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Comparison of plant nutrient and environmental soil tests to predict Pb in urban soils

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HIGHLIGHTS

► The ability of soil tests to predict total Pb and other contaminants in 65 urban soils was determined.
► Most soil testing labs use Mehlich 3 which could be implemented as a screening tool for soil Pb, Cu, and Zn.
► Total Pb can be conservatively estimated by the following equation Total Pb (mg kg^{-1}) = Mehlich 3 Pb (mg kg^{-1}) × 2.

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ABSTRACT

Most urban soils are not tested for Pb because of the high costs associated with sampling and laboratory analysis of soil contaminants. However, soil testing for plant nutrients is inexpensive and routinely performed for agricultural soils used for food production. The objectives of this study are to determine the ability of 1 M HNO_3, Mehlich 3, and Modified Morgan soil tests to predict total Pb and other contaminants in urban soils. Total Pb was determined from 65 urban vacant residential lots being considered for urban gardens and food production in Cleveland, OH. Extractable Pb was determined using common soil nutrient test methods Mehlich 3 and Modified Morgan extraction, and a 1 M HNO_3 extraction. Significant linear regressions between total Pb and Mehlich 3 ($r^2 = 0.83$), 1 M HNO_3 ($r^2 = 0.92$), and Modified Morgan ($r^2 = 0.77$) in study soils were found. Most commercial and university soil testing labs use Mehlich 3 which could be implemented as a screening tool for soil Pb, Cu, and Zn. The Mehlich 3 soil test is widely used and is relatively inexpensive (<$15). Our results show that total Pb can be conservatively estimated by the following equation Total Pb (mg kg^{-1}) = Mehlich 3 Pb (mg kg^{-1}) × 2.

1. Introduction

Lead (Pb) contamination is an increasingly important international public health issue with the resurging interest in urban gardening and the recent federal recommendations from the Center for Disease Control lowering the reference value for elevated blood Pb level to 5 μg dL^{-1} (CDC, 2012). Exposure to Pb can affect nearly every system in the human body, but the symptoms of lead poisoning often go undetected. Pb is a potent neurotoxin that can delay or prohibit development, which in turn can cause learning disabilities, behavioral problems and at very high levels seizures, coma or death (Agency for Toxicology of Substances and Disease Registry (ATSDR, 2001)). Ingestion of house dust or soil Pb is a particular threat to children because of hand to mouth behavior. Nationally, the Center for Disease Control reported that the number of urban children with blood lead levels above 10 μg dL^{-1} was 16%, but with the recent lowering of the reference value of 5 μg dL^{-1} the percentage of at risk children is likely to increase (ATSDR, 2007). In 2008, the Cuyahoga County Board of Health determined that over one fifth of children in the county had elevated blood levels > 5 μg dL^{-1} (Ohio Department of Health 2010).

In many old industrial cities, including Cleveland Ohio, urban redevelopment has created vacant land. City planners, community groups and other stakeholders seek to convert some of the vacant land for urban agriculture and gardening, parks, playgrounds and other common areas. It is important to assess these areas prior to redevelopment because this type of land use involves increased exposure to potentially contaminated soil.

All soils contain background levels of Pb at relatively low concentrations due to naturally occurring soil lead minerals such as anglesite, cerussite, and galena. Background soil Pb has been reported as 19 mg Pb kg^{-1} soil (Shacklette and Boerngen, 1984) and 16.5 mg kg^{-1} Pb (Gustavsson et al., 2001) with mean uncontaminated background concentration for the state of Ohio of 19 mg Pb kg^{-1} soil, respectively (Logan and Miller, 1983). The main source of Pb in urban soils comes from the historic use of Pb-based products including Pb-based paint and Pb-based gasoline. Dried Pb paint often contained 30 to 50% Pb by weight. Paint analysis from older urban homes shows high concentrations of multiple metals in exterior paint samples; Pb 35,000 μg g^{-1}, Zn 31,000 μg g^{-1}, Cd 439 μg g^{-1}, Cu 2000 μg g^{-1}, and Cr 775 μg g^{-1} (Turner and Sogo, 2012; Mielke et al., 2001). The manufacturing and use of these Pb-based products in the USA were banned by the end of

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the 1970s (Filippelli et al., 2004). The common forms of Pb in urban soils containing Pb based paint residues are Pb carbonates and Pb oxides. The relatively low solubility and mobility of this type of anthropogenic Pb in soils cause it to persist in the soil.

The quantity and spatial distribution of Pb in urban areas have been extensively studied over the last few decades due its persistence in urban soils (Schwarz et al., 2012; Petersen et al., 2006; Filippelli et al., 2004; Mielke et al., 1984, 2004; Jennings et al., 2002; Mielke, 1994; Ryan et al., 2004; Chaney et al., 1984; Davies and Roberts, 1987). These studies show that soil Pb concentrations are highest in urban epicenters (Schwarz et al., 2012; Mielke et al., 2004; Jennings et al., 2002). These studies also demonstrate the decline in soil Pb concentrations with increased distance from home foundations and roadways, which further supports the idea that the source of Pb contamination in urban areas is due to Pb-based paints and gasoline (Mielke et al., 1984; Chaney et al., 1984).

Urban children are the most vulnerable to Pb exposure through the ingestion of household dust as well as the ingestion of soil. To protect human health the USEPA has set the maximum Pb concentration in residential bare soil used for children's play at 400 mg Pb kg^{-1} soil. Soils which exceed the maximum concentration are recommended for further investigation to determine necessary restrictions on land use. The soil test used to assess the potential human health risk of Pb in urban soil provides an estimate of the total Pb content of soil, and will be referred to as total Pb. The most commonly used methods involve partial dissolution of soil using strong acids and/or pressure assisted microwave digestion such as USEPA Method 3051a. Total Pb only provides information specific to Pb. It does not provide additional information about soil quality and nutrition and it is expensive ($100 to $300 per soil sample). In addition, many soil testing laboratories do not perform total metal analysis for soil. Most soil testing laboratories do provide analysis of plant available nutrients in soil and soil management recommendations. Soil nutrient tests such as Mehlich 3 and modified Morgan are currently being used by several University soil testing labs, including Rutgers, Connecticut, Maine, Vermont, and Massachusetts to estimate Pb in soil. In addition, a 1 M HNO_3 extraction is also used at the University of Minnesota and the University of Delaware.

Positive correlation between soil test Pb and total Pb has been reported using the following soil tests: 1 M HNO_3 (McBride et al., 2011; Mielke et al., 1983, 1999; Chaney et al., 1984; Veneman et al., 1982; Day et al., 1979; John, 1971); 1 M HCl (Petersen et al., 2006; Jennings et al., 2002; Pfaff, 1996; Veneman et al., 1982); Mehlich 3 (Witzling et al., 2011; Elrashidi et al., 2003; Hamel et al., 2003; Sims et al., 1991); Mehlich 1 (Hamel et al., 2003; Sims et al., 1991); Morgan (Nicklow et al., 1981); Modified Morgan (McBride et al., 2011; Hamel et al., 2003; Nicklow et al., 1981); DPTA (Boon and Soltanpour, 1992; Sims et al., 1991; Soltanpour, 1985), and EDTA (Elrashidi et al., 2003; Kocialkowski et al., 1999; Berrow and Mitchell, 1991; Clayton and Tiller, 1979; Davies and Ginnever, 1979; Viro, 1955). Even though the 1 M HCl, Mehlich 1, DPTA and EDTA soil tests strongly correlated with total Pb, these tests were not widely adopted by soil testing laboratories. The soil tests most widely used to predict total Pb are 1 M HNO_3, Mehlich 3 and Modified Morgan, but there is not a consensus on which soil test is the best predictor of soil Pb. Several studies compared one or two of the above three soil tests and total Pb, but to my knowledge no studies have evaluated and compared all three of the most recently common soil tests (1 M HNO_3, Mehlich 3 and Modified Morgan) to predict total Pb in residential urban soils. The vast majority of studies have included residential soils contaminated from Pb paint and gasoline emission and soils with other Pb contaminant sources associated with industrial and orchard pesticides. These studies report that soil testing methods may extract different percentages of total soil Pb from residential vs. non-residential sources. All of the urban vacant lot soils in our study are from residential properties. The ability of the three commonly used soil extraction tests used to predict total Pb in *only* urban residential soils is investigated.

Therefore the objectives of this study are (1) to compare the extraction potential of 1 M HNO_3, Mehlich 3, and Modified Morgan in both the <2 mm and the <250 μm soil fractions, (2) to determine the ability of 1 M HNO_3, Mehlich 3, and Modified Morgan soil tests to predict total Pb in urban soils, and (3) to evaluate the ability of 1 M HNO_3, Mehlich 3, and Modified Morgan soil tests to estimate additional metals in soil.

2. Materials and methods

2.1. Soil selection and characterization

The 65 soils used in this study were collected from individual vacant urban lots located in Cleveland, OH during May and June 2010. These lots are currently being considered for urban gardens and food production. Surface samples (0–10 cm) were collected in an X shaped grid in each lot. For each site location, the surface samples were composited into one sample for a total of 65 samples. Each composite was air dried and processed to obtain a uniform material. The air dried sample was thoroughly mixed after removing roots, rocks, and other debris. Two size fractions, <2 mm and <250 μm, were prepared from the homogenized soil.

2.1.1. Determination of soil properties

All materials used to characterize the soils were reagent grade or higher purity, and were prepared using de-ionized (DI) water generated by a Barnstead NANOpure UV system (18 MΩ-cm). Soil texture was measured using the pipette method (Gee and Bauder, 1982). Soil electrical conductivity (EC) and pH were measured using 1:1 de-ionized water: soil (Rhoades, 1996; Thomas, 1996). Soil (20 g, <2 mm) was mixed with 20 mL of DI water and shaken on a reciprocating shaker for 10 min, then allowed to settle for another 10 min. The pH of the soil was measured using a combination pH electrode, and the EC was measured using an Oakton Acorn CON 6 conductivity probe. Total soil carbon and nitrogen (50 mg, <250 μm) were measured using the dry combustion method and thermal conductivity detection with a Perkin-Elmer CHN2400 High Temperature Induction Furnace (Nelson and Sommers, 1996).

2.2. Chemical analysis of soils

2.2.1. Total metal content

Total metal concentrations were determined by U.S. EPA method 3051a, a microwave assisted extraction using an aqua regia type solution of HCl and HNO_3. Concentrated HCl (3 mL) and HNO_3 (9 mL) were added to soil (0.5 g, <2 mm & <250 μm) in a sealed Teflon tube. Samples were digested using a MARS 1600 watt microwave (CEM Corporation, Mathews, NC). Once cooled, samples were then transferred into 50 mL volumetric flasks, brought to volume and thoroughly mixed. Samples were syringe filtered using 0.45 μm nylon syringe filters and analyzed using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) (U.S. EPA Method 6010C).

2.2.2. Soil nutrient tests

Two agricultural soil tests commonly used to determine soil fertility, plant nutrition and fertilizer recommendations were used. These include Mehlich 3 (Amacher, 1996) and Modified Morgan's Extraction (Helmke and Sparks, 1996). In the Mehlich 3 extraction, soil (1 g, <2 mm & <250 μm) was mixed with 10 mL of Mehlich 3 extracting solution in 2 oz. Solo cup and shaken on a Junior Orbital Shaker (Lab-Line Instruments, Inc) at 180 cycle min^{-1} for 5 min. Samples were syringe filtered using 0.45 μm nylon syringe filters and analyzed using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) EPA Method 6010C. In the Modified Morgan's extraction soil (4 g, <2 mm & <250 μm) was mixed with 20 mL of Morgan extractant in a 50 mL Nalgene centrifuge tube. The samples were shaken on a reciprocating