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Lead contamination of soils in an abandoned rifle range, Augusta County, Virginia

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Lead Contamination of Soils in an Abandoned Rifle Range, Augusta County, Virginia
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An Honors College Project Presented to

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by Logan Wren Mahoney

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FACULTY COMMITTEE: HONORS COLLEGE APPROVAL:

_____   _____
Project Advisor: L. Scott Eaton, Ph.D.   Bradley R. Newcomer, Ph.D.,
Professor, Department of Geology and Earth Science   Dean, Honors College

_____
Reader: Yonathan Admassu, Ph.D.
Assistant Professor, Department of Geology and
Earth Science

_____
Reader: Dhanuska Wijesinghe, Ph.D.
Research Scientist, Department of Geology and Earth
Science

PUBLIC PRESENTATION
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Acknowledgements

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Abstract

Presence of high concentrations of Lead within the soil and water could lead to various health related impacts on humans and wildlife. Exposure to Lead short termly or long termly could lead to many detrimental impacts due to Lead poisoning. The overall concentration of Lead in a soil can be measured using an Inductively Coupled Plasma Mass Spectrometry (ICPMS) under laboratory conditions. The present study investigates the Lead concentration of contaminated abandoned riffle range located in Augusta County, Virginia. Soil samples were obtained using soil augurs from three locations randomly based on the severity of contamination. Location three, which was located 50 ft away from the contaminated site, was considered as the control. Soil samples were analyzed for soil texture, color, wet sieve, dry sieve, pH, acidity and Lead concentrations. Initially samples were grinded and sieved through # 20 sieve. Standard methods were followed to analyze the samples. Soil texture was determined using wet sieve analysis while the particle sizes were determined based on the USCIS classification. Soil pH was determined using pH meter and the acidity was determined using a titration. The lead concentrations were determined using an ICPMS. The pH, wet sieve and dry sieve classification was used to correlate the Lead concentration of the site. Results indicated that the study site has been contaminated with soil mobile Lead. The mean concentrations of Lead for location one were 1327.1 ppm, which has exceeded the EPA standard limit. The Lead concentration of the location two was close to exceed the EPA standard limit. The texture of the soil in the study site was clay, which could potentially decrease the mobility of Lead and could lead in increase in the Lead concentrations in the soil due to existence of poor drainage conditions. Further investigations with more number of sampling locations and more replicates should be necessary

to confirm the findings of the present study. In the mean time it is necessary to adopt techniques to decrease the Lead concentrations below the EPA standard limit.

Introduction

Environmental exposure to lead can lead to various health related problems. Short-term exposure to lead results in various side effects due to lead poisoning (CDC, 2018). These symptoms include constipation, fatigue, loss of appetite, numbness, memory loss, irritability, and abdominal pain (CDC, 2018). Severity of these effects are greater in children than in adults, as short term exposure to lead can cause to cognitive disorder, including behavioral changes in children (CDC, 2018). In pregnant women, short-term exposure to lead can result in stillbirths and miscarriages (CDC, 2018). Infertility is also fairly common in both men and women who have short term exposer to lead (CDC, 2018). Additionally, long-term exposure to lead can result in serious side effects (CDC, 2018), which includes forgetfulness, depression, absent-mindedness, constipation, abdominal pain and nausea (CDC, 2018). Continued, prolonged exposure to lead leads to various diseases, such as heart and kidney diseases, high blood pressure, and reduced fertility (CDC, 2018). Lead exposure is also linked to an increase in cancer development (CDC, 2018).

Accumulation of lead in the environment could negatively impact the stability of an ecosystem. Though plant-absorbed lead does not transfer into animals and humans through consumption, it could negatively impact plant growth (Rampado, 2017). Lead accumulation in plants lead to growth inhibition by interfering with cell division and water absorption (Rampado, 2017). Subsequently, this will reduce of the plant's rate of photosynthesis, which ultimately results in stunted plant growth and even death (Rampado, 2017). This creates an unbalance environment, where the food chain is broken as herbivores are unable to consume the plants as they continue to die (Rampado, 2017). Accidental lead poisoning is fairly common within areas

of contamination, especially seen in livestock from infected farms (Rampado, 2017). The ability of animals to perform various activities is heavily impaired by the ingestion of large amounts of lead (Rampado, 2017). The selected site for this study is an old rifle range that was converted to a nature center in Camp Shenandoah in Swoope, Virginia. The rifle range was present in the study site from 1953-2005, when a new rifle range was approved to build up the ridge from the current location (Eaton, 2019). The rifle range became the new nature shelter in 2005 and started operation in 2006 (Eaton, 2019). The nature shelter has been used to conduct classes since then. Water for the camp is obtained through a stream (Cabin Run), which flows through this camp and past the old rifle range that is part of the study area (Eaton, 2019). Since Lead is insoluble in water, the water would not generally be affected by the amount of lead present in the soil via leaching (Mohammadzadeh, 2015). Discoloration of the plant leaves in the vicinity of the rifle range and the bridge in the study site was the first evidence of the lead contamination in the study site (Eaton, 2019). Upon further investigation, the bullet fragments were found in the soil, being broken down into the ground (Eaton, 2019).

The lead concentration of a soil sample can be determined using several methods. The most common method that has been used is using an ICPMS to measure the overall lead content (Blaustein, 2017). Use of chelating agents, such as EDTA, have proven ineffective, as they do help raise the solubility of the soil and extraction through contaminated plants, The overall expense of using the EDTA would prove ineffective in creating an appropriate agent of removal (Blaustein, 2017).

The overall goal of this study is to determine that the amount of lead present in the soil and how it might affect the wildlife and human activity in the area.

Methodology

Figure 1: Map of the study area. Study area is colored in light blue, stream affected is in dark blue, and the red dot is present on the county map to show where in Virginia the camp is (Boy Scouts, 2004)

Figure 2a: Image of the Study Area
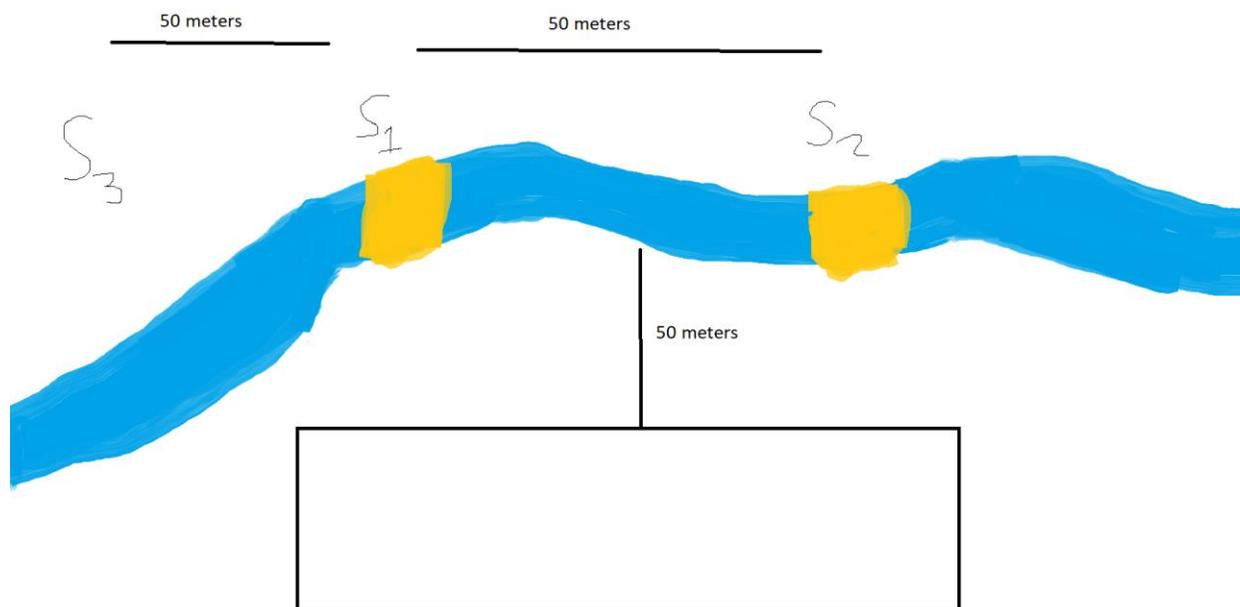
Figure 2b: Sketch of study site. Diagram is not to scale, with appropriate distances measured throughout.

Study Site and Characteristics: The study was conducted from September 2018 to March 2019 to determine the lead contamination in an abandoned rifle field located in Swope, Virginia (Figure 1). The site receives an average of 44 inches of rainfall throughout the year. The dominant soil series of the site is Weikert.

Methods

Soil Sampling and Analysis: Soil samples were collected from three locations randomly based on the severity of contamination (Figure 2a and 2b). Location three was used as the control, since it is out of the area that is considered as the traditional rifle range. Soil augurs were

used to obtain the samples and the auguring was performed until the augur hits the bedrock. Collected samples were put in the sampling bags, sealed and transported to the lab for further analysis. Soil samples were disaggregated and sieve through a #20 sieve (2mm) prior to analysis. Several tests were performed at the laboratory to determine the soil texture (feel method, wet sieve analysis and USCS classification), soil color (Munsell Color Chart), soil pH and Soil acidity. Finally the soil samples were run through an Inductively Coupled Plasma Mass Spectrometry to determine the lead concentrations in ppm.

Field Texture and Color: Field soil texture was determined by following the procedure suggested by Thein (1979). A flow diagram for teaching texture by feel method (modified from Thein, 1979) was used to determine the approximate sand, silt and the clay percentages and the textural class. Munsell color charts were used to determine the soil color. The color chart describes colors by three different properties, the hue, the value, and the chroma (Thein, 1979). The hue is defined as the pure spectrum color that the color shows whereas the value indicates the lightness or darkness of the color (Thein, 1979). The chroma describes the intensity of the color (Thein, 1979).

USCS Classification: The test was performed using the USCS classification method (ASTM D2487) followed by the shaking of samples through a series of recommended sieve sizes approximately for nine minutes (Practice, 1985). The sieves used are in accordance to the size, starting with the #4, #10, #20, #40, #60,#100, #200, and the pan (Practice, 1985). Any soil that was previously sieved was re-added into this sieving process. Finally the total measurements were weighed to quantify percent fines, percent sands, and the percent gravel, which were then used to classify the soil from the USCS classification system (Practice, 1985). For percent

passing, the sum of the soil weight is obtained. From there, the weight of the soil present in the sieve is divided by the total weight of the soil, and that number subtracted by 100 percent to get the percent passing. The total sum is from any passed before also needs to be added into the percent passing as well. For example, to calculate the percent passing of the #10 sieve, the weight of both the #4 and the #10 sieve need to be added. A Coefficient of Unconformity value, which reflects the degree of sorting, is calculated by using the equation below:

$$Cu = \frac{d_{60}}{d_{10}}$$

The coefficient is used to classify dry soil through the USCS classification.

Soil pH: Soil pH was determined by using a pH meter (Extech EC400), following the methods adopted by Smalls (1955). Initially 20 g of soil was weighed into a 100 ml beaker and then 20 ml of deionized water was added to the sample. The sample was then mixed using a centrifuge at a low setting for 30 minutes. Next the sample was allowed to settle for 60 minutes before the pH reading was taken. It is important to let the buffers and the sample return to room temperature before the sample is tested (Smalls, 1955). Finally the pH of the sample was taken using a pH meter. The pH meter was calibrated prior to obtain the reading using calibration solutions, 4, 7 and 10.

Soil Acidity: Acidity of the soil sample was determined using the methods adopted by Dohrmann (2006). Three major steps were involved in the soil acidity determination: extraction, forward titration, and backward titration. During the extraction step, 10 g of soil sample was saturated with 100 ml of 1M potassium chloride solution and kept it for an overnight. On the following day the saturated sample was filtered through 11 micron filter paper. Ten milliliters

from the filtrate was then transferred to an Erlenmeyer flask and initiated the forward titration.

Five drops of 0.1% phenolphthalein indicator was added to the filtrate prior to the forward

titration. Next, the sample was titrated with 0.025 M sodium hydroxide solution. At the end point

(when the solution turns pink), the volume of sodium hydroxide used was recorded. Then the

sample was prepared for the backward titration by adding few drops of 0.025 M hydrochloric

acid (until the sample becomes colorless). Next 5 ml of 4% sodium fluoride was added to the

sample (the sample turns pink if exchangeable aluminum present in the sample). Then the

sample was backward titrated with 0.025 M hydrochloric acid (until the solution becomes

colorless) and the volume of hydrochloric acid was recorded. The total exchangeable acidity was

calculated using the volume of sodium hydroxide used for the forward titration. The volume of

hydrochloric acid used for the backward titration was used to calculate the exchangeable

aluminum, while the difference between the total exchangeable acidity and exchangeable

aluminum yielded the exchangeable hydrogen. Total exchangeable acidity is measured through

the equation below (Dohrmann, 2006):

$$N \ NaOH \ \left(\frac{mol_c}{L}\right) * \frac{1L}{1000 \ mL} * \frac{vol \ NaOH \ (mL)}{mass \ soil \ (g)} * \frac{1000 \ g}{1 \ kg} * \frac{100 \ cmol_c}{1 mol_c}$$

From this equation, exchangeable aluminum is able to be calculated from this equation below

(Dohrmann, 2006):

$$N \ HCl \ \left(\frac{mmol_c}{L}\right) * \frac{1L}{1000 \ mL} * \frac{vol. \ HCL \ (mL)}{mass \ soil \ (g)} * \frac{1000 \ g}{1 \ kg} * \frac{100 \ cmol_c}{1 \ mol_c}$$

From that equation and the one before, exchangeable hydrogen can also be obtained from the

equation below (Dohrmann, 2006):

$$Exchangeable\ H^+ = Exchangeable\ acidity - Exchangeable\ Al^{3+}$$

Wet Sieve Analysis: Forty grams of soil was weighed and transferred to a container, and filled with 350 ml of deionized water. Then 10 ml of sodium hexametaphosphate solution was added to the sample and mixed well using a hand blender for four minutes. Next, the sample was poured into a set of sieves, which was consisted of #60 (to collect coarse to medium sand), #230 (to collect fine to very fine sand) sieves and a pan to catch the silt/clay fraction. Then the sand fraction was transferred to a pre-weighted 100 ml beaker and dried for 24 hours at 200 ^{0}C to calculate the percentage of sand. The remaining solution retained at the pan was transferred to a 1,000 ml graduated cylinder and diluted with deionized water up to the 1,000 ml mark. Then the sample was mixed thoroughly using a metal rod for two minutes. As soon as the mixing was completed, a hydrometer was inserted into the sample and a reading was recorded at exactly 44 seconds. This reading was used to calculate the percentage of silt and clay. Finally the sample was allowed to settle and another hydrometer reading was taken at 2 hours to calculate the percentage of clay. Wet sieve data is measured based on the hydrometer reading and the weighing the overall amounts of sand that was dried from measuring the sample. Samples are concluded to be valid if less than 10% of the soil is lost through the process.

Lead concentration: The concentration of lead of the soil samples was determined using an Inductively Coupled Plasma Mass Spectrometer (ICPMS) (X series I). Fifty grams of each sample was mixed with 50 mL of extracting solution of EDTA and kept for 24 hours (Manouchehri, 2006). In order to assure the maximum extraction of lead from the samples, the concentration of the EDTA was set to 0.05 M. The 0.05 M EDTA solution was prepared by mixing 14.612 grams of EDTA in 1 L of deionized water. The samples were then run through a

centrifuge at a speed of 4000 rpm for 90 minutes, with three breaks in between of the spinning.

These samples were then extracted and filtered through a 0.22 micron filter (Manouchehri,

2006). Filtration and the running through the centrifuge were to reduce the presence of large

particles, which could clog the machine during the initial run.. The samples were then loaded

into the ICPMS, with three different lead standards (5, 10 and 20 ppb). . The machine was started

2 hours before the beginning of the run to allow the plasma to warm up. Next, the samples were

run through the machine and were evaluated to see whether further dilution was needed or not.

Procedures for how to run the ICPMS are not explained due to the samples being sent away for

testing in another department. Lead concentrations above were calculated by removing the factor

of how much dilution occurred at first. Each sample was diluted at a different factor, with sample

site 1 was diluted at a factor 1 to 2000, sample site 2 was diluted at a factor of 1 to 1000, and

sample site 3 was diluted at a factor of 1 to 100. These concentrations were then placed into a

factor of being measured in one kilogram weight, similar to how the EPA measures their

samples. The initial measurements were taken at 50 grams of soil, so each sample was multiplied

by 20. These samples were then converted into ppm from ppb, which resulted in the final

concentration of lead in the samples. An average of the samples were taken, along with the

standard deviation between each sample.

Results

Based on the field soil texture analysis, most of the horizons (A and B) fall within the clay

textural class (Table 1). However, the lowest horizon has a silty clay texture (C).

Table 1: Table of Field Texture and Color of the Sample

Layer	Color	Texture
A	10 YR 4/3	Clay Loam
B1	10 YR 5/8	Clay Loam
B2	10 YR 5/4	Clay Loam
Bc	10 YR 5/4	Clay
C	10 YR 5/3	Silty Clay

Findings of the dry sieve analysis suggested that the soil had a low coefficient of Uniformity

(CU) resulting in a poorly graded soil (Table 2). The calculated percent passing concluded that

the soil belongs to the sand category based on the USCS classification (Table 2). Overall, the

data concluded that the soil was to be poorly graded sand.

Table 2: Table of dry sieve data

	4.76 mm	2 mm	0.84 mm	0.42 mm	0.25 mm	0.177 mm	0.149 mm	0.074 mm	0 mm	CU
Weight (g)	741.6	797.39	912.84	807.21	539.37	584.24	449.79	371.4	456.9	1.4
Sieve Weight (g)	499.5	409.18	415.81	354.3	318.51	332.45	351.31	311.71	448.36	
Weight of Soil (g)	242.0	388.21	497.03	452.91	220.86	251.79	98.48	59.69	8.54	
% Passing	89.09	71.60	49.21	28.80	18.85	7.51	3.07	0.38	0	

The soil pH was fluctuated with in the neutral pH range (mean = 7.16) (Table 3). Moreover, the calculated total exchangeable acidity was 0.0 Cmolc/kg of soil. Surprisingly, no exchangeable aluminum was detected from the soil acidity test (Table 3).

Table 3: Table of Wet sieve data, pH data, and Acidity data

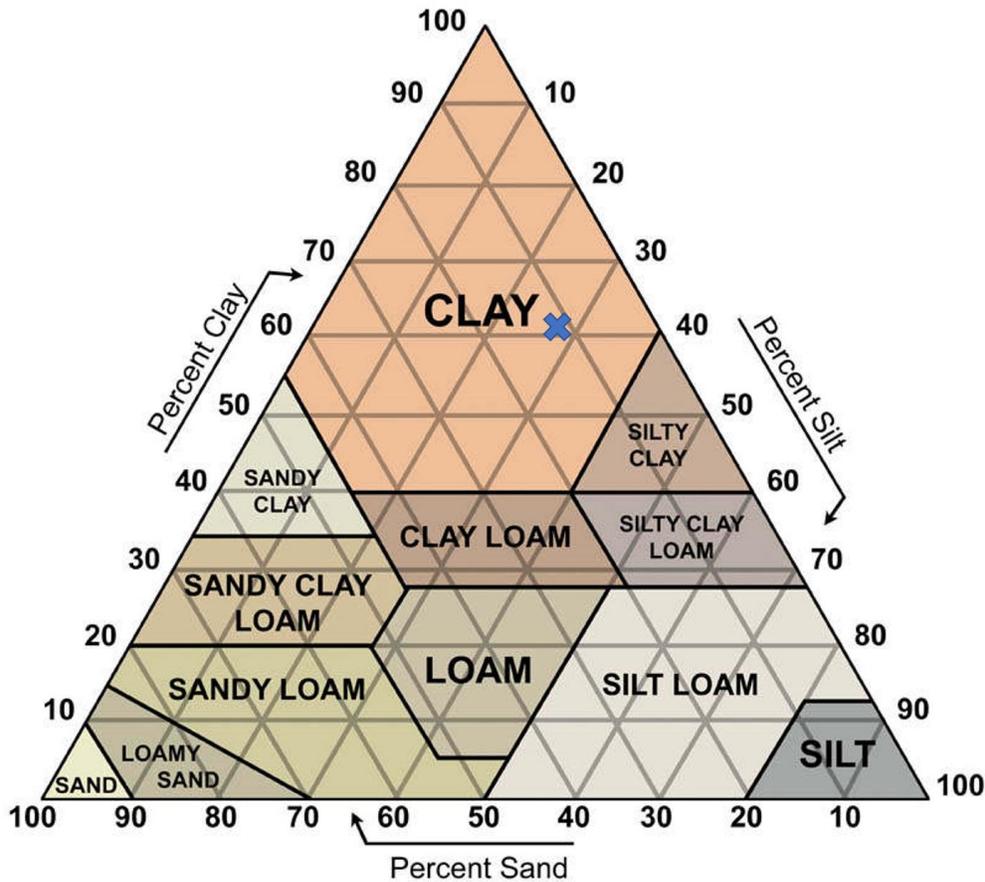|		Weikert Series
Wet Sieve Analysis	Sand (g)	3.92
	Silt (g)	10.5
	Clay (g)	22.5
Soil pH	pH test 1	7.16
	pH test 2	7.15
	pH test 3	7.17
Soil Acidity	Aluminum (cmolc/L)	0.0
	Hydrogen (cmolc/L)	0.0
Total Exchangeable Acidity	Aluminum + Hydrogen (cmolc/L)	0.0

Figure 3: Ternary diagram showing the classification of the samples.

Based on the wet sieve analysis the soil belongs to clay textural class (Figure 3).

The tables 4 and 5 illustrate the findings from the Lead analysis. The concentrations shown in table 4 were used to show the need for dilution. The initial standards that were used included 4, 9, and 20 ppb. Based on the findings from the first run (Table 4) it was evident that the initial standards were not accurate for a valid comparison since the average values were way above the standards. Thus, it was concluded that another set of standards were needed for the second run to make a valid comparison.

Table 4: Table of undiluted samples for lead testing

	Concentration (in µg/L)	Average (in µg/L)
S11	43204.0	
S12	44867.3	43764.9
S13	43223.3	
S21	11477.0	
S22	8949.0	9795.2
S23	8959.7	
S31	741.7	
S32	756.0	762.9
S33	791.0	

The fixed concentrations were then used to measure the samples within appropriate limits more accurately. The dilutions followed for the sample site one was 1:2,000 while 1: 1,000 for the for sample site 2. A dilution of 1:100 was followed for sample site three (Table 5). The findings concluded that the average concentrations for sample sites one and two was 1327.1 ppm per kg and 283.3 ppm per kg respectively. In contrast the sample site three recorded the lowest Lead

concentration (21.6 ppm). The calculated standard deviation values were low for all three-sample

sites (Table 5). .

Table 5: Table of diluted samples for lead testing

	Concentration from Dilution (in µg/L)	Concentration (in µg/L)	Concentration (in µg/L per Kg)	Concentration (in ppm per Kg)	Average Concertation (in ppm per Kg)	Standard Deviation (in ppm per Kg)
S11	32.5	65066.7	1301333.3	1301.3		
S12	34.6	69200.0	1384000.0	1384.0	1327.1	49.3
S13	32.4	64800.0	1296000.0	1296.0		
S21	17.1	17066.7	341333.3	341.3		
S22	13.8	13800.0	276000.0	276.0	283.3	54.7
S23	11.6	11633.3	232666.7	232.7		
S31	11.3	1130.0	22600.0	22.6		
S32	10.4	1040.0	20800.0	20.8	21.6	0.91
S33	10.8	1076.7	21533.3	21.5		

Discussion

Field textures and colors help to explain the overall nuances of the soil types that are present. Presence of high amount of clay and a Munsell color of 10 YR showed poor drainage conditions exist within the study site (Table 1). Poor drainage conditions leads to an environment that can build lead up, as the water that would make the lead mobile would be stuck in the layer above. The pH showed a fairly neutral soil sample, just slightly higher than a pH of 7. Natural pH conditions exist in the site indicate an oddity in the presence of lead in the soil. Lead's solubility in the soil increases with a lower pH, leading to higher concentrations of lead present in the soil. This information also lines up with the acidity, as the soil contained no available hydrogen or aluminum for cation exchange. For aluminum and hydrogen to be available for cation exchange in the soil, the soil pH would need to be significantly more acidic, ranging around between four and five (Smalls, 1955). The Dry sieve and Wet sieve tests were used to classify the soil from an environmental standpoint and from an engineering standpoint. The dry sieve classification is classified based on the percentage of gravel, sand, and fines along with the classified CU value. This soil has high amounts of sand with low amounts of gravel and fines, along with a very low CU value (Cu < 4). As a result, this soil would be classified as a poorly graded sand. For the wet sieve test, The values of the sample are plotted on a ternary diagram, for which it can be used to explain the type of soil that we have from an environmental standpoint. Plotting the data on the ternary diagram shows that the soil would be classified as a clay. This also lines up well with the field texture determination, as the field textures show similar values overall. The dry and wet sieve test have different classifications that are mutually exclusive. The cause is likely from inaccurate equipment to properly use the dry sieve test, resulting in much of the finer grains being unable to fall off into the pan. It is suspected that lead

findings should overall increase with the presence of clay versus gravel or sands, as a clay environment would lead to poor drainage resulting in the lead having restricted mobility. The findings of the present study suggested that the study site had significantly high concentrations of lead. The lead concentrations of the sampling site one had exceeded the EPA standards, being 400 ppm for a play site and 1200 ppm for a non-play site, for non-play sites by over 121 ppm. Similarly, sample site two showed lead concentrations that were close to exceed beyond the EPA standard limit for a play site by about 117 ppm. The effectiveness of the EDTA extraction was approximately 40%, which indicate that the EDTA is able to extract approximately 40% of the lead from the sample. This highlighted that the more than half of the lead is still presence in the sample in non-extractable form. (Manouchehri 2006). Assuming full extraction of lead (100%) from the soil sample, the lead concentration of the sampling site one should be significantly greater than the EPA contamination of a non-play limit, with lead values of 3300 ppm present throughout the site. Similarly, the approximate lead concentration of the sample site two should falls within the 700 ppm, indicating that the site two has being contaminated on the EPA contamination for play sites too (EPA, 2005). Presence of heavy concentrations of Lead in the study site could lead to many detrimental impacts on short term as well as on long-term basis. Since the site has been used as a nature shelter for education purposes for children, there is a greater possibility of exposing these children for lead.

An oddity that appears throughout this is the presence of lead in the study site with a neutral pH conditions. Lead is mobile under low pH conditions. Thus, the existence of neutral pH levels could suppresses the mobility of lead within the soil (Smalls, 1955). Two possible theories can be stated as to why the lead is present within the soil. The first explanation is around the idea that the soil is that the mobility of the lead decreased when the pH began to rise,

resulting in lead presence in the area. This idea matches up with the fact that the pH of the site has steadily been increasing throughout the years as a result from acid rain being reduced. This idea is not supported by the absence of a solid layer of lead. The other explanation revolves around the lead replacing the cations in the sample. This idea matches up with the low acidity as the cations would not exist had they been replaced throughout. This is largely unlikely as well as lead is significantly larger than most cations, resulting in it not being substituted correctly without breaking the clays.

Conclusion

The findings of the present study suggested that the site is heavily contaminated with soil mobile lead. Supplementary data also showed that the presence of lead in the field is an oddity, as the pH and acidity should be significantly higher. Moreover, existence of clay textured soils have restricted the mobility of lead in the surface horizons of the study site which could also lead to lead accumulation in the site

Suggestions and Recommendations

Lead mitigation is required in order to remove the lead from the site (EPA, 2005). Currently, the site is being restricted by covering all access to the contaminated bare soil (EPA, 2005). This method proves effective until it crosses through the threshold of 1200 ppm per kilogram of soil. There are two other methods (EPA, 2005) available to decrease the Lead concentrations below the EPA standard. The first method is to remove the soil completely up to 24 inches and replaced with clean soil. The second method is the paving off of the soil (EPA, 2005). Based on the current site conditions it is better to use the first method since the area could be disturbed by wildlife if paved. Further studies that could be explored revolve on the migration of the lead, which is predicted downstream based on flow of the water, and how plant mass is helping to remediate the stream, such as sunflowers.

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