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Characterization and within-site variation of environmental metal concentrations around a contaminated site using a community-engaged approach

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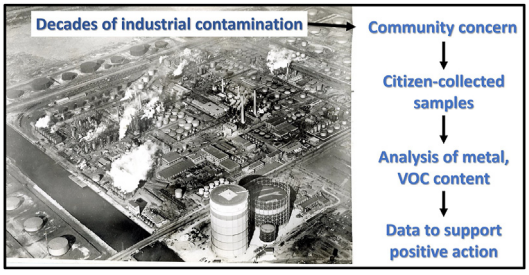
HIGHLIGHTS

- Community members collected soil and dust samples from an area with a former lead smelter and lead arsenate manufacturer.
- Many soil samples exceeded recommended US EPA screening guidelines for As (42.4%), Pb (35.4%), and Mn (79.1%).
- Several sites had measurements both above and below screening levels for As (35.7%), Pb (32.1%), and Mn (32.1%).
- Site proximity was associated with soil concentrations exceeding guidelines for As ($p = 0.028$) andPb ($p = 0.023$).
- Decisions about site remediation should consider within-site variability in order to optimally protect public health.

GRAPHICAL ABSTRACT



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ABSTRACT

Historic industrial activity led to extensive lead and arsenic contamination within residential areas of East Chicago, Indiana, United States. Although remediation is underway, community concerns about this contamination remain. Therefore, the goal for this analysis was to characterize environmental contamination in soil within and around these areas. A total of 228 samples from 32 different sites (addresses) were collected by community members or study staff. These were analyzed for metals using portable x-ray fluorescence or inductively coupled plasma optical emission spectroscopy. Concentrations exceeding EPA screening levels were found for 42% of the soil arsenic samples, 35% of the soil lead samples, and 79%

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of the soil manganese samples; a few samples also contained elevated copper or zinc. Concentrations above EPA screening levels were identified both within and outside of the formally designated contaminated area. Roughly 30% of all sites had at least one sample above and one sample below the screening level for arsenic, lead, and manganese. For sites within the contaminated area, more than 90% (arsenic), 60% (lead) and 60% (manganese) of the samples exceeded EPA screening levels. There was a significant association of proximity to the historic industrial site with elevated soil concentrations of arsenic and lead; a similar association was present for manganese. These results are consistent with existing data for lead and arsenic and we additionally report elevated concentrations of manganese and a high within-site variability of all metal concentrations. These findings should be considered in future remediation efforts.

1. Introduction

Exposure to elevated concentrations of several metals, including arsenic, lead and manganese, can result in substantial negative health outcomes. Long-term exposure to arsenic is associated with skin lesions (Haque et al., 2003), lung, liver, kidney, and bladder cancers (Jomova et al., 2011; Kuo et al., 2017; Smith et al., 1992), impairments in immunity and respiratory health (Attreed et al., 2017; Sanchez et al., 2016; Tolins et al., 2014), low birthweight (Hopenhayn et al., 2003) and impaired neurodevelopment (Rodrigues et al., 2016). Chronic exposure to low concentrations of lead (<10 μg/dL blood lead) among children has is associated with impaired neurodevelopment, delayed puberty, and reduced pre- and postnatal growth (National Toxicology Program, 2012). Among adults, low level lead exposure is associated with impaired renal function, hypertension, essential tremor, and there is increasing evidence for cognitive decline (Loef et al., 2011; National Toxicology Program, 2012; Vlasak et al., 2019). Manganese is an essential micronutrient, but elevated exposure can still result in neurotoxicity. Elevated exposure to manganese has been associated with both motor (Bowler et al., 2016; Ellingsen et al., 2015) and cognitive (Lucchini et al., 2014; Park et al., 2014) function impairments among adults. Additionally, several studies have reported that early-life exposure to manganese may result in impaired neurodevelopment (Chung et al., 2015; Claus Henn et al., 2012; Riojas-Rodríguez et al., 2010).

Many soils in urban areas have been reported to have high metal concentrations (Aelion et al., 2009; Filippelli et al., 2018; Harvey et al., 2017; Misenheimer et al., 2018). Environmental arsenic, lead, and manganese contamination can occur from their use in various industries, including mining, smelting, coal burning, and manufacturing processes which include these metals (ATSDR, 2019, 2012, 2007). For example, lead smelters can cause significant soil contamination in neighboring communities (Chai et al., 2015; Tawinteung et al., 2005); human exposure specifically from metal contaminated soils near smelters has also been documented (Carrizales et al., 2006; Ramirez-Andreotta et al., 2013; Zahran et al., 2013).

The U.S. Smelter and Lead Refinery, Inc. was a 79-acre lead smelting facility located in East Chicago, Indiana (Fig. 1). From 1906 to 1985, the smelter operated in multiple ways: first as a primary copper smelter, then as a primary lead smelter (processing ore), and in 1973 as a secondary lead smelter (recycling Pb). This site along with nearby commercial, governmental, and residential areas was added to the US EPA's National Priority List in 2009 as the USS Lead Superfund Site (US EPA, 2017). The residential areas were divided into three zones; Zone 1 was located on top of one of the former lead smelters. On the south border of the residential areas is a former DuPont facility, which manufactured the pesticide lead arsenate from 1910 to 1949. This site is also being remediated by the US EPA as a Resource Conservation and Recovery Act (RCRA) order (US EPA, 2018). A raw fuel storage facility and an iron smelter/steel manufacturer are also adjacent to this site. Thus, while the USS Lead Smelter undoubtedly played a large role in the environmental contamination, other industrial operations likely contributed.

Between 2006 and 2016, periodic soil testing and remediation of fewer than 4% of the properties in Zone 1 were completed (Reese, 2016). A 2011 report from the US Agency for Toxic Substances and Disease Registry concluded that health risks in the area had been successfully addressed (US ATSDR, 2011). However, subsequent soil testing from 2014 to 2015 identified several soil lead and arsenic concentrations which were above regulatory limits (US EPA, 2017). More extensive remediation activities were initiated in 2016. An elementary school and a low-income housing complex, both located in Zone 1, were closed and the housing complex residents were told they had to relocate (Reese, 2016). Increased monitoring and remediation for Zones 2 and 3 were initiated; residences were eligible to have their yards remediated only if soil testing indicated values of lead and/or arsenic above regulatory guidelines (US EPA, 2017).

Prior work has determined that soil lead and arsenic concentrations are substantially elevated in the USS Lead Superfund Site compared to other parts of East Chicago (US ATSDR, 2011; US EPA, 2017). However, there has only been limited research on the possibility of contamination outside of the site boundaries (Dietrich et al., 2019); meanwhile, prior studies have identified that contamination from lead smelters may extend for up to 1 km away from the site (Soto-Jiménez and Olvera-Balderas, 2018).

Residents living in and near the Superfund site have been understandably concerned about the extent and risks from this environmental contamination (Reese and Cross, 2017). Additionally, there are limited data regarding the variability of soil contamination in this area. Given that decisions about whether or not to remediate individual yards are based on a limited number of samples per yard, the issue of sample variability is important. Therefore, we worked with residents living in and beyond the Superfund site to conduct additional environmental testing. The goals of this analysis are to improve our understanding about the variability of metal concentrations in soils, the extent of metal contamination outside of the formal Superfund site and adjacent DuPont site, and whether there are elevated concentrations of metals other than lead and arsenic.

2. Materials and methods

2.1. Study design and community engagement

We present a cross-sectional analysis of lead, arsenic, and other elements measured in soil and dust in and around the USS Lead Superfund Site in East Chicago, Indiana from 2016 through 2018. A

Fig. 1. Satellite image of the USS Lead Superfund Site ("EPA Zones"), an additional site for USS Lead, and the DuPont Factory. White circles on map are fuel storage tanks. Inset: Location of northern Lake County, Indiana within the Great Lakes region of the United States.

flowchart depicting collection and selection of samples for use in this analysis is included in Appendix A: Supplementary data (Figure A1). Soil samples were brought in by individual community members to "XRF Days" held in East Chicago by the investigators. Sample collection methodologies typically involved collection of surface samples in plastic baggies that had been partially dried. Rigorous collection and processing methodologies were generally avoided to encourage maximal community participation. Investigators from Valparaiso University (VU) were also invited to some residences to collect both soil and dust samples. The choice of sites included in analysis, the number of samples analyzed per site, and the specific location within the residence that they were collected were all determined by the residents. The study investigators were solely responsible for the research questions, the experimental determination of metal content, and data analysis.

XRF Days were advertised in the local newspaper and via flyers distributed throughout northern Lake County (Reese, 2018). Some residents volunteered to assist with flyer distribution. At the XRF Day, residents brought in surface soil samples and waited while the investigators from all participating institutions analyzed their samples in resealable plastic bags with a hand-held x-ray fluorescence (XRF) instrument. Residents brought in samples from 32 different sites around northern Lake County. Community members were encouraged to provide the address and descriptive location where the sample was taken (i.e., front yard), but were not required to do so in order to have the samples screened. Results of the analysis were immediately provided to the residents along with a handout with additional information about the soil screening guidelines as well as tips about steps that can be taken to prevent exposure to metals in soil similar to those provided in public health campaigns about lead prevention, recommendation of a well-balanced diet including calcium, and avoiding eating food grown in potentially contaminated soils. Investigators were available to discuss the implication of the soil metal concentrations with residents at the time of soil screening. Investigators also attended several community meetings to answer any additional questions.

VU investigators visited 14 sites located within the USS Lead Superfund Site to collect both outdoor soil and indoor dust samples. These were analyzed with either X-ray Fluorescence (XRF) or Inductively-Coupled Plasma – Optical Emission Spectroscopy (ICP-OES). Samples within the USS Lead Superfund Site were collected with participation from local residents. Surface and core samples were taken at various depths reaching nearly 12″ below the surface with a T-style stainless steel coring sampler. Cores were extracted with divisions of roughly 6″ below surface level and cataloged for processing. Each sample was freeze dried prior to sieving to isolate the fine earth fraction (<2 mm) and homogenized before elemental analysis.

2.2. Metal and element measurement

A total of 414 samples were collected from 44 different sites. Arsenic (As), chromium (Cr), copper (Cu), manganese (Mn), lead (Pb), strontium (Sr), titanium (Ti), vanadium (V) and zinc (Zn) were measured in environmental samples. A summary of elements measured and detection limits are presented in Appendix A

(Tables A1 and A2).

Samples were analyzed with three different handheld XRF spectrometers. All analyses were conducted by trained research staff to ensure that these were completed with high accuracy and repeatability. A typical instrument used was the SciAps X-100 XRF Spectrometer which was calibrated each day with an alloy cap and regularly checked against a NIST lead-containing soil standard (NIST SRM 2586). The spectrometer was set upright on a benchtop and each sample was placed directly on top of the device; all samples remained inside the thin-walled resealable bags during analysis. The limit of detection for lead was about 12 ppm and the limit of detection for arsenic was about 1 ppm. Lead and arsenic overlap in the XRF spectra was handled by simultaneous fitting of K_α and K_β lines for arsenic and $L_{\alpha\beta\gamma}$ lines for lead. Replicate measurements of the NIST standard showed that accurate soil lead results were obtained through the thickness of the plastic sample bag. For this study, the SciAps X100 XRF was configured to measure 19 elements simultaneously. Each sample was analyzed with a 15 keV X-ray beam for 20 s and a 40 keV X-ray beam for 20 s, both generated from a 40 kV gold anode. The other XRFs had very similar operating parameters and limits of detections, and several samples were run in replicate to confirm that similar results were obtained.

ICP-OES analysis was performed on a Perkin Elmer Optima 8000 instrument. Similarly, all sample preparation and analysis for ICP-OES was conducted by trained research staff to increase quality and consistency of results. Acid digestion of solid samples followed EPA method 3050B for sediments, sludges, and soils; specifically, procedural subsection 7.5 for antimony and lead. Briefly, samples of around 1 g were refluxed with heat in a 1:4 mixture of concentrated HCl and HNO_3 acids, and the resulting solution filtered and diluted appropriately. Measurements were taken with both axial and radial viewing optics with 2 replicate measurements and averages for lead concentrations. A yttrium internal standard was used in every sample. Multi-elemental standards were prepared for in 5% nitric acid to match the matrix of the samples. The gas flow rates and sample flow rates are listed in Appendix A (Table A3). The radio-frequency power was set to 1500 W.

2.3. Additional variables

Regional soil screening levels are health-based guidelines used by the US EPA for decisions regarding soil remediation (US EPA, 2020). Here, we use regional screening levels for residential soil except for Pb and As, which have established soil and dust screening levels for the USS Lead Superfund Site (As in soil or dust: 26 ppm; Pb in soil: 400 ppm; Pb in dust: 316 ppm) (US EPA, 2017). A total hazard quotient of 0.1 was used because we were screening for multiple compounds, as recommended by the US EPA (US EPA, 2020). Notably, soil screening levels may vary considerably based on assumptions; for example, industrial soil screening levels are generally higher, and when screening for an individual contaminant a hazard quotient of 1 is recommended. For manganese, this results in soil screening values that range from 180 ppm to 26,000 ppm (US EPA, 2020). In this analysis, we used the lowest screening level within this range as this matched our criteria above. Most residential addresses were provided at the time of the soil screening. These were used to determine whether the site was located in the USS Superfund Site as well as for the US Census block group. Block group level demographic data were obtained from the 2017 estimates from the US Census's American Community Survey (US Census Bureau, 2020) for median age and median income in the past 12 months. Census-trace level race/ethnicity data was also obtained; percent non-Hispanic white was used in analyses as a predictor of racial impact.

2.4. Data analysis

Statistical analyses were performed using Stata 13.1 (College Station, Texas, USA). Maps were created using ArcGIS (ESRI Systems, Redlands, CA) and R (R Core Team, Vienna, Austria). A p-value <0.05 was considered to be statistically significant.

A total of 44 sites with 415 samples were provided or requested for analysis by residents (Figure S1). For this analysis, we excluded sites that we were not able to code to a specific city block group (8 sites with 26 samples) because these could not be matched with Census data. Samples that were not soil or dust samples were also excluded (82 samples from 13 sites). This was done to enhance comparability within the dataset. A total of 52.3% of the soil samples collected with XRF were provided by only three different sites. To prevent these sites from providing any undue influence on the results, a subset of $N = 15$ samples from each of these three sites were randomly selected to include in analyses. This left a total of 32 sites with 228 samples. The combination of media/analysis method for each sample were as follows: soil/XRF (28 sites, 158 samples), dust/XRF (4 sites, 39 samples); soil/ICP-OES (7 sites, 21 samples) and dust/ICP-OES (3 sites, 10 samples). Several sites contributed samples to multiple media/method sample categories. Very few samples used both XRF and ICP-OES analytic methods, so we were unable to directly compare results by analysis method. To ensure that results are not influenced by media or analysis method, analyses of the soil/XRF data are presented in the main paper; results using all samples are available in Appendix A (Tables A4-A7; Figure A2-A5).

The metal concentrations are lognormally distributed and are described using maps and tabulated summaries of variable distributions. Box plots were used to visually present the variance in sample concentrations between and within sites. Soil element concentrations were also compared to EPA screening levels. Both unadjusted and adjusted generalized estimating equations (GEE) were used to compare individual metal concentrations which exceeded guidelines with proximity to the former lead smelter and pesticide manufacturer. Categories, in increasing proximity to the former industrial sites, were Lake County excluding sites in East Chicago, East Chicago excluding sites in the USS Lead Superfund Site, Zone 3 of the USS Lead Superfund Site, and Zone 2 of the USS Lead Superfund Site. Adjusted models included the following covariates: distance from the nearest major roadway (continuous), median age within the block group (continuous), median income within the block group (continuous), median percent white within the block group (continuous), the median year of housing construction within the block group (continuous; lead models only), the type of sample (soil/dust; models with all samples only) and the method of analysis (XRF/ICP-OES; models with all samples only). GEE analysis is a common method for handling clustered data that estimates the parameters of a generalized linear model (Liang and Zeger, 1986; Lumley, 1996).

3. Results

This analysis includes 228 dust or soil samples taken from 32 sites located in northwest Indiana; the largest subset of these are soil samples analyzed by XRF ($n = 158$ samples, $N = 28$ sites). There were two sites which contributed samples using both ICP-OES and XRF. Site characteristics for soil XRF data are presented in Table 1; similar data for all samples are presented in Table A4. Roughly half of the sites were located in East Chicago. The majority of sites were located in census blocks with a median housing age earlier than 1950, 15% or fewer non-Hispanic white residents, and a median income less than $40,000 per year. Results were similar in the group of all samples as well as the subset of soil samples analyzed

Table 1
Site characteristics, N = 28 sites.

Characteristic	N	Percent
Proximity to former smelter site		
Lake County, excluding East Chicago	14	50.0
East Chicago, excluding USS Lead	4	14.3
USS Lead, Zone 3	8	28.6
USS Lead, Zone 2	2	7.1
Number of samples per site		
1 sample/site	5	17.9
2–3 samples/site	8	28.6
4–7 samples/site	6	21.4
≥8 samples/site	9	32.1
Distance to nearest major road		
≤1500 m	16	57.1
>1500 m	12	42.9
Median year housing built[a]		
Before 1950	21	75.0
1950 to 1978	5	17.9
1978 or later	2	7.1
Median age[a]		
21.3–40.9 years old	10	35.7
41.0–50.2 years old	18	64.3
Percent NH White[a]		
≤15%	16	57.1
>15%	12	42.9
Median income[a]		
< $40,000	12	42.9
$40,000 to $60,000	7	25.0
> $60,000	9	32.1

NH = non-Hispanic. Includes sites with soil samples analyzed with X-ray fluorescence. Sum of percentages may not be 100 due to rounding. a.. Summary measures by block group.

by XRF.

Table 2 shows descriptive statistics for soil concentrations analyzed by XRF. At least one sample was found to be above the recommended soil screening levels for As, Cu, Mn, Pb and Zn. The majority of samples which exceeded the screening level were located within the USS Lead Superfund Site. However, there were still concentrations that exceeded the screening level among of the 56 samples collected outside the Superfund site: N = 16 (28.6%) for As; N = 39 (69.6%) for Mn; and N = 7 (12.5%) for Pb. Results were very similar among all samples (Table A5). Maps indicating the median soil As, Pb, and Mn concentration for all samples, are included in Appendix A (Figures A2-A4).

Table 3 presents the proportion of sites which had samples higher than EPA screening levels for soil samples analyzed with XRF; this is stratified by a) all sites and b) sites that were not in the USS Lead Superfund Site. Among all sites, a larger percentage of sites had at least one sample above the screening level versus having the median site concentration above the screening level. Among all the sites (N = 28), median soil concentrations above the screening level were 35.7% for As, 78.6% for Mn, and 25.0% for Pb. Results were similar, but overall percentage of exceedances was lower, among the subset of sites that were not located within the USS Lead Superfund Site. Results were similar among all included samples (Table A6).

Fig. 2 displays the within- and between-site variability for As, Pb, and Mn concentrations as well as the distribution of sample concentrations with respect to the EPA screening levels. Very few sites had all samples above the EPA screening level (N = 1 for As; N = 1 for Pb, and N = 2 for Mn); it was more common to have a mix of samples that were above and below the screening level. Out of the N = 28 sites, there were several sites where at least one sample was below the screening level and at least one sample was above the screening level. Specifically, for As this was N = 10 (35.7%), for Mn this was N = 9 (32.1%) and for Pb it was N = 9 (32.1%). These percentages were higher for site located within Superfund Zones 2 or 3 (N = 10): for As this was N = 9 (90%); for Mn this was N = 6 (60%) and for Pb this was N = 6 (60%). Results were similar among all included samples (Figure A5).

Generalized estimating equations were used to determine the association having an elevated metal concentration with closer proximity to the Superfund site (Table 4). For soil samples analyzed with XRF, there was a statistically significant association between having an average elevated As or Pb concentration per site with closer proximity to the USS Lead Superfund Site (adjusted model *p*-values: As = 0.03; Pb = 0.02). This association was somewhat attenuated for Mn (adjusted model *p*-value = 0.11). When all samples were included in the analysis, model results were similar, except that the unadjusted model for Mn was also statistically significant (Table A7).

4. Discussion

This report summarizes the elemental composition of soil and dust both within and nearby a Superfund site with noted Pb and As contamination. There were individual samples which exceeded EPA soil screening guidelines for As, Cu, Mn, Pb and Zn. When looking at median site concentrations, screening levels for As, Mn and Pb were exceeded. A high within- and between-site variability of As, Mn, and Pb concentrations was also observed. Results from unadjusted and adjusted GEE models suggest that higher concentrations of XRF-measured soil As and Pb were significantly associated with proximity to the USS Lead Superfund site and the adjacent Dupont plant. When considering all collected samples, a significant association of Mn with proximity to these sites was also observed in the unadjusted model; the adjusted model was approaching statistical significance.

Our results add to the already published EPA data about this

Table 2
Distribution of elemental content, by sample.

Element	SL, ppm	Sample N	Percentile, ppm				Samples > SL, N (%)
			25	50	75	95	
As	26[a]	158	ND	16.9	62.2	89.7	67 (42.4)
Cr	12000[b]	157	ND	70.9	120.7	192.6	0 (0.0)
Cu	310	158	ND	51.0	77.8	218.0	2 (1.3)
Mn	180	158	243.1	374.0	590.9	1211.0	125 (79.1)
Pb	400[a]	158	96.1	255.7	575.0	878.8	56 (35.4)
Sr	4700	158	83.6	101.6	122.8	209.5	0 (0.0)
Ti	14000[c]	157	1205.0	1641.5	2063.2	3292.5	0 (0.0)
V	390	157	ND	38.4	87.4	128.2	0 (0.0)
Zn	2300	158	147.0	501.2	931.6	2219.5	5 (3.1)

Includes soil samples analyzed with X-ray fluorescence. a. Based on USS Lead Superfund Site screening levels. b. Screening level for chromium(III) c. Screening level for titanium tetrachloride. SL = screening level. SFS = superfund site; ND = not detected.

Table 3
Elemental content compared to screening levels, by site.

Element	SL, ppm	All sites (N = 28)		Sites not in SFS (N = 18)	
		Sites with at least one measure > SL, N (%)	Sites with median concentration > SL, N (%)	Sites with at least one measure > SL, N (%)	Sites with median concentration > SL, N (%)
As	26 [a]	15 (53.6)	10 (35.7)	6 (33.3)	4 (22.2)
Cr	12,000 [b]	0 (0.0)	0 (0.0)	0 (0.0)	0 (0.0)
Cu	310	1 (3.6)	0 (0.0)	0 (0.0)	0 (0.0)
Mn	180	26 (92.9)	22 (78.6)	15 (83.3)	14 (77.8)
Pb	400 [a]	10 (35.7)	7 (25.0)	3 (16.7)	1 (5.6)
Sr	4700	0 (0.0)	0 (0.0)	0 (0.0)	0 (0.0)
Ti	14,000 [c]	0 (0.0)	0 (0.0)	0 (0.0)	0 (0.0)
V	390	0 (0.0)	0 (0.0)	0 (0.0)	0 (0.0)
Zn	2300	2 (7.1)	0 (0.0)	0 (0.0)	0 (0.0)

Includes sites which had soil samples analyzed with X-ray fluorescence. a. Based on USS Lead Superfund Site screening levels. b. Screening level for chromium(III) c. Screening level for titanium tetrachloride. SL = screening level; SFS = Superfund Site.

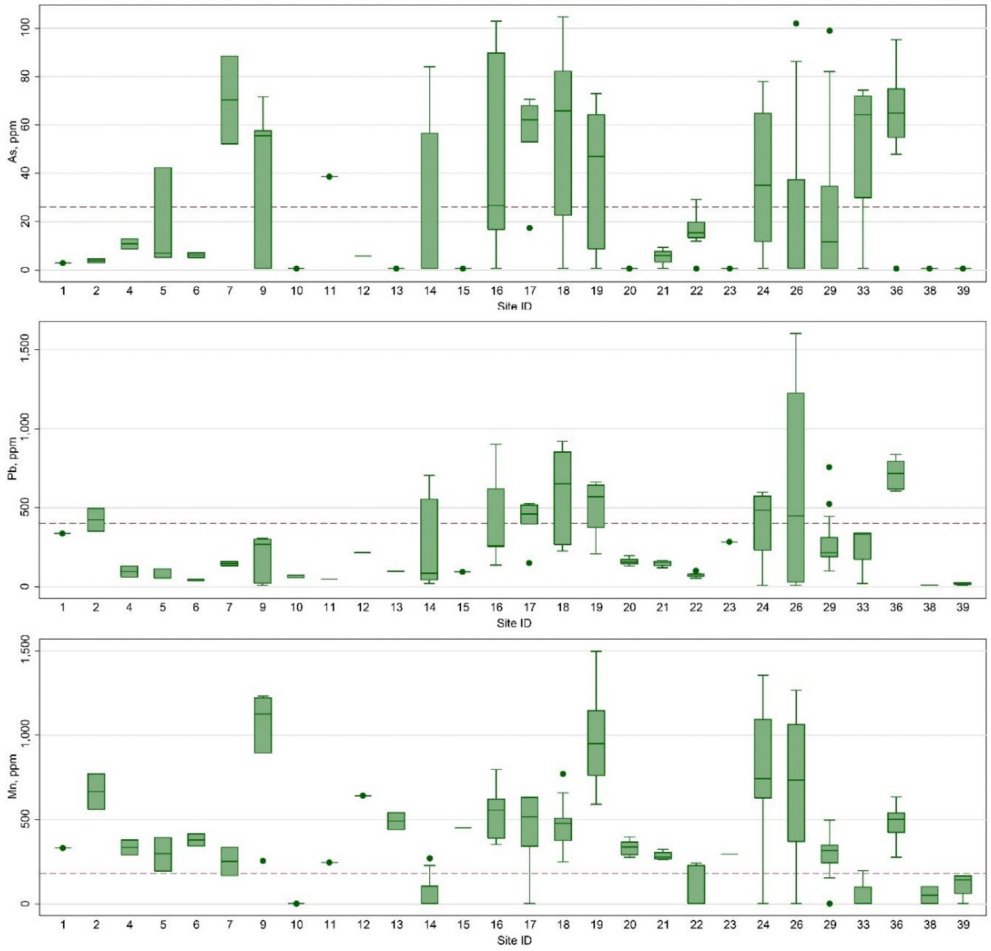

Fig. 2. Box plots of soil XRF concentrations, in ppm, by site, including N = 28 sites and n = 158 samples. Dashed line indicates soil screening level (SSL; As: 26 ppm; Pb: 400 ppm; Mn: 180 ppm).

Superfund site in its evaluation of a greater range of metals, and presentation of the high within-site variability. We also included some indoor dust samples (see Appendix A). Among the dust samples collected, 56.1% (As), 87.8% (Mn), and 75.5% (Pb) were above EPA screening levels. Notably, a higher proportion of dust samples were collected within the Superfund site compared to all samples; thus the higher percentages here are consistent with our soil analyses findings that locations within the Superfund site had higher metal concentrations among both dust and soil. Lead in indoor sources can originate from outdoor sources (e.g, contaminated soil) and/or indoor sources (e.g., lead-based paint). While our data suggest contaminated soil is of concern this analysis has insufficient data to evaluate potential indoor sources of lead or other metals.

Overall, our evidence is consistent with the EPA results, that soil Pb and As concentrations remain of concern, particularly in homes closer to the original smelter site (US EPA, 2017). An 2018 ATSDR health consultation corroborates our results for lead, as it found

Table 4
Odds ratios (95% confidence interval) for the association between elevated soil metal concentration with increasing proximity to former smelter site.

Model	As	Pb	Mn
Unadjusted	1.96 (1.18, 3.24)[a]	3.35 (1.64, 6.84)[a]	1.60 (0.89, 2.89)
Adjusted	2.61 (1.11, 6.16)[a]	7.09 (1.31, 38.28)[a]	2.26 (0.94, 5.41)

Includes soil samples analyzed with X-ray fluorescence. Based on generalized estimating equation (GEE) models with a binomial family and logit link. Models adjust for distance from nearest roadway, median age within block group, median income in block group, median percent white in block group and median year housing built (Pb model only). a. p-value<0.05.

that children with elevated blood lead levels were nearly three times more likely to be living in Zone 2 and Zone 3 compared to other areas of East Chicago (ATSDR, 2018). There were no residents in Zone 1 during our data collection because all Zone 1 residences were evacuated in 2016. This work adds to the EPA and ATSDR reports, as we identified a handful of samples with elevated Cu and Zn concentrations and we also found a high percentage of samples and/or sites had soil Mn concentrations above the EPA's regional screening levels. As noted earlier, there is a wide range of published soil screening levels for Mn, and we used a conservative estimate for this analysis; if we had used any other soil screening level the number of exceedances for Mn would have been greatly reduced. More data are needed to determine whether Mn poses a substantial health concern. That said, the presence of Mn in soil is not unexpected, as Mn is a component of steel and there are currently operating steel and metal recycling plants in the region. A recent paper, which sampled in the area just to the south of our site, also identified elevated Mn concentrations as a major concern (Dietrich et al., 2019).

Our data demonstrate a large variability in As, Pb, and Mn concentrations in soil and dust among samples taken at the same address. This is consistent with previous literature which noted substantial variations of Pb, Cd, Zn and As in soil (Arai et al., 2006; Laidlaw et al., 2018; Machemer and Hosick, 2004; Ullrich et al., 1999), as well as As and Cr in soil water (Hopp et al., 2006). In our data, among soil samples analyzed with XRF we observed sites where the range of soil concentrations at the same address ranged from below the limit of detection to 105 ppm (As), 1603 ppm (Pb), and 1354 ppm (Mn). In the USS Lead Superfund Site, homes were only designated for soil remediation if an elevated soil sample collected from that specific address. Residents have expressed concern that an insufficient number of samples have been taken from each address, which could lead to sites with contaminated soil not being remediated. Machemer and Hosick recommended that if highly detailed environmental assessment were not completed, soil remediation plans should include the entire area with suspected contamination (Machemer and Hosick, 2004). Our results also support this recommendation.

This study has a few limitations. Although XRF spectroscopy is a well validated approach to screening environmental samples for metal content (Hou et al., 2004; Weindorf et al., 2014), the use of multiple XRF instruments can result in some systematic measurement errors. We compared measurements from the two instruments used in this study in the Appendix (Table A8) and in a prior publication (Tighe et al., 2020b). While both instruments gave strongly correlated results, compared with NIST Standard Reference Materials, one instrument matched well for accuracy (correlation $R^2 > 0.95$) and there was ~10% systematic error on the second instrument which consistently read lower than the first. However, the instrument that performed best with respect to the NIST SRM was the instrument that had trouble distinguishing between As and Pb when both were present in high concentrations. This is because the K-alpha x-ray of As is isoenergetic with the L-alpha x-ray of Pb at 10.5 keV. This analysis error for one of our instruments could result in potentially ~30% underestimates of As in the presence of high lead concentrations. Taken together, it is likely that some of our measurements are underestimating some of these soil metal concentrations, but that does not significantly impact the conclusions drawn from these data.

Another potential source of error stems from the fact that participants were asked to provide their own samples for analysis and provided the metadata with each sample. It is possible that participant data collection could affect data accuracy (Whitelaw et al., 2003). However, other research (Hoyer et al., 2012; Oldekop et al., 2011), including our previous work which used collection methods similar to those described here (Beidinger-Burnett et al., 2019; Tighe et al., 2020a), has demonstrated that participant-collected data can be of high quality.

Other concerns are that participant collection of data could detrimentally impact the sampling design (Conrad and Hilchey, 2011) or reflect participant bias (Whitelaw et al., 2003). In this analysis, the sites with the highest number of replicates per site were also some of the more contaminated sites, suggesting that there was more concern on the part of participants who lived within the superfund zone compared to those who lived outside the zone. To address this, we present data indexed by site as well as by sample, and used methods for analyzing clustered data. Unlike many other studies, we did not observe any significant associations of soil contamination with demographic factors; however, in this population this is likely due to the fact that there was little variation among these demographic variables with our participants.

A major strength of this study was the study's engagement with affected community members. The inspiration for this work came from the community members who are understandably concerned about the impact of this mixed metal contamination on their own health. This concern is reflected in the large number of samples that were collected by community members. The engagement of the community members allowed us to collect a large number of samples from an area which the researchers would otherwise not have had access. We have also found that residents provide valuable insight and recommendations for key sampling locations and times as they are invariably more familiar with potential sources of pollution in the area than researchers who live in different communities.

In return, the community members benefit from learning about the potential for metal contamination within their own homes. This can lead to increased involvement in grassroots efforts to clean and prevent pollution, and/or individuals taking action on their own which could reduce their own exposures such as reducing the amount of soil/dust brought into the home from outside. Ultimately, this work is important because it has been demonstrated that elevated soil metal concentration contribute to metal exposures (Filippelli and Laidlaw, 2010; Laidlaw et al, 2016, 2018); however, reduction or remediation of soil contamination may be effective in reduceing exposure (Mielke et al., 2019). Future work is planned to understand whether this contamination is correlated with detrimental health impacts and identifying effective but cost-efficient methods to continue to reduce metal exposure for this community.

5. Conclusion

Results from this study largely supported prior work, in that we identified elevated As and Pb concentrations in soil and dust within the USS Lead Superfund site. In addition, our work also suggests that concentrations which exceed EPA recommendations may be occurring in areas just outside the Superfund Zone boundaries.

Additionally, Mn exposure may be a concern that has previously not been addressed significantly. Our results indicate that soil samples taken within a single address can be highly variable, which is critical when designating remediation targets. These results should be considered when making decisions regarding remediation of environmental contamination.

CRediT statement

Patrick Ringwald: Data Curation, Formal Analysis, Writing – Original Draft. **Cecelia Chapin**: Data Curation, Formal Analysis, Writing – Original Draft. **Christopher Iceman**: Conceptualization, Methodology, Investigation, Project Administration, Resources, Supervision, Writing – Review and Editing. **Meghanne E. Tighe:** Investigation. **Matthew Sisk**: Formal Analysis, Visualization. **Graham F. Peaslee**: Conceptualization, Investigation, Resources, Supervision, Writing – Review and Editing. **Julie Peller**: Conceptualization, Investigation, Methodology, Resources, Supervision, Writing – Review and Editing. **Ellen M Wells**: Conceptualization, Formal Analysis, Methodology, Supervision, Writing – Review and Editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2021.129915.

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