G.; formal analysis, O.G.; investigation, O.G.; resources, A.A.R.; data curation, O.G.; writing—original draft preparation, O.G.; writing, review and editing, O.G. and A.A.R.; visualization, O.G.; supervision, A.A.R.; project administration, A.A.R.; funding acquisition, A.A.R. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Science Foundation, grant number EAR 1530582 and a Rutgers University Newark Chancellor’s SEED Grant.

Data Availability Statement: The data presented in this study are available in the manuscript and supplementary materials.

Acknowledgments: The authors thank Bruce Ravel, Beamline 6-BMM, National Synchrotron Light Source-II (NSLS-II), Brookhaven National Laboratory (BNL), Upton New York, for access to the beamline, technical support and assistance with XAFS data collection.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Poulsen, M.N.; Hulland, K.R.S.; Gulas, C.A.; Pham, H.; Dalglish, S.L.; Wilkinson, R.K.; Winch, P.J. Growing an Urban Oasis: A Qualitative Study of the Perceived Benefits of Community Gardening in Baltimore, Maryland. *CAFÉ* **2014**, *36*, 69–82. [CrossRef]
2. Brown, K.H.; Jameton, A.L. Public Health Implications of Urban Agriculture. *J. Public Health Policy* **2000**, *21*, 20–39. [CrossRef] [PubMed]
3. Siegner, A.; Sowerwine, J.; Acey, C. Does Urban Agriculture Improve Food Security? Examining the Nexus of Food Access and Distribution of Urban Produced Foods in the United States: A Systematic Review. *Sustainability* **2018**, *10*, 2988. [CrossRef]
4. Lovell, S.T. Multifunctional Urban Agriculture for Sustainable Land Use Planning in the United States. *Sustainability* **2010**, *2*, 2499–2522. [CrossRef]
5. National Gardening Association. Available online: <https://garden.org/special/pdf/2014-NGA-Garden-to-Table.pdf> (accessed on 14 October 2020).
6. Attanayake, C.P.; Hettiarachchi, G.M.; Harms, A.; Presley, D.; Martin, S.; Pierzynski, G.M. Field evaluations on soil plant transfer of lead from an urban garden soil. *J. Environ. Qual.* **2014**, *43*, 475–487. [CrossRef]
7. Chaney, R.; Mielke, H.W. Standards for soil lead limitations in the United States. *Trace Subst. Environ. Health* **1986**, *20*, 357–377.
8. Mielke, H.W.; Reagan, P.L. Soil is an important pathway of human lead exposure. *Environ. Health Perspect.* **1998**, *106* (Suppl. 1), 217–229. [CrossRef]
9. Smith, D.B.; Cannon, W.F.; Woodruff, L.G.; Solano, F.; Kilburn, J.E.; Fey, D.L. Geochemical and Mineralogical Data for Soils of the Conterminous United States. 2013. Available online: <https://pubs.usgs.gov/ds/801/pdf/ds801.pdf> (accessed on 13 October 2020).
10. Minca, K.K.; Basta, N.T. Comparison of plant nutrient and environmental soil tests to predict Pb in urban soils. *Sci. Total Environ.* **2013**, *445–446*, 57–63. [CrossRef]
11. Datko-Williams, L.; Wilkie, A.; Richmond-Bryant, J. Analysis of U.S. soil lead (Pb) studies from 1970 to 2012. *Sci. Total Environ.* **2014**, *468–469*, 854–863. [CrossRef]
12. Mielke, H.W.; Anderson, J.C.; Berry, K.J.; Mielke, P.W.; Chaney, R.L.; Leech, M. Lead concentrations in inner-city soils as a factor in the child lead problem. *Am. J. Public Health* **1983**, *73*, 1366–1369. [CrossRef]
13. Pouyat, R.V.; Szlavecz, K.; Yesilonis, I.D.; Wong, C.P.; Murawski, L.; Marra, P.; Casey, R.E.; Lev, S. Multi-scale assessment of metal contamination in residential soil and soil fauna: A case study in the Baltimore–Washington metropolitan region, USA. *Landscape Urban. Plann.* **2015**, *142*, 7–17. [CrossRef]
14. Pavilonis, B.; Maroko, A.; Cheng, Z. Lead in New York City’s soils: Population growth, land use, and contamination. *Int. J. Hygiene Environ. Health* **2020**, *229*, 113564. [CrossRef] [PubMed]
15. Clarke, L.W.; Jenerette, G.D.; Bain, D.J. Urban legacies and soil management affect the concentration and speciation of trace metals in Los Angeles community garden soils. *Environ. Pollut.* **2015**, *197*, 1–12. [CrossRef] [PubMed]

16. Kaminski, M.D.; Landsberger, S. Heavy Metals in Urban Soils of East St. Louis, IL, Part I: Total Concentration of Heavy Metals in Soils. *J. Air Waste Manag. Assoc.* **2000**, *50*, 1667–1679. [[CrossRef](#)]
17. Mielke, H.W.; Laidlaw, M.A.S.; Gonzales, C. Lead (Pb) legacy from vehicle traffic in eight California urbanized areas: Continuing influence of lead dust on children's health. *Sci. Total Environ.* **2010**, *408*, 3965–3975. [[CrossRef](#)]
18. Attanayake, C.P.; Hettiarachchi, G.M.; Ma, Q.; Pierzynski, G.M.; Ransom, M.D. Lead Speciation and In Vitro Bioaccessibility of Compost-Amended Urban Garden Soils. *J. Environ. Qual.* **2017**, *46*, 1215–1224. [[CrossRef](#)]
19. Filippelli, G.M.; Adamic, J.; Nichols, D.; Shukle, J.; Frix, E. Mapping the Urban Lead Exposome: A Detailed Analysis of Soil Metal Concentrations at the Household Scale Using Citizen Science. *Int. J. Environ. Res.* **2018**, *15*, 1531. [[CrossRef](#)]
20. Finster, M.E.; Gray, K.A.; Binns, H.J. Lead levels of edibles grown in contaminated residential soils: A field survey. *Sci. Total Environ.* **2004**, *320*, 245–257. [[CrossRef](#)]
21. Laidlaw, M.A.S.; Filippelli, G.M.; Brown, S.; Paz-Ferreiro, J.; Reichman, S.M.; Netherway, P.; Truskewycz, A.; Ball, A.S.; Mielke, H.W. Case studies and evidence-based approaches to addressing urban soil lead contamination. *Appl. Geochem.* **2017**, *83*, 14–30. [[CrossRef](#)]
22. Mielke, H.W.; Blake, B.; Burroughs, S.; Hassinger, N. Urban lead levels in Minneapolis: The case of the Hmong children. *Environ. Res.* **1984**, *34*, 64–76. [[CrossRef](#)]
23. Mitchell, R.G.; Spliethoff, H.M.; Ribaud, L.N.; Lopp, D.M.; Shayler, H.A.; Marquez-Bravo, L.G.; Lambert, V.T.; Ferenz, G.S.; Russell-Anelli, J.M.; Stone, E.B.; et al. Lead (Pb) and other metals in New York City community garden soils: Factors influencing contaminant distributions. *Environ. Pollut.* **2014**, *187*, 162–169. [[CrossRef](#)] [[PubMed](#)]
24. Moller, K.M.; Hartwell, J.G.; Simon-Friedt, B.R.; Wilson, M.J.; Wickliffe, J.K. Soil Contaminant Concentrations at Urban Agricultural Sites in New Orleans, Louisiana. *J. Agric. Food Syst. Community Dev.* **2018**, *8*, 139–149. [[CrossRef](#)]
25. Defoe, P.P.; Hettiarachchi, G.M.; Benedict, C.; Martin, S. Safety of Gardening on Lead- and Arsenic-Contaminated Urban Brownfields. *J. Environ. Qual.* **2014**, *43*, 2064–2078. [[CrossRef](#)] [[PubMed](#)]
26. Shinn, N.J.; Bing-Canar, J.; Cailas, M.; Peneff, N.; Binns, H.J. Determination of Spatial Continuity of Soil Lead Levels in an Urban Residential Neighborhood. *Environ. Res.* **2000**, *82*, 46–52. [[CrossRef](#)]
27. Sterrett, S.B.; Chaney, R.L.; Gifford, C.H.; Mielke, H.W. Influence of fertilizer and sewage sludge compost on yield and heavy metal accumulation by lettuce grown in urban soils. *Environ. Geochem. Health* **1996**, *18*, 135–142. [[CrossRef](#)]
28. Wagner, T.; Langley-Turnbaugh, S. Case study: Examining the contribution of historical sources of lead in urban soils in Portland, Maine, USA. *J. Environ. Plan. Manag.* **2008**, *51*, 525–541. [[CrossRef](#)]
29. Brown, S.L.; Chaney, R.L.; Hettiarachchi, G.M. Lead in Urban Soils: A Real or Perceived Concern for Urban Agriculture? *J. Environ. Qual.* **2016**, *45*, 26–36. [[CrossRef](#)]
30. United States Environmental Protection Agency. Protect Your Family from Exposures to Lead. Available online: <https://www.epa.gov/lead/protect-your-family-exposures-lead> (accessed on 22 May 2020).
31. Agency for Toxic Substances and Disease Registry. Toxicological Profile: Lead. Available online: <https://www.atsdr.cdc.gov/toxprofiles/tp.asp?id=96&tid=22> (accessed on 14 May 2020).
32. Nevidomskaya, D.; Minkina, T.; Soldatov, A.; Shuvaeva, V.; Zubavichus, Y.; Podkovyrina, Y. Comprehensive study of Pb (II) speciation in soil by X-ray absorption spectroscopy (XANES and EXAFS) and sequential fractionation. *J. Soils Sediments* **2016**, *16*, 1183–1192. [[CrossRef](#)]
33. Wong, C.S.C.; Li, X.D. Pb contamination and isotopic composition of urban soils in Hong Kong. *Sci. Total Environ.* **2004**, *319*, 185–195. [[CrossRef](#)]
34. Imperato, M.; Adamo, P.; Naimo, D.; Arienzo, M.; Stanzione, D.; Violante, P. Spatial distribution of heavy metals in urban soils of Naples city (Italy). *Environ. Pollut.* **2003**, *124*, 247–256. [[CrossRef](#)]
35. Ryan, J.A.; Zhang, P.; Hesterberg, D.; Chou, J.; Sayers, D.E. Formation of Chloropyromorphite in a Lead-Contaminated Soil Amended with Hydroxyapatite. *Environ. Sci. Technol.* **2001**, *35*, 3798–3803. [[CrossRef](#)] [[PubMed](#)]
36. Hashimoto, Y.; Takaoka, M.; Oshita, K.; Tanida, H. Incomplete transformations of Pb to pyromorphite by phosphate-induced immobilization investigated by X-ray absorption fine structure (XAFS) spectroscopy. *Chemosphere* **2009**, *76*, 616–622. [[CrossRef](#)] [[PubMed](#)]
37. Hettiarachchi, G.M.; Pierzynski, G.M. Soil lead bioavailability and in situ remediation of lead-contaminated soils: A review. *Environ. Prog.* **2004**, *23*, 78–93. [[CrossRef](#)]
38. Funasaka, K.; Tojo, T.; Katahira, K.; Shinya, M.; Miyazaki, T.; Kamiura, T.; Yamamoto, O.; Moriwaki, H.; Tanida, H.; Takaoka, M. Detection of Pb-LIII edge XANES spectra of urban atmospheric particles combined with simple acid extraction. *Sci. Total Environ.* **2008**, *403*, 230–234. [[CrossRef](#)] [[PubMed](#)]
39. Funasaka, K.; Tojo, T.; Kaneco, S.; Takaoka, M. Different chemical properties of lead in atmospheric particles from urban roadside and residential areas. *Atmos. Pollut. Res.* **2013**, *4*, 362–369. [[CrossRef](#)]
40. Yi, K. Green Gardens Sprouting from Vacant Lots. *Urban Farmers Hope to Grow City's Economy*. Available online: <https://www.nj.com/essex/2019/12/green-gardens-sprouting-from-vacant-lots-urban-farmers-hope-to-grow-citys-economy.html> (accessed on 22 May 2020).
41. City of Newark. Available online: <https://www.newarknj.gov/zoning> (accessed on 29 October 2020).
42. Weil, R.; Brady, N. *The Nature and Properties of Soils*, 15th ed.; Pearson Education: London, UK, 2017; ISBN 9780133254488.

43. United States Environmental Protection Agency. *Observational Economy Series Volume 1: Composite Sampling*; EPA: Washington, DC, USA, 1995. Available online: <https://www.epa.gov/sites/production/files/2016-03/documents/comp-samp.pdf> (accessed on 24 December 2020).
44. Carter, M.R.; Gregorich, E.G. *Soil Sampling and Methods of Analysis*, 2nd ed.; CRC Press: Boca Raton, FL, USA, 2007. [CrossRef]
45. Chaney, R.L.; Sterrett, S.B.; Mielke, H.W. The potential for heavy metal exposure from urban gardens and soils. In *Proceedings of the Symposium on Heavy Metals in Urban Gardens*; University of District of Columbia Extension Service: Washington, DC, USA, 1984; pp. 37–84. Available online: https://www.researchgate.net/publication/242693949_THE_POTENTIAL_FOR_HEAVY_METAL_EXPOSURE_FROM_URBAN_GARDENS_AND_SOILS (accessed on 28 December 2020).
46. N.J.A.C. 7:26D Remediation Standards. Available online: https://www.nj.gov/dep/rules/rules/njac7_26d.pdf (accessed on 17 October 2020).
47. Tessier, A.; Campbell, P.G.C.; Bisson, M. Sequential extraction procedure for the speciation of particulate trace metals. *Anal. Chem.* **1979**, *51*, 844–851. [CrossRef]
48. John, D.A.; Leventhal, J.S. Bioavailability of Metals. Available online: <https://pubs.usgs.gov/of/1995/ofr-95-0831/CHAP2.pdf> (accessed on 17 October 2020).
49. Ravel, B.; Newville, M. ATHENA, ARTEMIS, HEPHAESTUS: Data analysis for X-ray absorption spectroscopy using IFEFFIT. *J. Synchrotron Radiat.* **2005**, *12*. [CrossRef]
50. McBride, M.B.; Mathur, R.R.; Baker, L.L. Chemical Extractability of Pb in Field-Contaminated Soils: Implications for Estimating Total Pb. *Commun. Soil Sci. Plant. Anal.* **2011**, *42*, 1581–1593. [CrossRef]
51. Wharton, S.E.; Shayler, H.A.; Spliethoff, H.M.; Marquez-Bravo, L.G.; Ribaud, L.; McBride, M.B. A Comparison of Screening Tests for Soil Pb. *Soil Sci.* **2012**, *177*, 650–654. [CrossRef]
52. Kessler, R. Urban Gardening: Managing the Risks of Contaminated Soil. *Environ. Health Perspect.* **2013**, *121*, A326–A333. [CrossRef]
53. Van Roosmalen, G.R.E.M.; Lustenhouwer, J.W.A.; Oosthoek, J.; Senden, M.M.G. Heavy metal sources and contamination mechanisms in compost production. *Resour. Conserv.* **1987**, *14*, 321–334. [CrossRef]
54. Hakanson, L. An ecological risk index for aquatic pollution control. a sedimentological approach. *Water Res.* **1980**, *14*, 975–1001. [CrossRef]
55. Remeikaitė-Nikienė, N.; Garnaga-Budrė, G.; Lujanienė, G.; Jokšas, K.; Stankevičius, A.; Malejevas, V.; Barisevičiūtė, R. Distribution of metals and extent of contamination in sediments from the south-eastern Baltic Sea (Lithuanian zone). *Oceanologia* **2018**, *60*, 193–206. [CrossRef]
56. Muller, G. Index of Geoaccumulation in Sediments of the Rhine River. *GeoJournal* **1969**, *2*, 108–118.
57. Sanders, P.F. *Ambient Levels of Metals in New Jersey Soils*; NJDEP: Trenton, NJ, USA, 2003; Available online: <https://www.state.nj.us/dep/dsr/research/ambient-levels-metal.pdf> (accessed on 24 December 2020).
58. Clark, H.F.; Brabander, D.J.; Erdil, R.M. Sources, Sinks, and Exposure Pathways of Lead in Urban Garden Soil. *J. Environ. Qual.* **2006**, *35*, 2066–2074. [CrossRef] [PubMed]
59. Wuana, R.A.; Okieimen, F.E. Heavy Metals in Contaminated Soils: A Review of Sources, Chemistry, Risks and Best Available Strategies for Remediation. *ISRN Ecol.* **2011**, 402647. [CrossRef]
60. Rieuwerts, J.S.; Thornton, I.; Farago, M.E.; Ashmore, M.R. Factors influencing metal bioavailability in soils: Preliminary investigations for the development of a critical loads approach for metals. *Chem. Speciat. Bioavailab.* **1998**, *10*, 61–75. [CrossRef]
61. Elder, J.F. Metal biogeochemistry in surface-water systems; a review of principles and concepts. *Circular* **1988**. [CrossRef]
62. Zimdahl, R.L.; Skogerboe, R.K. Behavior of lead in soil. *Environ. Sci. Technol.* **1977**, *11*, 1202–1207. [CrossRef]
63. Harrison, R.M.; Laxen, D.P.H.; Wilson, S.J. Chemical associations of lead, cadmium, copper, and zinc in street dusts and roadside soils. *Environ. Sci. Technol.* **1981**, *15*, 1378–1383. [CrossRef]
64. Sutherland, R.A.; Tack, F.M.G. Metal phase associations in soils from an urban watershed, Honolulu, Hawaii. *Sci. Total Environ.* **2000**, *256*, 103–113. [CrossRef]
65. Norrström, A.C.; Jacks, G. Concentration and fractionation of heavy metals in roadside soils receiving de-icing salts. *Sci. Total Environ.* **1998**, *218*, 161–174. [CrossRef]
66. Preer, J.R.; Akintoye, J.O.; Martin, M.L. Metals in downtown Washington, DC gardens. *Biol. Trace Elem. Res.* **1984**, *6*, 79–91. [CrossRef] [PubMed]
67. Nirel, P.M.V.; Morel, F.M.M. Pitfalls of sequential extractions. *Water Res.* **1990**, *24*, 1055–1056. [CrossRef]
68. Dollar, N.L.; Souch, C.J.; Filippelli, G.M.; Mastalerz, M. Chemical Fractionation of Metals in Wetland Sediments: Indiana Dunes National Lakeshore. *Environ. Sci. Technol.* **2001**, *35*, 3608–3615. [CrossRef]
69. Mi, Y.; Zhan, F.; Li, B.; Qin, L.; Wang, J.; Zu, Y.; Li, Y. Distribution characteristics of cadmium and lead in particle size fractions of farmland soils in a lead–zinc mine area in Southwest China. *Environ. Syst. Res.* **2018**, *7*, 14. [CrossRef]
70. Park, H.; Park, H.; Yang, H.I.; Park, S.; Lim, S.; Kwak, J.; Lee, G.; Lee, S.; Park, M.; Choi, W. Sorption of Pb in chemical and particle-size fractions of soils with different physico-chemical properties. *J. Soils Sediments* **2019**, *19*, 310–321. [CrossRef]
71. Sharp, R.M.; Brabander, D.J. Lead (Pb) Bioaccessibility and Mobility Assessment of Urban Soils and Composts: Fingerprinting Sources and Refining Risks to Support Urban Agriculture. *GeoHealth* **2017**, *1*, 333–345. [CrossRef]
72. Dermatas, D.; Chrysochoou, M. Lead particle size and its association with firing conditions and range maintenance: Implications for treatment. *Environ. Geochem. Health* **2007**, *29*, 347–355. [CrossRef]

73. Manceau, A.; Boisset, M.; Sarret, G.; Hazemann, J.; Mench, M.; Cambier, P.; Prost, R. Direct Determination of Lead Speciation in Contaminated Soils by EXAFS Spectroscopy. *Environ. Sci. Technol.* **1996**, *30*, 1540–1552. [[CrossRef](#)]
74. Ostergren, J.D.; Brown, G.E.; Parks, G.A.; Tingle, T.N. Quantitative Speciation of Lead in Selected Mine Tailings from Leadville, CO. *Environ. Sci. Technol.* **1999**, *33*, 1627–1636. [[CrossRef](#)]
75. Rouff, A.A.; Elzinga, E.J.; Reeder, R.J.; Fisher, N.S. X-ray Absorption Spectroscopic Evidence for the Formation of Pb (II) Inner-Sphere Adsorption Complexes and Precipitates at the Calcite–Water Interface. *Environ. Sci. Technol.* **2004**, *38*, 1700–1707. [[CrossRef](#)] [[PubMed](#)]
76. Hamel, S.; Heckman, J.; Murphy, S. Lead Contaminated Soil: Minimizing Health Risks. Available online: <https://njaes.rutgers.edu/fs336/> (accessed on 17 October 2020).
77. Chaney, R.L.; Mielke, H.W.; Sterrett, S.B. Speciation, mobility, and bioavailability of Soil Lead. In *Lead in Soils: Issues and Guidelines*; Davies, B.E., Wixson, B.G., Eds.; CRC Press: Boca Raton, FL, USA, 1989; pp. 105–129.
78. Miretzky, P.; Fernandez-Cirelli, A. Phosphates for Pb immobilization in soils: A review. *Environ. Chem. Lett.* **2008**, *6*, 121–133. [[CrossRef](#)]
79. Zahran, S.; Laidlaw, M.A.S.; McElmurry, S.P.; Filippelli, G.M.; Taylor, M. Linking Source and Effect: Resuspended Soil Lead, Air Lead, and Children’s Blood Lead Levels in Detroit, Michigan. *Environ. Sci. Technol.* **2013**, *47*, 2839–2845. [[CrossRef](#)]
80. Pingitore, J.; Nicholas, E.; Clague, J.W.; Amaya, M.A.; Maciejewska, B.; Reynoso, J.J. Urban Airborne Lead: X-Ray Absorption Spectroscopy Establishes Soil as Dominant Source. *PLoS ONE* **2009**, *4*, e5019. [[CrossRef](#)]
81. Burt, R.; Hernandez, L.; Shaw, R.; Tunstead, R.; Ferguson, R.; Peaslee, S. Trace element concentration and speciation in selected urban soils in New York City. *Environ. Monit. Assess.* **2014**, *186*, 195–215. [[CrossRef](#)]
82. Essex County Trash Incinerator Unfairly Burdens Poor Critics Say. Available online: <https://patch.com/new-jersey/newarknj/essex-county-trash-incinerator-unfairly-burdens-poor-critics-say> (accessed on 7 October 2020).
83. Essex Covanta. Available online: <https://www.covanta.com/where-we-are/our-facilities/essex> (accessed on 7 October 2020).
84. Earthjustice: Newark Youth March on Covanta, Trash-Burning Incinerator Masquerading As ‘Energy Plant’. Available online: <https://earthjustice.org/news/press/2018/newark-youth-march-on-covanta-trash-burning-incinerator-masquerading-as-energy-plant> (accessed on 14 October 2020).
85. Olson, D.E. Newark’s Lead Crisis Continues: Even Higher Levels in 2019. Available online: <https://www.nrdc.org/experts/erik-d-olson/newarks-lead-crisis-continues-even-higher-levels-2019> (accessed on 21 October 2020).
86. Yi, K. Newark Lead Levels Are Lower But Still Elevated, New Water Tests Show. Available online: <https://www.nj.com/essex/2020/01/newark-lead-levels-are-lower-but-still-elevated-new-water-tests-show.html> (accessed on 21 October 2020).
87. Urban Agriculture: Practices to Improve Cities. Available online: <https://urbanland.uli.org/news/urban-agriculture-practices-to-improve-cities/> (accessed on 22 May 2020).