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TECHNICAL REPORT

Urban Pollutants

Nutrient and toxic elements in soils and plants across 10 urban community gardens: Comparing pXRF and ICP-based soil measurements

Ainsley C. McStay[1] | **Sandra L. Walser**[2] | **Eric C. Sirkovich**[1] | **Nicolas Perdrial**[2] | **Justin B. Richardson**[1]

[1]Dep. of Geosciences, Univ. of Massachusetts Amherst, Amherst, MA 01003, USA

[2]Dep. of Geology, Univ. of Vermont, Burlington, VT 05405, USA

Correspondence
Justin B. Richardson, Dep. of Geosciences, Univ. of Massachusetts Amherst, 611 North Pleasant St., Amherst, MA 01003, USA.
Email: Jbrichardson@umass.edu

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Abstract

Urban community gardens are becoming increasingly important to rehabilitate developed lands and combat the lack of access to fresh produce. Portable X-ray fluorescence (pXRF) offers a rapid, cost-effective method for assessing the elemental composition of soils but needs further study to determine its efficacy in urban agriculture. The objectives of this study were to evaluate if pXRF measurements of macronutrients (Ca, K, P), micronutrients (Cu, Mn, Zn), and toxic elements (As, Pb) generate results comparable with traditional soil analyses and if the soil measurements correlate with plant tissue concentrations at 10 community gardens across the eastern United States. From field-condition analyses of soils by pXRF and pseudo-total digestions, we observed that both methods provide agreeable estimates of concentrations for some elements (Mn, Cu, Zn, Pb) but not for macronutrients (Ca, K, P). We hypothesize that low accuracy in pXRF measurements and macronutrients within silicates caused the poor agreement between the methods. Sieved and dried soil pXRF concentrations were in strong agreement with field-condition pXRF concentrations, suggesting rock removal and drying did not improve measurements. Our results highlight that pXRF can be an accurate and effective tool for screening for Mn, Cu, Zn, and Pb. Some elements, such as Pb in fruits; Mn, Cu, and Zn in leaves; and Zn and Pb in roots, could be estimated by soil pXRF or inductively coupled plasma–based analyses. Macronutrients were poorly estimated for fruits, leaves, and roots. Instead of soil concentrations, identifying genus-specific and garden-specific factors may be important for generating plant uptake predictive models.

Abbreviations: ICP, inductively coupled plasma; ICP-MS, inductively coupled plasma–mass spectrometry; ICO-OES, inductively coupled plasma–optical emission spectrometry; LOI, loss-on-ignition; PCA, principal component analysis; pXRF, portable X-ray fluorescence; SOM, soil organic matter.

1 | INTRODUCTION

Urban farming is becoming increasingly important as a local food source in cities to combat the lack of access to fresh produce, to reconnect citizens with food production, and to repurpose developed land to green space (Azunre et al., 2019; Opitz et al., 2016; Pearson et al., 2010). Community gardens are a meso-urban agricultural production system in which gardeners are allotted plots of land and have exclusive rights to the plot or share in management of the entire garden cultivated in a commons model (Pearson et al., 2010). Community gardens enhance social, economic, and health aspects of gardeners and surrounding communities. When individuals participate directly, community gardens can increase fruit and vegetable consumption by a factor of 1.4–3.5 (Alaimo et al., 2008) and provide social and psychological benefits from participation (Litt et al., 2011). In developing countries, income and food from community gardens increase food access and security and can be used to support local schools and health care access (Azunre et al., 2019; Prain & Lee-Smith, 2010). Indirectly, community gardens improve neighborhood aesthetics, improve social cohesion, increase home values, and convert brownfields to a productive land-use (Voicu & Been, 2008; Litt et al., 2011; Maantay & Maroko, 2018).

Urban community gardens share many of the issues of traditional agriculture, such as nutrient limitations of macronutrients (Ca, P, K), soil pH balancing, and maintaining soil organic matter (SOM) and soil moisture. Community gardens can also be negatively affected by soil contamination due to an overabundance of micronutrients (Cu, Mn, Zn) and potentially toxic elements (e.g., As, Pb) from automobiles and industrial emissions, historical pesticide and biocide use, and waste dumping and incineration (Defoe et al., 2014; Richardson, 2020). Therefore, ensuring the soil has low concentrations of potentially toxic metals and adequate inorganic nutrients is essential to maximize yield and protect consumer health. However, soil tests that examine macronutrients, micronutrients, and potentially toxic elements can be prohibitively expensive for community gardens. Portable X-ray fluorescence spectroscopy (pXRF) offers a low-cost, rapid method to assess these elements in soils and can be as accurate and precise as traditional inductively coupled plasma (ICP) methods (Gutiérrez-Ginés et al., 2013; Markey et al., 2008; Pozza et al., 2020; Weindorf & Chakraborty 2020). However, there are several limitations regarding the accuracy and precision of measurements, which often are element specific. First, heterogeneity in sample matrix (rocks, organic matter, moisture) can decrease the efficacy of the measurements by increasing error, increasing background noise, and decreasing overall signal (Declercq et al., 2019; Ravansari et al., 2020). Second, built-in and user-built methodologies can cause differing results for elemental concentrations (Weindorf & Chakraborty, 2020). Lastly, pXRF measures total element concentrations, which may not reflect what is plant available and could lead to overestimation of nutrients (Andrade et al., 2020).

Core Ideas

- We compared soil pXRF and pseudototal nutrients and toxic elements from 10 community gardens.
- Soil pXRF and pseudototal were correlated for Mn, Cu, Zn, and Pb but not for As, Ca, K, or P.
- Soil pXRF and pseudototal were correlated with leaf Mn, Cu, and Zn and root P, Zn, and Pb.

The objectives of this study were to quantitatively evaluate if pXRF measurements of macronutrient, micronutrient, and potential toxic trace element concentrations in urban community garden soils correlate with concentrations determined by ICP-based methods and if the soil measurements correlate with plant tissue concentrations. For our first objective, we hypothesized that pXRF measurements would not be as accurate or precise as ICP-based methods but would be significantly correlated. For our second objective, we predicted that oven-drying and sieving soils would reduce sample heterogeneity and improve the accuracy and precision of pXRF measurements. Third, we hypothesized that pXRF and digestion-based ICP analyses would correlate with plant tissue concentrations, allowing for uptake prediction based upon soil concentrations. This comparison of soil concentrations via pXRF measurements with ICP methods is important to evaluate if pXRF can adequately assess nutrients and potentially toxic elemental concentrations in community garden soils. We expected that uptake of macronutrients (Ca, K, P), micronutrients (Mn, Cu, Zn), and toxic elements (As, Pb) by crops would be driven by soil concentrations, allowing for predictions of plant uptake and tissue concentrations.

2 | MATERIALS AND METHODS

2.1 | Description of community gardens

We sampled 10 community gardens (CG1–CG10) in Massachusetts, Vermont, New York, Connecticut, and Missouri during late July and early August 2020. To select these sites, we contacted 20 community gardens located in urbanized areas predominantly across the northeastern United States. We coordinated with garden managers for permission to sample and for information about management practices. To sample each community garden, we determined randomly chosen garden plots, which are defined as 6–14 m^2 areas that are

planted, watered, amended, harvested, and individually managed by an active member. For community gardens <0.40 ha, we sampled five garden plots distributed evenly across the whole garden, with the exceptions of CG4 and CG10, where we collected four and nine garden plot samples, respectively. For community gardens >0.40 ha, we sampled 10 garden plots distributed evenly across the entire garden.

Most of the community gardens are publicly owned and managed by their respective city (CG1, CG2, CG5, CG7, CG8, CG9); CG3, CG4, and CG6 are privately owned. Community gardens CG1, CG6, and CG7 had raised beds; CG3 and CG10 had in-ground beds; and CG2, CG4, CG5, CG7, CG8, and CG9 used a combination of the two. All plots within each community garden are managed by individual members. All community gardens follow organic growing methods and require organic fertilizer applications, have banned the use of pesticides and herbicides, and encourage use of natural mulches (i.e., wood chips and compost). Applications of organic fertilizers, mulches, and water are made at the discretion of each individual garden plot owner. Most gardens offered mechanical tilling at the beginning of every season, except CG3, which used strictly no-till management techniques.

2.2 | Soil and plant sampling

Mineral soil samples were collected following composite sampling guidelines as used by Mitchell et al. (2014). In brief, ~200 g of mineral soil subsamples were collected from five points within each garden plot from the top 13 cm using a clean hand trowel. The five samples were composited and physically homogenized by crushing aggregates and mixing within the bag to generate a single soil sample from each plot. This sampling scheme was carried out at each garden plot for all community gardens. In total, 73 soil samples were collected and stored at field moisture in polyethylene bags.

To further evaluate the relationship between elements in soils and plants, we collected plant samples at each plot studied, which included root-vegetable crops, fruit-producing crops, and herbaceous-leaf crops and plants from 18 genera. At each garden plot, approximately three plant species were collected, except in cases where only one plant species was present. Leaves, roots, and fruits were processed and considered separately. In total, 183 plant samples were analyzed from the 10 community gardens.

2.3 | Soil physicochemical analyses

Soil pH, SOM, and soil texture were determined for each soil sample. First, soils were oven dried at ~70°C for 24 h and sieved to <2 mm. For soil pH, 5 g of soil and 20 g of 0.01 M $CaCl_2$ were added to centrifuge tubes, shaken, and allowed to settle for 2 h. Measurements were taken using a calibrated pH meter (Fisherbrand accumet AE150 Benchtop pH meter and probe) (Thomas, 1996). Loss-on-ignition (LOI) was used to estimate SOM. To determine the SOM from LOI, 5 g of oven-dried subsample was combusted at 550°C for 6 h. Loss-on-ignition is a simple and qualitative method but may overestimate organic matter content due to mineral dehydration and carbonate thermal decomposition (Santisteban et al., 2004). For soil C analysis, 25–35 mg of ground soil was weighed into tin capsules and combusted with a Thermo Scientific Flash EA 1112 NC Analyzer (CE Elantech Inc.). Texture was determined using sedimentation columns following a modified Bouyoucos method (Gee & Bauder, 1979).

2.4 | pXRF sample processing and analyses

Soils were analyzed for elemental composition in quadruplicate measurements using a user-made pXRF calibration at two preparation stages: under field conditions (at field moisture and unsieved in 7-ml polypropylene vessels lined with 6-μm-thick polyethylene terephthalate sheet) and after drying and sieving to capture the impact of standard sample preparation (also in the 7-ml vessels). Soils were oven-dried at 70°C for at least 48 h and sieved to <2 mm. Total concentrations of macronutrients (Ca, K, P), micronutrients (Cu, Mn, Zn), and toxic elements (As, Pb) in soils were determined using a X-200 XRF (SciAps Inc.) equipped with a 20-mm^2 silicon drift detector with a 135 eV resolution (determined from the full width half maximum of the 5.95 keV Mn Kα line). Each soil measurement was carried out at three energies (40, 10, and 50 kV(for 30 s each from a Rh anode alloy X-ray tube. Spectra were analyzed using Compton Normalization (USEPA Method 6200). Quantification of soil nutrient and toxic metal concentrations was completed using a user-built analysis mode that used the internal set of standards as well as an expanded list of soil and rock standards (USGS standards BHVO-2, DNC-2, BIR-1, BCR-2, SBC-2, SDO-1, STM-2, GSP-2, W-2a) and NIST soil standards, 2709 San Joaquin Soil, 2711a Montana II Soil, for calibration. Standard Reference Material recoveries for GSP-2, NIST 2709, and NIST 2711a were Ca: 87–93%, K: 73–88%, P: 82–94%, Cu: 96–107%, Mn: 96–118%, Zn: 93–119%, As: 97–142%, and Pb: 94–103%.

2.5 | Soil and plant digests

Dried and sieved soil samples were extracted to quantify the pseudototal fractions of elements to determine their plant availability and potential ecotoxicological impact on plants. We used a strong acid digestion following USEPA

method 3050B to quantify the pseudototal fraction of elements (Richardson, 2021). A drawback to the use of a pseudototal digestion using concentrated hydrochloric-nitric acid is the inability to measure metals that are within the primary silicate mineral lattice (Chen & Ma, 1998). Each sample was digested in duplicate, and for every 30 samples, a preparation blank and SRM NIST 2710 Montana I Soil were included. Soil pseudototal metal concentrations across the community gardens are provided in Supplemental Table S1.

Plant samples were oven dried at 90°C for 24 h and mechanically ground. To determine macronutrient and trace element concentrations, digestions were carried out using a modified USEPA 3050B Method (Rechcigl & Payne, 1990), in which samples are combusted prior to strong acid, pseudototal digestion (Mackowiak et al., 2021). For every 30 samples, a preparation blank and SRM NIST 1547a Peach Leaves were included. Leaf tissue; fruit tissue; and root vegetable macronutrient, micronutrient, and toxic element concentrations across the community gardens are provided in Supplemental Tables S2–S4.

Digests were further diluted using 18.2 MΩ cm^{-1} deionized water and analyzed with an optical emission spectrometer (5110, Agilent Technologies) and 7700x ICP-Mass Spectrometer (Agilent Technologies). Concentrations of AS, Ca, Cu, Fe, K, Mg, Mn, P, Pb, and Zn in the preparation blanks were <0.1% of their respective measured concentrations, and all duplicates were within 15% CV. NIST 2710 Montana Soil SRM recoveries were 86–106% of the respected certified values for As, Cu, Fe, Mn, Pb, and Zn (Mackey et al., 2010). Recovery rates for Ca, K, Mg, and P were 71–86%, due to the incomplete dissolution of silicate minerals. Recovery rates of NIST Peach Leaf 1547a SRM were 86–106% of the respected certified values for As, Cu, Fe, Mn, Pb, and Zn (Mackey et al., 2010). Recovery rates for Ca, K, Mg, and P were 71–86%, due to the incomplete dissolution of silicate minerals.

2.6 | Descriptive and statistical analyses

Descriptive statistics were calculated using Microsoft Excel. In-text mean values are arithmetic means ± 1 SD. Linear regressions were calculated to determine the relationship between pXRF measurements and ICP optical emission spectrometry (ICP-OES) or ICP–mass spectrometry (ICP-MS) measurements and between pXRF measurements of field-condition and dried and sieved soils using Matlab (Mathworks). We determined the CV for individual samples to compare across community gardens and between field-condition pXRF and ICP methods. For pXRF measurements, the CV was determined using the quadruplicate measurements on each garden plot sample. For ICP measurements, the CV was determined using the duplicates of each garden plot sample. Due to the limited sample size, nonparametric Wilcoxon rank sign test and Kruskal–Wallis test prepackaged in Matlab were used to compare concentrations and CVs between community garden soil macronutrient, micronutrient, and toxic metal concentrations with garden plot as the experimental unit as well as plants tissue concentrations.

Relationships among soil element concentrations, soil physicochemical properties, and leaf and fruit tissue concentrations were explored using a normalized principal component analysis (PCA) in Matlab. In the PCA, macronutrient, micronutrient, and toxic element groups of data were normalized with their respective standard deviations to remove the weighted effect of data with larger numerical values. The data were nondimensionalized, and the explanatory power for components was determined. Only the two components with the highest explanatory power were considered.

3 | RESULTS AND DISCUSSION

3.1 | Soil physicochemical properties across urban community gardens

The soils across the community gardens shared some similarities in pH and sand content (Table 1) but had significant differences in C, N, and clay. Soil pH had an average of 6.7 ± 0.3 across all garden plots, with a minimum of 6.3 at CG4 and maximum of 7.2 at CG6. This was expected because carbonate amendments are commonly used to increase soil pH in gardens (e.g., Bechet et al., 2018). The sandy loam texture determined across the gardens was also expected because the areas studied were glaciated and/or near rivers. Further, sandy-textured soils are generally sought for urban agriculture (Kong et al., 2009). However, there was considerable variability in the SOM, C, and N across the gardens. For C, the garden plot soils had an average of 8.7% with a minimum of 2.5% at CG8, a high of 11% at CG6, and outliers of 22% at CG7 and CG2. Soils within the garden plots had an average N of 0.6%, with a minimum of 0.2% (CG8 and CG10) and maximum of 2.3%. Plots CG8 and CG10 had significantly lower C and N than the other gardens. We interpret the differences in C and N content as linked to differences in the application of mulch, compost, and wood chips across the gardens, especially because material availability and practices in soil management varies among community gardens (Lin et al., 2018; Tresch et al., 2018).

Supplemental Table S1 shows the ICP-OES and ICP-MS results of the pseudototal digestion of the soils in each plot. For macronutrients, soil Ca, K, and P concentrations were not significantly different ($p < .05$). For micronutrients, soil Mn concentrations were comparable ($p < .05$). Soil Cu and Zn concentrations at CG1 were significantly higher than most community gardens, whereas at CG10 they were significantly lower than all other community gardens ($p < .05$). Soil As

TABLE 1 Location, size, and USDA plant hardiness zone of each community garden

Communitygarden	Location	Size class	USDA plant hardiness zone	pH	C	N	Soil texture	Clay
		ha			%			%
CG1	Northampton, MA	1–5	6A	6.8 ± 0.4	4.5 ± 1.1	0.3 ± 0.1	sandy loam	6
CG2	South Hadley, MA	<1	6A	6.9 ± 0.1	11 ± 5	0.7 ± 0.3	sandy loam	10
CG3	Springfield, MA	<1	6A	6.8 ± 0.2	8.6 ± 3.3	0.5 ± 0.2	loamy sand	8
CG4	Troy, NY	1–5	6B	6.3 ± 0.2	7.1 ± 2.6	0.4 ± 0.1	sandy loam	7
CG5	West Hartford, CT	5–10	6B	6.8 ± 0.2	10 ± 7	0.6 ± 0.4	sandy loam	8
CG6	Buffalo, NY	<1	5B	7.2 ± 0.2	11 ± 4	0.8 ± 0.4	sandy loam	11
CG7	Springfield, MO	<1	6B	6.9 ± 0.2	22 ± 6	1.7 ± 0.4	sandy loam	14
CG8	Essex Junction, VT	<1	4B	6.5 ± 0.1	2.5 ± 0.6	0.2 ± 0.0	sandy loam	9
CG9	Burlington, VT	>10	5A	6.8 ± 0.1	6.1 ± 1.9	0.4 ± 0.1	sandy loam	8
CG10	Amherst, MA	1–5	5B	6.1 ± 0.1	3.1 ± 0.4	0.2 ± 0.0	sandy loam	2

concentrations were comparable across all sites. Soil Pb was significantly higher for CG1 than for many other sites, and CG8, CG9 and CG10 had lower Pb concentrations than all other sites.

To parse these relationships, we used Pearson linear regressions of soil properties with pseudototal concentrations (Supplemental Table S5). The higher C and clay were associated with higher Ca, K, and Mn. This suggests the addition and accumulation of organic C and N from soil amendments strongly affected Ca, K, Mn, and Zn, most likely through either adsorption and retention of the metals onto added organic complexes and exchange sites or additional sourcing from within compost and decomposing plant matter (McGrath et al., 1988; Moyin-Jesu, 2007). Moreover, the positive correlation between Cu and Pb suggests some gardens have received extensive historical pollution, likely from local point sources and non-point sources, such as automobiles (Laidlaw et al., 2012; De Silva et al., 2016).

3.2 | Comparison of field-condition soil pXRF measurements and traditional ICP analyses across urban community gardens

Field-condition pXRF measurements of Mn, Cu, Zn, and Pb concentrations exhibited moderate to strong correlation ($R^2 = .41–.73$) with pseudototal concentrations determined by traditional ICP-MS measurements on dried, sieved, homogenized soils (Figure 1). Linear regressions between pXRF and ICP-MS pseudototal concentrations yielded slopes between 0.60 and 1.0 for Mn and Pb, suggesting an underestimation by pXRF; in contrast, slopes ranged from 1.0 to 1.6 for Cu and Zn, suggesting overestimation by pXRF. Effective quantification of micronutrient (Mn, Cu, and Zn) and Pb concentrations via pXRF with moderate to high explanatory power ($R^2 > .50$) was obtained in several previous studies (Caporale et al., 2018; Gutiérrez-Ginés et al., 2013; Killbride et al., 2006; Silva et al., 2019). These results suggest that pXRF can detect most Pb concentrations >100 mg kg^{-1} but is unable to capture the variability in the lower Pb range, particularly for Pb concentrations <50 mg kg^{-1}. However, potentially hazardous concentrations of Cu may be overestimated by pXRF, as we obtained a 160% estimation of ICP concentrations by pXRF.

Field-condition pXRF measurements of Ca, K, P, and As concentrations were either weak or not significantly correlated with pseudototal digestion concentrations from traditional ICP-OES measurements on dried, sieved, homogenized soils (Figure 1). For Ca, pXRF measurements were far from the ideal 1:1 ratio and slope of 1.0. Calcium concentrations had a slope of only 0.12 and an R^2 of .20, indicating a very weak correlation and substantial underestimation of Ca by pXRF as opposed to digestion values. For K, P, and As, pXRF measurements and ICP-OES measurements were not significantly correlated with each other ($R^2 < .25$; $p > .10$) and had negative slopes.

These results (plotted in Figure 1) show substantial limitations for macronutrient correlation between pXRF measurements on field-condition soils and traditional ICP-based analyses of garden soils. For Ca, quantification by pXRF underestimated Ca concentration; the linear slope was well below the expected 1:1 ratio with low explanatory power, and there was a smaller range in measured values (note pXRF did not predict values <10 or >40 g kg^{-1}). The weak correlation of Ca, K, and P in soils by pXRF has been documented in previous studies (Zhu & Weindorf, 2009), and we primarily hypothesize two mechanisms. First, the poor correlations appear to derive from pXRF measuring elements within silicate phases, unlike the pseudototal digestion or underestimation by the calibration with concentrations above or below the dynamic range. The NIST 2709 and

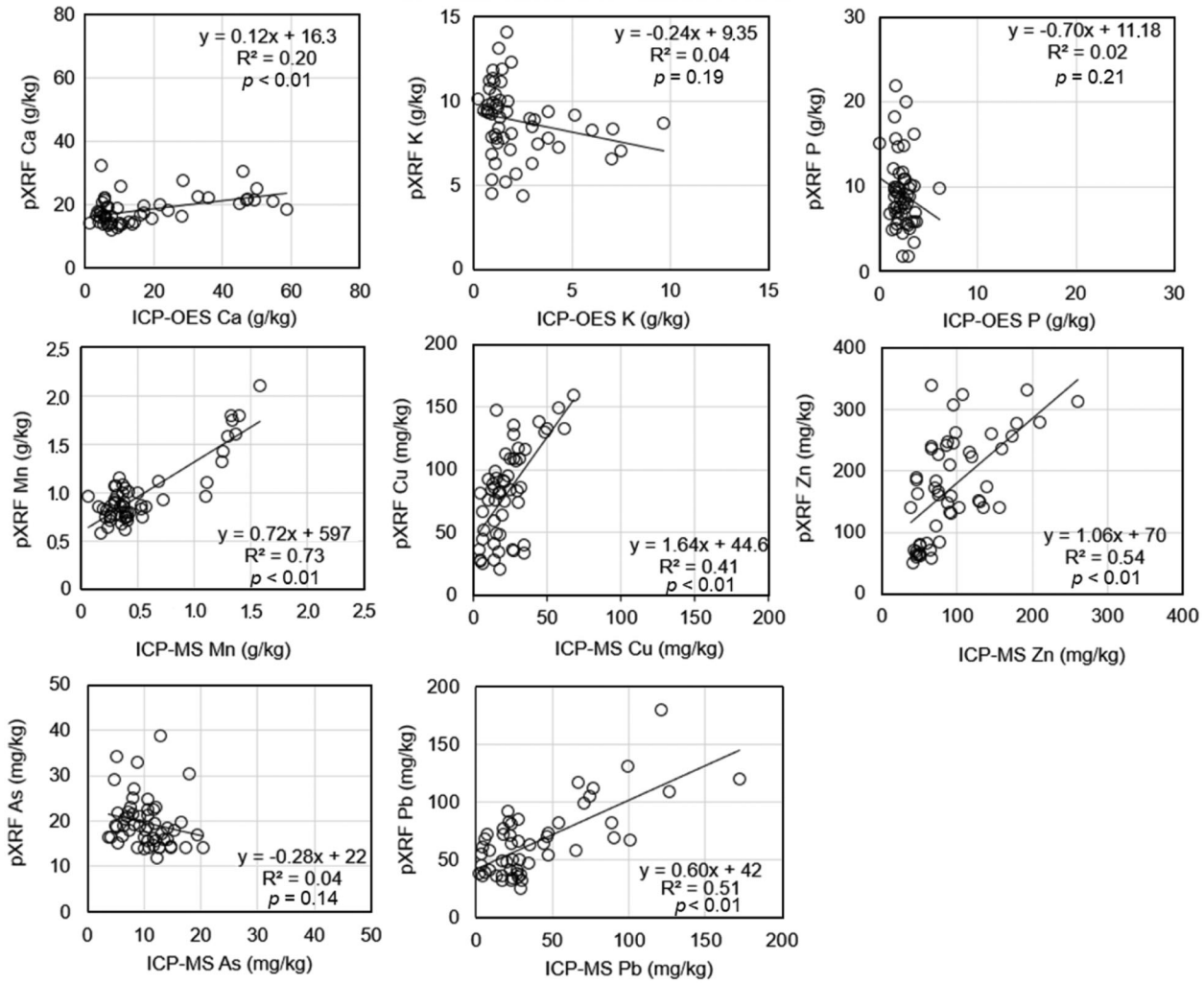

FIGURE 1 Correlations between field-condition soil portable X-ray fluorescence (pXRF) measurements with pseudototal inductively coupled plasma–optical emission spectrometry (ICP-OES) or inductively coupled plasma–mass spectrometry (ICP-MS) measurements for bulked soils from plots across all community gardens. Pearson linear regressions models are plotted, and R^2 values are shown

2711a pseudototal digestions only recovered 71–86% of the certified values for Ca, K, and P, demonstrating the inability to dissolve silicates. Becausee we did not find a significant relative correlation for K and P, there may be limited opportunities for correction factors to improve estimates. Although the pseudototal digestion of nonsilicate elements is a mismatch with the total measurement by pXRF, pseudototal concentrations are a more effective measure of metals that can be accessed by plants immediately or over several growing seasons (Gutiérrez-Ginés et al., 2013; Pelegrino et al., 2021).

Previous studies obtained mixed results for measuring Ca, K, P, and As when comparing pXRF measurements with traditional soil analysis methods. Sharma et al. (2015) and Silva et al. (2019) were able to quantify total Ca and K concentrations by pXRF with high explanatory power ($R^2 > .50$), whereas Caporale et al. (2018), Gutiérrez-Ginés et al. (2013), Nawar et al. (2019) had poor correlation between pXRF and ICP-based analyses. In addition to differences in instruments and advancing hardware and software, there are some key differences between the previous studies and our study in methodology that explain the low predictability of Ca, K, P, and As. Our study investigated a smaller set of sites than other studies that leveraged samples from substantially varying soils, such as Sharma et al. (2015), who investigated 75 soil profiles across two large states, and Nawar et al. (2019), who analyzed 105 samples from 10 different countries. Due to the limited range in soil properties and element concentrations, linear regressions were less efficient in building accurate model predictions because of the smaller ranges in values and were more likely to be affected by outliers (as a prime example, see As, Fe, and K in Caporale et al. [2018]). Lastly, the high C in many of the soils likely decreased the accuracy and precision of measurements by increasing the attenuation of X-ray and Compton scattering associated with higher SOM (Declercq et al., 2019; Ravansari & Lemke, 2018).

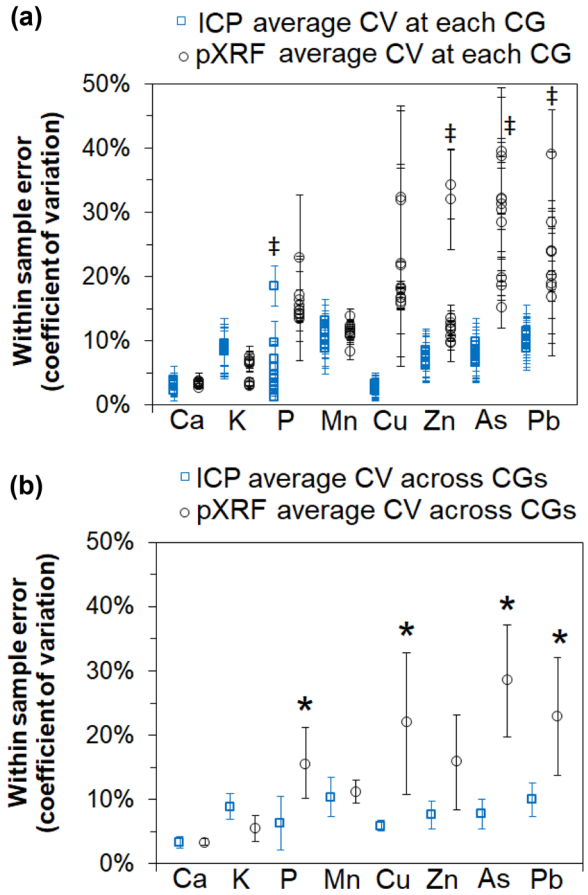

FIGURE 2 (a) Average within-sample CVs for each of the 10 community garden (CG) samples for inductively coupled plasma (ICP) measurements in blue squares and portable X-ray fluorescence (pXRF) measurements in black circles. ‡Significant difference among average CVs at community gardens with pXRF or ICP measurements. (b) Average within-sample CVs across the 10 CGs. *Significant difference between withi- sample CVs for pXRF and ICP measurements. Error bars are SD

We determined the CV for individual samples across community gardens and between field-condition pXRF and ICP methods to assess the variability of the two methods and among gardens. From Figure 2a, we see that many of the macronutrients and micronutrients had comparable CVs among the community gardens, ranging between 3 and 22%. Only ICP analyses of P and field-condition pXRF analysis of Zn had a significant difference among the 10 community gardens. Arsenic and Pb had several significant differences among the 10 community gardens, with CVs ranging from 15 to 39%. Considering the 10 community gardens together, we observed significantly higher CVs for field-condition pXRF measurements of P, Cu, As, and Pb than ICP measurements. These results highlight that traditional ICP methods can dramatically reduce the within-sample variability for elemental analyses, particularly for trace elements. We did not observe increases in CV with decreases in concentrations (Supplemental Figure S1). Our results agree with previous studies on the higher variability of elemental concentrations measured (Caporale et al., 2018; Gutiérrez-Ginés et al., 2013; Killbride et al., 2006; Silva et al., 2019). The CV can be attributed to overlapping peaks (e.g., Pb for As) or interferences from high concentrations of light elements (e.g., C, O, and Si).

3.3 | Comparison of field-condition soil pXRF measurements and dried, sieved soil pXRF measurements

We investigated if pXRF measurements of macronutrients, micronutrients, and toxic elements in field condition soils would be significantly correlated to pXRF measurements on soil samples prepared using traditional drying and sieving (Figure 3). Slopes for linear regressions comparing field-condition soil pXRF measurements and dried, sieved soil pXRF measurements were close for Ca, P, Mn, Cu, Zn, and Pb (range, 0.89–1.14) (Figure 3). The slopes were not far from the 1:1 line at 0.13 (Figure 3). Potassium had a close to ideal 1:1 ratio with a slope of 0.95 but a low explanatory power (R^2 = .28), suggesting an overall substantial interference and variation in K measurement by pXRF. This could be due to heterogeneity of K within and not within silicates when mixed between quadruple measurements. Arsenic measurement had very low reliability and demonstrated high within-sample variability. Arsenic concentrations are at the lower limit for quantification, which negatively affected the ability to quantify As.

Although some previous studies found that moisture and particle size heterogeneity can reduce the accuracy and precision of measurements (e.g., Ravansari et al., 2020), our study agrees with other studies that found field moisture did not substantially affect the pXRF-measured concentrations of macronutrients, micronutrients, and Pb (Killbride et al., 2006; Markey et al., 2008; Parsons et al., 2013), except As, which failed to be measured under field moist as well as dried and sieved conditions. From our results, we can conclude that pXRF measurements of field condition soils were generally as accurate as dried, sieved soil pXRF measurements, but there were still limitations for K and As by the SciAps X-200 pXRF.

3.4 | Plant tissue concentrations

3.4.1 | Differences across community gardens and soil concentrations

Plant tissues were separated into three categories: fruits (fruit-producing crops), leafy (tissues from herbaceous crops in which leaves are consumed), and root (crops from which

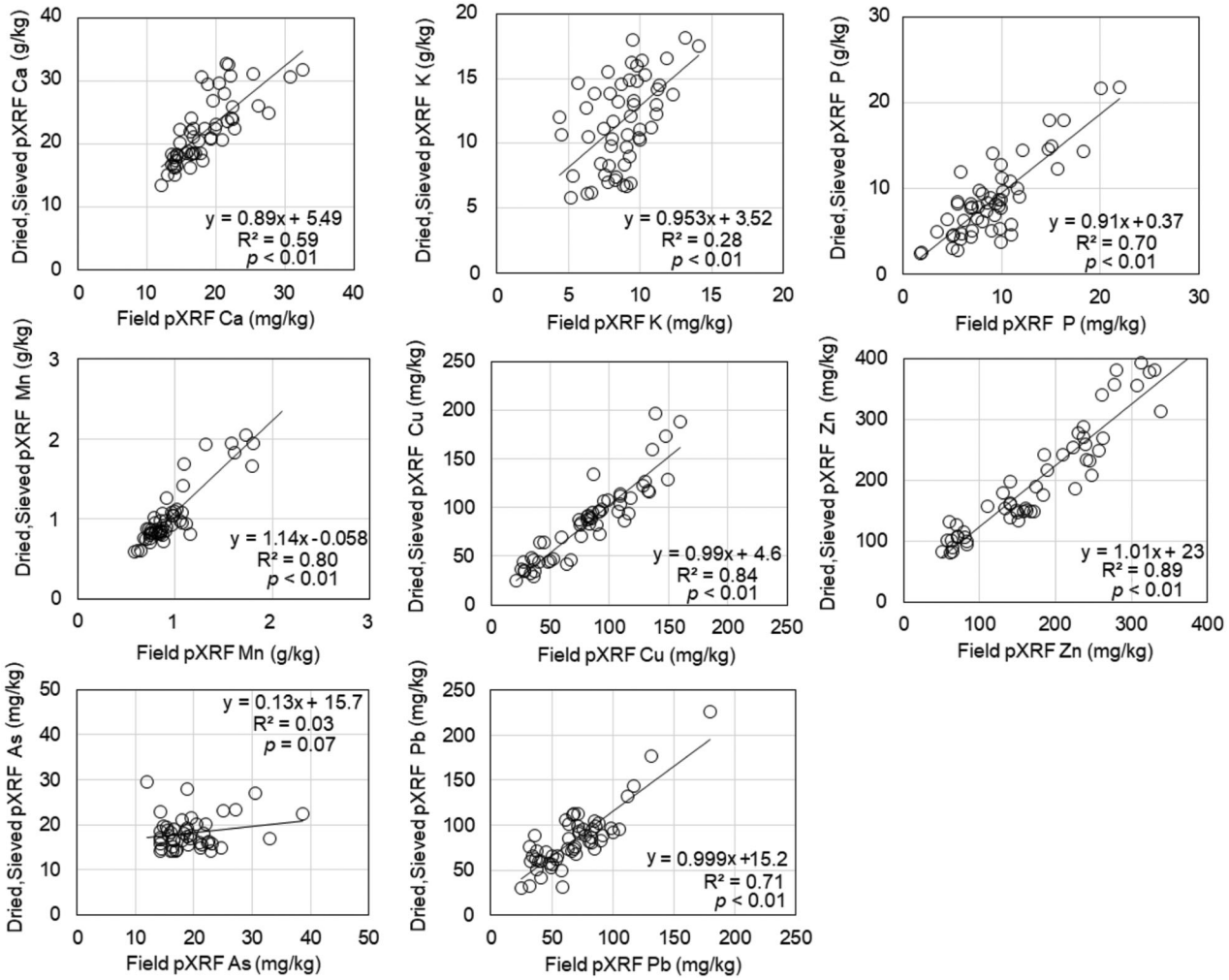

FIGURE 3 Correlations between field-condition soil portable X-ray fluorescence (pXRF) measurements with dried, sieved soil pXRF measurements. Pearson linear regressions models are plotted, and R^2 values are shown

the roots are consumed). Genera present at each community garden are listed in Supplemental Table S6, and their fruit, leaf, or root concentrations are given in Supplemental Table S7. Most community gardens had comparable leafy macronutrient concentrations, but CG8 had higher and CG2 had lower Ca, K, and P concentrations. Leafy micronutrient concentrations were generally comparable across community gardens for Zn, but there were significant differences for Mn and Cu. Leafy As and Pb concentrations were without a clear pattern among community gardens.

Similarly, fruit crops also exhibited significant differences among community gardens. Fruit Ca and K had significant variations among community gardens, but fruit P concentrations were comparable. Fruit micronutrient concentrations of Mn, Cu, and Zn varied by nearly an order of magnitude across community gardens. Fruit As concentrations were significantly higher for CG7 and CG9 than for CG2 and CG5, but fruit Pb concentrations did not significantly differ across community gardens. There were few substantial variations in macronutrient, micronutrient, and toxic element concentrations in root crops (carrots of the genus *Daucus* and beets of the genus *Beta*) among community gardens, most likely due to limited sample size. Root crops were only collected at six gardens with small sample sizes, which limited the ability to detect significant differences in root tissue concentrations of macronutrients, micronutrients, and toxic elements.

These results show that fruit, leaf, and root macronutrient, micronutrient, and toxic trace element concentrations in the urban gardens did not follow a consistent pattern whether in larger cities or in more rural areas or in larger or smaller community gardens. Instead, we observed element-specific enrichment for some community gardens, which we interpret as site management and site history being dominant factors. This agrees with many previous studies that found site-specific characteristics as key factors controlling enrichment in soils and plants (Bechet et al., 2018; Li et al., 2013; Mitchell et al., 2014; Tresch et al., 2018). Furthermore, differences among genera and their propensity to accumulate nutrient and toxic metals within their tissues also limited the ability to detect significant differences among the

community gardens due to their physiology and growth habits (Awino et al., 2021; Bechet et al., 2018; Gupta et al., 2019; Ricachenevsky et al., 2018).

3.4.2 | Predicting plant tissue uptake from soil concentrations

We aimed to determine if pXRF or ICP-based pseudototal concentrations (operationally defined, ICP-based, salt extraction) were able to predict fruit, leaf, and root total macronutrient, micronutrient, and toxic element concentrations. Only fruit Pb concentrations were predicted by pXRF or pseudototal concentrations. All other macronutrient, micronutrient, and As concentrations were poorly estimated by pXRF and pseudototal concentrations in their respective soils. Leaf Mn, Cu, and Zn were significantly correlated with pXRF and pseudototal Mn, Cu, and Zn soil concentrations (Supplemental Table S2). Pseudototal Pb soil concentrations, but not pXRF Pb soil concentrations, were also correlated with leaf concentrations. Leaf macronutrient and toxic element concentrations were not correlated with soil pXRF and pseudototal concentrations. Root P, Zn, and Pb concentrations were positively correlated with pXRF and pseudototal soil concentrations (Supplemental Table S8). Pseudototal Ca soil concentrations were correlated with root concentrations but not soil pXRF measurements (Supplemental Table S8).

The variances in soil concentrations, soil physicochemical properties, and plant tissue (leaves and fruits only) concentrations were examined using PCA (Figure 4). For macronutrients, PC1 and PC2 had an explanatory power of 30 and 17%, respectively. The variance in plant tissue Ca, P, and K was largely not related with the respective soil pXRF or ICP concentrations or with soil physicochemical properties. For micronutrients, PC1 and PC2 had an explanatory power of 33 and 23%, respectively. The variance in plant Mn, Cu, and Zn exhibited association with their respective soil pXRF and ICP concentrations but were not related or were inversely related to soil properties. For As and Pb, PC1 and PC2 had an explanatory power of 35 and 18%, respectively. Variance in soil pXRF and ICP Pb concentrations was associated with variance in plant tissue Pb concentrations. Soil ICP and plant tissue As concentrations as well as SOM and clay content were associated.

Our results suggest that soil pXRF and ICP measurements were unable to generally predict macronutrient, micronutrient, and toxic elements in leaves and fruits. This agrees with similar findings by McBride et al. (2014), who did not find a strong correlation between soil concentrations and plant tissue concentrations of Pb. The wide variation in plant genera likely negatively affected the creation of linear regression models to link soil elemental concentrations with aboveground tissue concentrations. The plants in our study likely have varying rates of bioaccumulation of elements or actively restrict translocation from roots to shoots to fruits (Ricachenevsky et al., 2018). In addition, plants can receive nutrient and toxic metals from the atmosphere as gaseous, dissolved, or particulate matter (Egendorf et al., 2021), which can increase

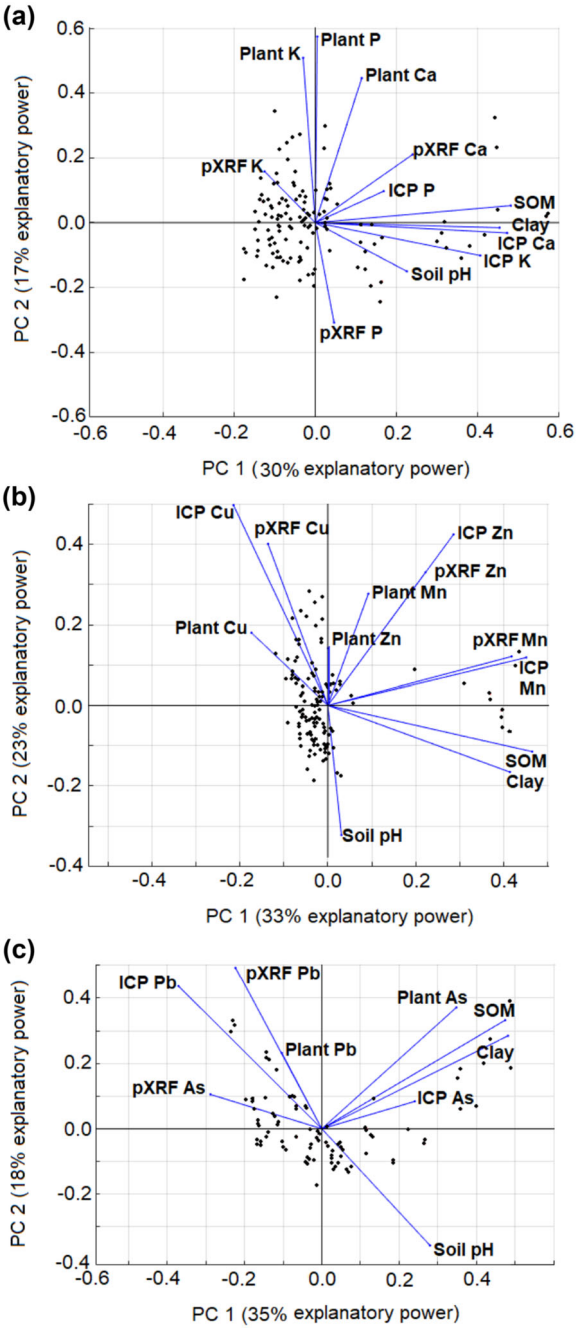

FIGURE 4 Principal component analysis of soil element concentrations by inductively coupled plasma (ICP) and field-condition portable X-ray fluorescence (pXRF), soil physicochemical properties (pH, soil organic matter [SOM], clay), and plant tissue (leaf and fruit) concentrations. (a) Macronutrients (Ca, K, and P) in soils and plant tissues. (b) Micronutrients (Mn, Cu, and Zn) in soils and plant tissues. (c) Toxic elements (As and Pb)

concentrations in aboveground structures (Xiong et al., 2016). The combination of these effects has limited the ability to link soil concentrations with fruit, leaf, and root concentrations.

4 | CONCLUSIONS

From the field analyses of soils and plants by pXRF and ICP-based digestion analyses, we can conclude that both methods can provide agreeable estimates of some micronutrient (Cu, Mn, and Zn) and toxic element (Pb) concentrations but may not agree for other elements (Ca, K, P, As) due to differences in their measurement at the fundamental level. Macronutrients within silicates can be measured by pXRF but not by pseudototal digestions or plant-available extractions, which partially explains the poor correlation between methods. Sieving and drying did not improve the correlation between pXRF and pseudototal digestions. Moreover, measurements of sieved and dried soils were comparable with field-condition soils, except for As and K. Our results highlight that pXRF can be effective for determing concentrations of Mn, Cu, Zn, and Pb. As shown in previous studies, additional models built from regional soils are often needed to overcome analytical limitations.

Macronutrient and micronutrient concentrations in leaves, fruits, and roots could not be effectively estimated by pXRF or ICP-based soil analyses. Root uptake of As and Pb was strongly correlated with soil concentrations, demonstrating that direct uptake by roots is driven by toxic metal availability in soils, but translocation to aerial parts is limited by physiological factors. However, building genus- or species-specific models and larger sample sizes could improve prediction of fruit and leaf macronutrient, micronutrient, and toxic element concentrations in community gardens.

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AUTHOR CONTRIBUTIONS

Ainsley C. McStay: Data curation; Methodology; Visualization; Writing – original draft. Sandra L. Walser: Formal analysis; Methodology; Resources; Writing – review & editing. Nicolas Perdrial: Funding acquisition; Investigation; Methodology; Project administration; Supervision; Writing – review & editing. Eric C. Sirkovich: Conceptualization; Methodology; Writing – review & editing. Justin B. Richardson: Conceptualization; Data curation; Formal analysis; Funding acquisition; Investigation; Methodology; Project administration; Visualization; Writing – original draft; Writing – review & editing.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

ORCID

Nicolas Perdrial https://orcid.org/0000-0002-0308-3708
Justin B. Richardson https://orcid.org/0000-0002-7699-4596

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