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ASSESSMENT OF SELECTED TRACE METALS IN SOILS OF A UNIVERSITY CAMPUS BY INDUCTIVELY COUPLED PLASMA-OPTICAL EMISSION SPECTROMETRY

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Abstract

Selected trace metals of cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), and vanadium (V) were determined in soils from five locations of a college campus by inductively couple plasma-optical emission spectrometry. Two locations were sites of buildings that were destroyed by fire, and the other three locations were selected due to high anthropogenic activity. According to the results of the study, the ranges of concentrations was as follows: Cd was below detection limits for all sites, Cr (7.49 – 24.7 mg/kg), Cu (9.15 – 22.5 mg/kg), Pb (2.96 – 22.4 mg/kg), and V (3.14 – 27.6 mg/kg). Soil samples associated with fires showed significantly higher levels of Pb and Cu than sites that did not have fires according to t-tests. The site by the campus lake showed the lowest concentrations of all metals tested, most likely due to runoff. All measured site concentrations did not surpass the Louisiana Department of Environmental Quality regulatory thresholds.

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INTRODUCTION

Urban and institutional soils serve as long-term reservoirs for trace metals introduced through both natural weathering processes and anthropogenic activity. Elements such as Cd, Cu, Pb, Cr, and V are of particular concern because they can accumulate in surface soils through vehicular emissions, building materials, landscape amendments, fuel combustion, and atmospheric deposition. Previous studies of urban soils have consistently shown elevated concentrations of Pb, Cd, Cu, and Cr in areas influenced by traffic density and human infrastructure, highlighting the value of localized soil monitoring for environmental risk assessment[1-4].

Although numerous investigations have characterized trace metal burdens in metropolitan and residential soils, comparatively fewer studies have examined college and university campuses as distinct micro-urban environments. Campus landscapes contain a combination of parking areas, roadways, pedestrian corridors, landscaped green spaces, and aging infrastructure that may create heterogeneous zones of metal accumulation like those reported in larger urban systems[5, 6]. Similarly, studies of urban soils have demonstrated strong land-use effects, with roadside and industrial areas often exhibiting greater metal enrichment and localized hotspots than parks and other less-disturbed land uses[3, 7-11].

College campuses provide useful settings to examine trace metal distribution in soils because of their diverse land uses, human activity, and long operational history[6]. Despite growing interests in urban soil chemistry, to our knowledge no published baseline dataset currently exists for trace metal concentrations on this campus or within similar academic environments in central Louisiana. Establishing such a dataset is novel because it allows comparison with broader urban background studies while also identifying localized anthropogenic signatures unique to campus activities.

In this study, soils collected acrossNorthwestern State University campus in Natchitoches, LA. were analyzed for Cd, Cu, Pb, Cr, and V using inductively coupled plasma–optical emission spectroscopy (ICP-OES). The objective was to determine concentrations of trace metals from sites that have had different uses in the university setting. This study also determined if the site where buildings were destroyed by fire had elevated levels of trace metals compared to other sites around the campus.

EXPERIMENTAL METHODS

Sample Collection

Five samples were collected at each location depicted in Figure 1 and 2 using polyvinyl chloride (PVC) pipes. Detailed information for each sampling site is presented in Table 1. The pipe was approximately 2 cm in diameter and pushed up to 15 cm into the ground and pulled out to collect a core sample. The top 1-6 cm of the core was discarded. The bottom 6-15 cm of the core samples were placed into a Ziploc bag for storage and transportation. The bags were labeled based on site location of the collection.



Figure 1. Sample collection sites A – E[12].

Sample Preparation

Approximately 2.000 g of sample was placed on a watch glass and dried at 105 °C for 48 h in a drying oven. Following drying, the samples were cooled to room temperature in a desiccator prior to further preparation and analysis. The samples were afterwards ground with a mortar and pestle. For digestion, EPA method 3050B was followed. In summary, five 1.000 g aliquots of each sample were placed in a 50 mL round bottom flask. Additionally, five replicates of approximately 1.000 g of EnviroMat SS-1 certified reference material (CRM) were analyzed as well as laboratory procedure blanks. Digestion was done by adding 10 mL 1:1 (v/v) nitric acid (trace metal grade)/ultrapure water solution to the 50 mL round bottom and a condenser was added to the flask. The sample and acid mixture was refluxed without boiling for 15 min. The samples were cooled and mixed with 5.0 mL of trace metal grade concentrated nitric acid and refluxed for an additional 30 min. In total, the samples were heated to approximately 95 °C for 2 hrs. without boiling then cooled. Afterward, 2.0 mL of 18 MΩ water and 3.0 mL of 30 % hydrogen peroxide were added. After the first 3.0 mL portion of hydrogen peroxide was added, 1.0 mL portions were added until bubbling ceased. The sample was again heated to approximately 95 °C without boiling for 2 hrs. The samples were then cooled and combined with 10.0 mL of concentrated hydrochloric acid and refluxed for an additional 15 min. Finally, the samples were cooled to room temperature and filtered through 0.45 μm pore sized filter paper and diluted to 100.0 mL with18 MΩ water.



Figure 2. Sampling site for USGS C-183832

Table 1 Description of Sample Collection Sites

Location	Site description	Latitude	Longitude	Altitude
A	Former site of Guardia Hall which burned in 1965	31° 45’ 52” N	95° 05’ 46” W	36 m
B	Former site of Caldwell Hall which burned in 1982	31° 45’ 04” N	95° 05’ 34” W	36m
C	Parade grounds next to the University Lab School	31° 44’ 58” N	95° 05’ 29” W	32 m
D	Area behind field house where tailgating takes place	31° 44’ 45” N	95° 05’ 46” W	36 m
E	Site next to Chaplin’s Lake	31° 44’ 35” N	95° 05’ 40” W	34 m
H	Site location of USGS C-183832 (farmland)	31° 48’ 02” N	95° 00’ 45” W	34 m

Instrumentation

Concentrations of selected trace metals were determined using ICP-OES (Thermo iCAP 6500 Duo, Thermofischer Scientific, Waltham, MA). The sample introduction system consisted of a peristaltic pump, nebulizer, and spray chamber. The instrument is capable of measuring emission intensities in both radial and axial view mode. The instrument was set to auto view mode which determines based upon the element of interest whether to view in axial or radial view. The instrument is equipped with a high-resolution Echelle polychromator and charge injection device (CID). The CID can measure wavelengths from 166 nm to 847 nm. The CID is mounted to a Peltier device and cooled to -40 °C for low dark current and noise. The wavelengths measured for each element and view mode are listed in Table 2.

Quality Control

Calibration curves for each element listed in Table 2 were generated using one calibration blank and three calibration standards with solution concentrations of 0.5, 1, and 5 ppm. For quality assurance, the 1 ppm standard was used as a check every after every ten samples. The limit of detection for all elements was ~0.1 ppm with a limit of quantitation at approximately 0.3 ppm. All metals were below the detection limits for the laboratory procedure blanks. The percent recovery of five replicates CRM (EvironMat SS-1) for each element is listed in Table 3.

Calculations

Concentrations (dry mass) of the samples were calculated from solution concentrations by the following equation:

$$C_d(\frac{mg}{kg}) = \frac{C_{ex}\,(\frac{mg}{L})\; x\; V_{soln.}(L)}{m_d(kg)} \qquad (1)$$

where C_d = dry mass concentration, C_{ex} = concentration of the extract, $V_{soln.}$ = total volume of the solution, and m_d = dry mass of the sample digested.

Statistical Analysis

To determine if there was a significant difference between metal content of soils that had buildings destroyed by fire (A and B) to those that did not (C and D), sites A and B measurements

Table 2. Selected Elements and corresponding wavelength (nm)

Elements	Wavelengths	View Mode
Cd	228.802	Axial
Cr	267.716	Radial
Cu	327.396	Radial
Pb	220.353	Axial
V	292.402	Radial

Table 3. Average concentrations (mg/kg) of selected trace metals (mg/kg), 95% confidence interval, and % recovery of the CRM.

Element	CRM Actual Concentration	95% conf. interval	Experimental Concentration	95% conf. interval	% recovery	RSD (%)
Cd	3.2	3.0 - 3.4	3.30	3.14 – 3.46	103	4.01
Cr	103	97.9 -108	119	100 – 130	116	13.0
Cu	403	393 - 413	426	395 - 457	106	6.00
Pb	764	749 - 779	613	576 - 650	80	5.00
V	27.2	26.1 – 28.3	33.6	29.8 - 37.4	123	9.13

were pooled, and C and D were also pooled forming two populations. The site close to the lake was excluded because of its uniqueness (next to water) compared to the other sites. F-tests for sample variances and appropriate t-tests for comparison of means were then performed for each element measured using the Analysis ToolPak add-in in Microsoft Excel (Version 2609, Microsoft Corporation).

RESULTS AND DISCUSSION

Dry mass concentrations from the five sampled sites are listed in Table 4. Cadmium was below detection limits for all sites. While four of the sampled sites give a broad range of concentrations for the rest of the metals tested, the site located next to the campus lake consistently showed the lowest concentrations of all the metals analyzed. This is possibly due to runoff into the lake. Sites A and B were the location of fires in the 1960s and 1980s respectively. Sites where fires took place were compared to two other sites (C and D) that had no fires, but still is a frequently used area of campus, using t-tests for each metal ($p < 0.05$). Significant differences were present for the elements of Cu and Pb, but no other elements showed a significant difference according to the t-tests. Pb and Cu were historically prevalent in building and plumbing materials, including lead and copper piping, lead-based solders, and copper-containing brass and bronze components, particularly in buildings constructed before the 1980s[13].

The Louisiana Department of Environmental Quality (LDEQ) Risk Evaluation/Corrective Action Program (RECAP) provides soil screening standards for a wide range of chemical constituents under both industrial and nonindustrial land-use scenarios. These regulatory benchmarks were developed to assess potential risks to human health and ecological systems associated with the environmental release of chemical contaminants[14]. Specifically, RECAP provides screening criteria for Cd, Cu, Pb, and V. Because all soil samples analyzed in this study were collected from non-industrialized locations, the corresponding nonindustrial screening standards were selected as the appropriate basis for comparison. These reference values are presented in Table 4 alongside the experimentally determined soil concentrations. Comparison of the measured concentrations with the applicable RECAP criteria demonstrated that all quantified elements were below the established screening thresholds.

The United States Geological Survey (USGS) maintains the National Geochemical Survey database, which contains soil and sediment geochemical data from locations across the United States[15]. Metal concentrations from geological samples collected in Natchitoches Parish are available within this database. For comparative purposes, sample C-183832, a soil sample located in close proximity to the sampling sites examined in this study, was selected as a regional reference. Concentrations determined by the USGS sample C-183832 are presented in Table 4. Sites A–D exhibited elevated Cr concentrations relative to the USGS reference sample, while Cu concentrations were higher at Sites A and B. In contrast, Sites C–E showed lower concentrations for all remaining quantified metals compared to sample C-183832. These localized enrichments in Cr and Cu may suggest potential anthropogenic contributions.

Table 4. Mean dry mass concentration (mg/kg) and 95 % confidence intervals of campus soils. Included is the results for USGC sample C-183832 and the LDEQ standards for non-industrial soils for comparison.

Element	A	B	C	D	E	USGS	LDEQ
Cd	BD	BD	BD	BD	BD	<2	39.00
Cr	19.6 ± 3.62	24.7 ± 6.6	20.8 ± 6.4	17.5 ± 3.4	7.49 ± 2.74	13	a
Cu	22.5 ± 4.6	14.0 ± 2.8	9.14 ± 0.97	10.36 ± 3.03	9.15 ± 4.42	10	3100
Pb	22.4 ± 3.7	19.3 ± 1.6	6.29 ± 1.13	11.1 ± 0.8	2.96 ± 0.65	23	400
V	18.9 ± 3.9	27.6 ± 0.9	19.6 ± 3.1	24.7 ± 3.9	3.14 ± 1.09	31	550

BD = below detection limits

a = No standard for total Cr concentration, but standards are available for Cr^{3+} and Cr^{6+}

The mobility of Cd, Cr, Cu, Pb, and V in soils is governed by their chemical speciation and interactions with soil constituents, including clay minerals, iron and manganese oxides, and organic matter. Cd is generally considered more mobile than the rest of the metals because it is relatively weakly retained by soil surfaces, particularly under acidic conditions, which can promote leaching into groundwater[16,17]. Therefore, it is not entirely surprising to not detect Cd in soils where fires have taken place 40-50 years prior. In contrast, Cu and Pb typically exhibit stronger sorption to soil organic matter and mineral surfaces, resulting in lower mobility under neutral to alkaline conditions; however, dissolved organic matter and changes in soil pH can alter their transport behavior[18]. Cr mobility is strongly dependent on oxidation state: Cr(III) generally exhibits low solubility and mobility because of its strong sorption and precipitation, whereas Cr(VI) occurs predominantly as soluble oxyanions that may be more mobile under oxidizing conditions[19]. V mobility is similarly controlled by speciation and redox conditions, with its behavior influenced by pH, organic matter, and interactions with iron oxides. In particular, oxidized V(V) species may remain relatively mobile under certain soil conditions, whereas reduction to V(IV) and sorption to soil constituents can promote retention[20, 21]. Consequently, the relative mobility of these elements cannot be established solely from their total concentrations, because differences in soil physicochemical properties and metal speciation can substantially influence their potential for leaching, biological uptake, and environmental transport.

CONCLUSION

ICP-OES has a proven track record as a tool to assess soil contamination by metals[16, 17]. Significant differences were discovered for the elements of Cu and Pb for sites that had fires compared to the sites that did not. However, all sampled sites met the LDEQ RECAP standards and do not pose any serious health risks. All elements apart from Cu for sites A and B, and Cr for all sampled sites, were comparable to the USGS sampled soil close to the location of the study. While the elevated concentrations of Cr and Cu do not pose serious health risks, their elevation may point to anthropogenic sources. More in-depth chemical and statistical analyses would need to be conducted to pinpoint the source of the elevated levels.

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