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Arsenic and Lead Leaching from the Waste Derived Fertilizer Ironite

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The toxicity characteristic leaching procedure (TCLP) and the synthetic precipitation leaching procedure (SPLP) were performed on commercially purchased samples of the waste-derived soil amendment marketed as Ironite. Ten samples of the 1–0–0 grade (the most widely available in Florida) were tested. Two samples of the 12–10–10 grade and three samples of the 6–2–1 grade (a liquid version) were tested as well. TCLP leachate concentrations from the 1–0–0 grade samples ranged from 5.0 to 8.0 mg L^{-1} for lead and 2.2 to 4.8 mg L^{-1} for arsenic. SPLP concentrations from the 1–0–0 samples ranged from 0.62 to 3.1 mg L^{-1} for lead and 1.9 to 8.2 mg L^{-1} for arsenic. All of the 1–0–0 grade samples exceeded the U.S. hazardous waste toxicity characteristic (TC) limit for lead (5 mg L^{-1}), while five of the 10 SPLP samples exceeded the TC limit for arsenic (5 mg L^{-1}). The greater arsenic leachability in the SPLP relative to the TCLP was determined to be a result of lower pH conditions in the SPLP. A composite sample of the 1–0–0 grade was found to leach much greater concentrations of both arsenic and lead using California's waste extraction test (WET). Lead leachate concentrations were much lower in the two 12–10–10 samples (0.03 mg L^{-1} or less); arsenic concentrations in these leachates (both TCLP and SPLP) exceeded 5 mg L^{-1}. None of the 6–2–1 samples contained lead or arsenic above TC limits. An experiment performed on the 1–0–0 grade which examined leachability as a function of pH found that at pH values in the range of what is encountered in the human digestive system (pH 4.0 to 1.5) lead leached 2–36% of its initial content, and arsenic leached 1–6% of its initial content. A simple gastric acid leaching experiment found 83 and 37% of the lead and arsenic present to leach, respectively.

Introduction

Ironite is a commercially sold soil amendment available in many home-consumer garden supply outlets. The manufacturer (Ironite Products Company, Scottsdale, AZ) describes Ironite as a soil supplement and fertilizer that promotes healthy plant color and root growth through the slow release of elements such as iron and zinc and the presence of a small amount of nitrogen (http://www.Ironite.com). Reported uses include use in vegetable crops, lawns, golf courses, and home gardens. Ironite is available in several forms, most commonly as a solid granule (marketed formulations include 1–0–0, 12–10–10) but also in liquid form (6–2–1). The source of Ironite is mining waste from a former silver mine in Arizona

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(1). The mining waste used to produce Ironite is presently exempt from hazardous waste regulation under the Bevill exemption of the Resource Conservation and Recovery Act (RCRA) [Section 3001(b)(3)(A)(ii)] (2).

While the minerals in the mining waste provide the source of the product's efficacy as a soil amendment, the minerals also contain several trace elements that may pose a concern to human health and the environment, most notably arsenic and lead. The presence of these trace metals has led some state governments to investigate potential safety and environmental concerns associated with Ironite. In a review of trace metals in fertilizers, the Washington State Department of Agriculture found the 1–0–0 grade of Ironite to contain 4380 mg kg^{-1} of arsenic and 2910 mg kg^{-1} of lead (3). Testing of Ironite by the Minnesota Department of Health found arsenic in the 1–0–0 grade to contain from 3540 to 6020 mg kg^{-1} of arsenic and 2800 to 3400 mg kg^{-1} of lead (4, 5). A U.S. Environmental Protection Agency (EPA) summary of fertilizer use, contaminant content, and regulations reported Ironite lead and arsenic concentrations of 3290 and 4510 mg kg^{-1}, respectively (6). In a recent federal register notice (2) regarding hazardous waste used to manufacture fertilizer products, the U.S. EPA stated that while Ironite is currently exempt from the definition of hazardous waste under RCRA, concerns over arsenic are worthy of serious consideration. Several states (e.g., Minnesota and Maine) now limit the sale of this product.

While the concentration of lead and arsenic may be large, especially as compared to standards or guidelines for direct human exposure to soils, the mineral forms of the elements must also be considered. A technical report cited by the manufacturer indicates that lead and arsenic are present as the minerals arsenopyrite and galena and that the elements in these forms are "very stable in the environment and not available in a form which is toxic". While the total concentration of trace elements in Ironite has been reported (3–6), data on the leachable concentrations are not widely available. This paper reports the results of leaching tests conducted on samples of Ironite purchased at retail outlets in Florida. The most common form for the product available was the 1–0–0 grade, and the majority of tests were performed on these samples. Several samples of the 12–10–10 and the 6–2–1 grades were also purchased and analyzed. The TCLP and SPLP, as well as a measurement of total lead and arsenic concentration, were performed on every sample. Several other experiments were performed on a composite 1–0–0 sample, including California's WET, an experiment evaluating leaching as a function of pH, and a simple gastric acid leach test. The objective of the research was to provide leachability data that could be used in future decision-making regarding the regulatory status and appropriate management of this product.

Method and Materials

Sample Collection and Preparation. Ten bags of grade 1–0–0 Ironite (samples A1–A10), two bags of grade 12–10–10 Ironite (samples B1 and B2), and three spray bottles of grade 6–2–1 Ironite (samples SB1–SB3) were purchased from 10 retail outlets in seven Florida cities. The limited number of 12–10–10 samples tested was a result of lack of availability in the outlets visited. A composite sample of the 1–0–0 grade was prepared in the laboratory by adding 200 g from each of the 1–0–0 bags to an acid-rinsed stainless steel bowl and mixing. The composite 1–0–0 sample was found to be uniformly distributed in particle size (D_{60}/D_{10} = 1.8) with a D_{50} of 1.75 mm.

Analysis for Total Heavy Metals. The total arsenic and lead concentrations (mg kg^{-1}) in the granular Ironite samples were measured by digesting 2 g of undried sample (original moisture content = 2.5% by wet weight) with nitric acid, hydrochloric acid, and hydrogen peroxide following EPA Method 3050B (7) for subsequent analysis using inductively coupled plasma-atomic emission spectroscopy (ICP-AES). For some matrixes, the Method 3050B digestion procedure does not liberate 100% of the elements of concern, and results from the procedure are often described as total recoverable concentrations. For the Ironite samples tested, the EPA 3050B procedure was observed to completely dissolve the solid matrix. Arsenic and lead concentrations in the liquid samples were measured using ICP-AES following EPA Method 6010B, after digesting 100 mL of the liquid fertilizer with nitric and hydrochloric acid using an open vessel digestion method following EPA Method 3010B (7).

Laboratory Leaching Procedures. All 12 of the granular Ironite samples were leached using the TCLP (EPA Method 1311, ref 7) and the SPLP (EPA Method 1312, ref 7). The TCLP utilizes a buffered acetate solution as an extraction fluid and is used (among other things) to determine whether a solid waste is a toxicity characteristic (TC) hazardous waste under U.S. hazardous waste regulations. The TCLP extraction fluid was prepared by adding 11.4 mL of glacial acetic acid (CH$_3$-COOH) to 1 L of deionized water in a 2 L volumetric flask. Then 128.6 mL of 1 N sodium hydroxide (NaOH) was added to the flask, and the solution was brought to volume (2 L) with deionized water. The resulting solution pH was 4.93 ± 0.05. One hundred grams of the sample was placed in a 2.2 L polyethylene extraction vessel, and 2 L of the extraction solution was added. The slurry was mixed on a rotary extractor at 30 ± 2 rpm for 18 ± 2 h and then filtered through a 0.7 μm glass fiber filter. The filtrate was collected in 1 L plastic bottles and preserved by adjusting to a pH of less than 2 using nitric acid (7).

The SPLP was carried out in a similar manner as the TCLP but using a different extraction solution. The SPLP extraction solution (pH of 4.20 ± 0.05) was designed to represent acidic rainwater; this procedure is often used to assess the leachability of contaminated soils or wastes disposed or reused in the environment (8). The extraction solution was prepared by adding a dilute sulfuric acid and nitric acid solution (60:40 mix) to a 19 L container and diluting to volume (20 L) with reagent water. The TCLP and the SPLP were also carried out on the composite 1−0−0 sample in triplicate.

The WET, performed on the composite 1−0−0 sample, is a leaching procedure used in California to determine whether a solid waste is a TC hazardous waste. The WET extraction solution is a buffered citric acid solution. The WET leaching fluid was prepared by titrating a 0.2 M citric acid (C$_6$H$_8$O$_7$) solution with 4.0 N NaOH to a pH of 5.0 ± 0.1 (9). One liter of this fluid was added to a 100 g sample in triplicate and rotated for a period of 48 h. The WET performed in this research was modified in that deoxygenation of the extraction solution (by bubbling nitrogen gas) was not performed.

The impact of leaching solution pH was examined by leaching subsamples of the composite 1−0−0 sample over a broad range of pH values. The method was similar to that used for the TCLP and SPLP, but instead of the prescribed leaching fluids, a solution of a desired pH was used. Target pH values ranging from 1 to 13 were created using nitric acid (HNO$_3$) or sodium hydroxide (NaOH) in deionized water. A liquid-to-solid ratio of 20:1 was targeted (as prescribed for TCLP and SPLP), with 180 mL of leaching solution initially added to 10 g of sample. After rotating for 30 min, the pH was measured and adjusted as necessary. This process was repeated for several hours, after which the final volume of the extraction solution was brought to 200 mL, and the samples were rotated for 18 additional hours. While an effort

TABLE 1. Concentrations of Arsenic and Lead in from 15 Samples of Ironite Purchased in Florida

sample ID	grade	total arsenic (mg kg^{-1})	total lead (mg kg^{-1})
A1	1−0−0	3450 ± 29[a]	2700 ± 28
A2	1−0-0	3380 ± 75	2690 ± 87
A3	1−0−0	3540 ± 22	2710 ± 70
A4	1−0−0	3600 ± 68	2900 ± 40
A5	1−0−0	3550 ± 135	2830 ± 120
A6	1−0−0	3080 ± 57	2550 ± 29
A7	1−0−0	2930 ± 83	2370 ± 61
A8	1−0−0	2825 ± 41	1790 ± 35
A9	1−0−0	3025 ± 80	2490 ± 74
A10	1−0−0	2840 ± 77	2127 ± 88
B1	12−10−10	394 ± 37	300 ± 34
B2	12−10−10	345 ± 40	255 ± 38
		(mg L^{-1})	(mg L^{-1})
SB1	6−2−1	0.15 ± 0.03[a]	0.23 ± 0.03[a]
SB2	6−2−1	0.23 ± 0.02	0.24 ± 0.02
SB3	6−2−1	0.23 ± 0.02	0.25 ± 0.02

[a] Average concentration ± standard deviation from three replicates.

was made to maintain the pH of the solution at the desired pH, the pH did drift slightly in some samples. Upon completion of the leaching test, the final pH of the samples was measured. Each target pH was leached in triplicate.

Bioaccessibility Extractions. Leaching tests are sometimes used to estimate the amount of an element that might be bioaccessible when consumed by a human (10, 11). Bioaccessibility was measured on eight replicate subsamples of the 1−0−0 composite Ironite sample. The method used for this extraction was similar to a modified version of the physiologically based extraction test (PBET, ref 10) used by Yang et al. (11) to examine the bioaccessibility of arsenic in soils. The experiments conducted on Ironite were conducted at room temperature (25 °C), while those performed by Yang et al. (11) were maintained at 37 ± 2 °C. A 0.4 M glycine solution was prepared by mixing 30 g of 98% glycine (Acros Organics) in 1 L of reagent water. The pH of the solution was lowered to 1.5 using hydrochloric acid. This solution was designed to simulate the gastric fluid in a child's digestive system. Two grams of sample was placed in a 250 mL high-density polyethylene bottle, and 200 mL of the extraction solution was added. The samples were extracted for 1 h on a rotary extractor at 30 ± 2 rpm. During the extraction period, solution pH was monitored and maintained at approximately 1.5. After extraction, the sample was filtered using 0.45 μm filter. The metal concentrations in filtrate were measured using the ICP-AES.

Metals Analysis. Prior to analysis, the leachate samples were digested using the open-vessel method, which requires the addition of concentrated nitric acid and hydrochloric acid to a representative 100 mL sample. The analysis of arsenic and lead was conducted using a Thermo Jarrel Ash inductively coupled plasma atomic emission spectrometer (Trace Analyzer). Laboratory blanks, replicates, and calibration checks were performed as appropriate for quality control (QC) purposes.

Results and Discussion

Total Metal Concentrations. The results of the total arsenic and lead concentrations for the samples analyzed are presented in Table 1. The granular samples (1−0−0, 12−10−10) are reported in mg kg^{-1}, while the liquid samples (6−2−1) are reported in mg L^{-1}. Arsenic concentrations in the 1−0−0 samples ranged from 2840 to 3600 mg kg^{-1}, and the lead concentrations ranged from 1790 to 2900 mg kg^{-1} (Table 1). In general, these concentrations are typical of other

sample ID	grade	arsenic (mg L^{-1})		lead (mg L^{-1})		final pH	
		TCLP	SPLP	TCLP	SPLP	TCLP	SPLP
A1	1−0−0	1.77	5.72	5.34	2.85	4.53	3.76
A2	1−0−0	3.12	8.22	5.03	2.23	4.45	3.46
A3	1−0−0	1.43	2.35	6.91	2.88	4.62	3.94
A4	1−0−0	1.34	4.76	7.05	2.97	4.61	3.90
A5	1−0−0	0.62	3.37	7.95	2.83	4.57	3.92
A6	1−0−0	1.73	5.26	6.41	3.08	4.18	3.72
A7	1−0−0	1.63	5.14	6.71	3.37	4.31	3.09
A8	1−0−0	0.69	1.88	6.78	3.29	4.51	3.90
A9	1−0−0	1.88	5.44	6.58	2.57	4.43	3.74
A10	1−0−0	0.92	2.79	7.56	4.77	4.51	4.05
B1	12−10−10	6.18	6.28	0.02	<0.012	5.14	5.57
B2	12−10−10	6.21	6.33	0.03	<0.012	5.32	5.90

Ironite 1−0−0 formulations reported elsewhere (*3−6*). The arsenic and lead concentrations in the 12−10−10 samples were on the order of one magnitude lower than the 1−0−0 samples. Again, the 12−10−10 samples were only found in two of the stores surveyed. For comparison purposes, risk-based standards or guidelines for clean soils in residential settings are typically on the order of less than 1.0−10 mg kg^{-1} for arsenic and 400−500 mg kg^{-1} for lead (*12*). California uses total concentrations as part of TC hazardous waste determination; the California total threshold limit concentration (TTLC) for arsenic and lead are 500 and 1000 mg kg^{-1}, respectively (*9*). The 6−2−1 liquid Ironite samples contained from 0.15 to 0.23 mg L^{-1} of arsenic and 0.23 to 0.25 mg L^{-1} of lead.

Leaching Test Results. Table 2 presents the results of the SPLP and TCLP performed on the 12 granular Ironite samples purchased. In the 1−0−0 samples, lead leached more using the TCLP (5.0−8.0 mg L^{-1}) in comparison to the SPLP (2.2−4.8 mg L^{-1}). In a contrary fashion, the SPLP leached more arsenic (1.9−8.2 mg L^{-1}) as compared to the TCLP (0.69−3.1 mg L^{-1}). For most wastes, the TCLP usually results in greater metal leaching than SPLP (*13*, *14*). Factors that impact differences in leaching between the two tests include pH and the ability of the chemicals to complex with acetate. The final pH of the extraction solution was lower in the SPLP relative to the TCLP (Table 2) and will be discussed in relation to relative leaching differences in the next section.

The two 12−10−10 samples had different leaching characteristics as compared to the 1−0−0 samples. Lead leaching was much lower, with concentrations ranging from 0.02 to 0.03 mg L^{-1} with TCLP; SPLP lead concentrations were below detection. Arsenic, however, continued to leach at elevated concentrations, and these concentrations were similar between the TCLP and the SPLP. The final leachate pH values of the 12−10−10 samples were higher than the 1−0−0 samples, indicating that 1−0−0 was the more acidic formulation of the two. The dramatic differences in arsenic and lead leaching concentrations between the 1−0−0 and the 12−10−10 samples suggest that these two products have their own distinct composition and mineralogy.

Table 3 presents the results of the TCLP, SPLP, and WET performed on the composite 1−0−0 sample, for arsenic and lead, respectively. The composite sample contained an average 3370 mg kg^{-1} of arsenic and 2640 mg kg^{-1} of lead. In both cases, the TCLP and SPLP results followed similar trends as observed with the individual 1−0−0 samples with lead leaching more using TCLP and arsenic leaching more using SPLP. While the mean concentrations of arsenic calculated from the individual samples were somewhat lower than the average of the composite sample runs (apparently

extraction	arsenic (mg L^{-1})	lead (mg L^{-1})	final pH
TCLP	3.5 ± 1.3[b]	7.4 ± 0.2	4.39
SPLP	10.4 ± 1.7	2.6 ± 0.1	3.28
WET	106 ± 5	143 ± 4	4.91

[a] Total concentrations: As = 3370 mg kg^{-1} and Pb = 2640 mg kg^{-1}.
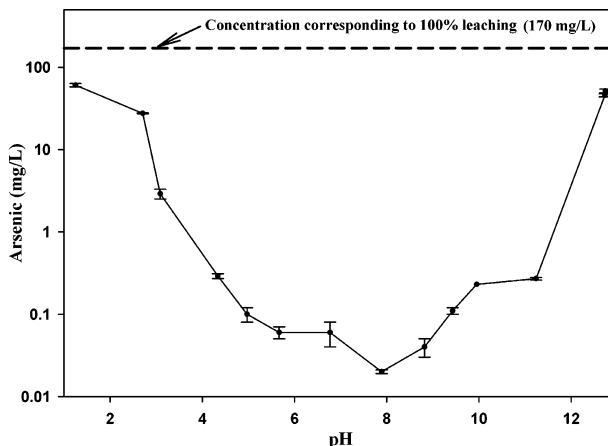[b] Arithmetic mean ± standard deviation of three replicates.

FIGURE 1. Arsenic leaching as a function of leaching solution pH (composite 1−0−0 Ironite sample; error bars represent standard deviation of triplicate runs).
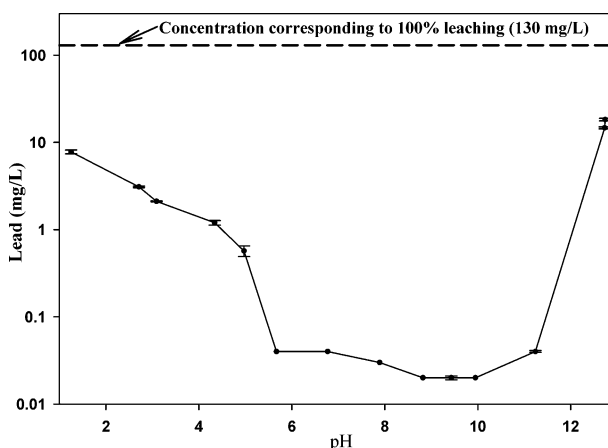

FIGURE 2. Lead leaching as a function of leaching solution pH (composite 1−0−0 Ironite sample; error bars represent standard deviation of triplicate runs).

a result of sample heterogeneity), the difference was not statistically significant (α = 0.01). For both elements, the WET leached over an order of magnitude more (106 mg L^{-1} for arsenic and 143 mg L^{-1} for lead). The WET has been shown to leach more than TCLP and SPLP for most elements and most wastes (*13*, *14*, *15*). Higher leaching with WET results from the ability of citrate to chelate metals. The final leachate pH for the WET extractions was higher than that for both the TCLP and SPLP.

Impact of Solution pH on Leaching. Figures 1 and 2 present the results of the experiment conducted to evaluate impact of pH on arsenic and lead leaching. The element concentrations measured in the leachates are plotted as a function of the leaching solution pH measured at the end of the 18 h extraction period. Overall, both lead and arsenic exhibited a typical amphoteric pattern where highest leachability was measured under low and high pH conditions. Also plotted on the graphs is the concentration that would

be expected if 100% of the element leached (based on measured total concentrations). Arsenic leached most at pH values of 1.25 and 12.74, with 36 and 31% of the total arsenic leaching, respectively. Arsenic leached least at a pH of 7.90, with 0.012% of the total arsenic leaching. At the lowest pH tested, lead leached 6% of that initially present, and at the highest pH tested, lead leached 14%. Lead leached lowest in the pH range of 7.90−9.95, with approximately 0.015% of the total lead leaching.

The information presented in Figures 1 and 2 helps to explain the differences between the TCLP and SPLP described earlier. For the 1−0−0 samples, the SPLP had a lower pH than TCLP, resulting in more arsenic leaching in the SPLP than the TCLP. The TCLP is a buffered weak acid solution, while the SPLP is a dilute solution of a strong acid. Since the initial pH of the SPLP solution is lower than the TCLP solution, the leaching test pH for acidic wastes or wastes with no or very little alkalinity will be lower for SPLP than TCLP. For wastes with even a moderate amount of alkalinity, the leaching test pH will usually be greater in SPLP than TCLP because of the TCLP's buffering capacity (*15*). The fact that lead leaches more in the TCLP and the WET even at a higher pH values than the SPLP results from the ability of acetate to complex and the ability of citrate to chelate with lead (*13*, *14*).

Bioaccessibility of Metals. Bioaccessibility refers to the fraction of the substance that is available for absorption by an organism. Leaching tests have been used to evaluate the bioaccessibility of contaminated soils in the past (*10*, *11*). These leaching tests typically involve one or two leaching steps where the chemical conditions in the stomach (and possibly the intestine) are simulated. A typical stomach pH condition of 1.5 is often utilized (*10*, *11*). As a first step, the magnitude of the bioaccessibility can be gathered from the data presented in Figures 1 and 2. At the two lowest pH values examined (1.25 and 2.70), 36 and 16% of the total arsenic leached, respectively, while 6 and 2% of the total lead leached, respectively. When the simple gastric acid leaching tests were performed, 37% of the arsenic leached, and 83% of the lead leached. Yang et al. (*11*) reported mean arsenic bioaccessibilities from arsenic-spiked soils in the range of 33−44%, a function of the age of the spiked soil.

When comparing the fraction of arsenic or lead leached at the lower pH values reported in Figures 1 and 2 with the values observed in the bioaccessibility experiment, the fraction of arsenic leaching compares relatively well despite differences in liquid-to-solid ratio and extraction time. The fraction of lead leaching, on the other hand, is much greater in the bioaccesibility experiment. This indicates that pH was the greatest controlling factor for arsenic leachability, while lead leachability was heavily influenced by the glycine. An amino acid, glycine chelates the lead resulting in a greater total concentration of lead in solution (*16*). Figure 3 compares the amount of arsenic and lead leached (%) from the gastric acid leach test with that extracted using the TCLP, SPLP, WET, and 3050B digestion.

Discussion

Ironite leachability data provide input on several issues related to the regulatory status and risks posed by this material. First, the data indicate that without the current regulatory exclusion from the definition of hazardous waste under RCRA (*17*), Ironite would meet the definition of a TC hazardous waste. The TCLP results found the 1−0−0 Ironite samples to exceed the TC limit for lead (5 mg L^{-1}), while the two 12−10−10 samples tested exceeded the TC limit for arsenic (5 mg L^{-1}). The 6−2−1 samples would not be a hazardous waste under U.S. rules. In California, unless otherwise exempted, the 1−0−0 samples would be hazardous for both arsenic and lead because of their results using the
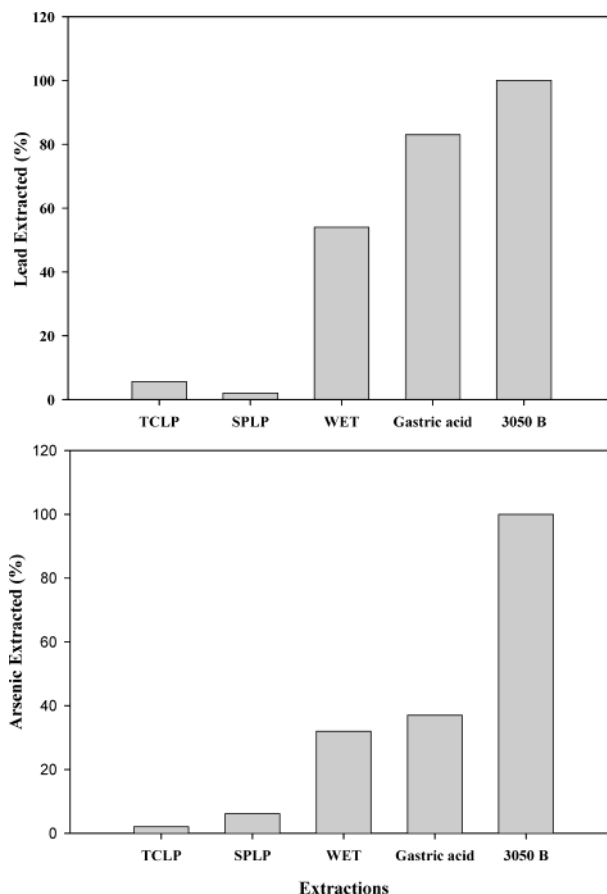

FIGURE 3. Comparison of fraction of lead and arsenic extracted from the 1−0−0 composite Ironite sample using different extraction methodologies.

WET and because the total arsenic and lead concentrations exceed the TTCLs. Because the exclusion from the definition of hazardous waste applies to the mine waste itself, unused and discarded Ironite would require management as a hazardous waste unless otherwise excluded (such as the household waste exclusion). Discarded Ironite would fit the profile of materials that local government household hazardous waste programs would collect.

The leachability data also help clarify issues regarding potential environmental risks posed by Ironite. While arsenic and lead concentrations are large, the mobility of the metals is highly dependent upon pH. The 1−0−0 Ironite product is acidic in nature, and when batch leaching tests are conducted, the pH will be relatively low (in the range of 3−4) even if water is used as an extraction solution. Thus, relatively high lead and arsenic concentrations will be observed in laboratory leaching experiments involving only the product. The mobility of lead and arsenic in the soil will largely depend on the pH occurring in the soil environment. Leaching would be expected to be higher in soils with minimal alkalinity relative to more buffered alkaline soils as illustrated in Figures 1 and 2. If arsenic and lead leachability of Ironite in a soil environment remains a concern, additional research examining long-term mobility in different soil types should be conducted.

Another concern expressed by some is the direct exposure of humans, particularly children, through the accidental ingestion of Ironite. In this case, the bioaccessibility of lead and arsenic in the human digestive system should be considered. The data from the pH experiment indicate that both lead and arsenic will be somewhat bioaccessible under lower pH conditions that might be encountered in the human

stomach. Arsenic leachability was controlled by pH, with the fraction of arsenic leaching in the range of 30−40% at pH values of approximately 1.5. In addition to pH, the presence of glycine greatly impacted lead leachability (6% with nitric acid vs 83% with the simulated digestive solution). Different methods are used to measure in vitro bioaccessibility, and not all of them use glycine (*18*). Since only a simple bioaccessibility experiment was conducted here, additional more rigorous experiments might prove valuable.

Acknowledgments

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