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Spring 2008

Development and Recycling of Novel Arsenic Removal Technology

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Development and Recycling of Novel Arsenic Removal Technology

Senior Honors Thesis

Morgan Liane Jones

Spring 2008

Approved by:

Abstract

As of 2006, the U.S. Environmental Protection Agency (EPA) changed the Maximum Contaminant Level (MCL) for arsenic from 50 parts per billion (ppb), to 10ppb because of links to cancer. Current remediation technologies are expensive; therefore, this change will result in increased economic pressure on rural communities with high levels of arsenic in their drinking water. Lowering of the standard has spurred the development of a novel remediation technology that has shown the ability to reduce arsenic in drinking water at the source, with the added benefit of low-cost disposal of a stable and benign waste product in ordinary landfills. Limestone-based materials appear to be an effective arsenic removal process that has great potential for source reduction in drinking water. A key requirement for any arsenic removal system is that the waste be disposed in a safe and cost effective manner. Phase one of this project focused on characterizing the long term stability of the arsenic iron-limestone waste product. The potential for the waste product to be disposed either in a normal landfill or recycled in cement is examined. Phase two of this research examined the potential use of pervious concrete as an *in situ* arsenic remediation system. This involved the designated construction of the Pervious Cement Reaction Barrier (PCRB). The research clearly showed that the PCRB has the potential to reduce arsenic in drinking water developed at the source. This is a significant expansion of the technical applications of the arsenic remediation technology. Toxicity Characteristic Leaching Protocol (TCLP) and California WET tests have indicated that the arsenic waste material is stable and can safely be disposed of in a landfill or incorporated into cement.

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"I can do all things through Christ who strengthens me" Philippians 4:13

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Introduction

 Impacts on humans from arsenic contaminated drinking water are a global phenomenon. Currently, numerous people are at risk of arsenic related diseases due to the consumption of underground water contaminated with arsenic. As seen on Table 1, an estimated 60 million are at risk in Asia, alone. The situation is especially critical in Bangladesh because the primary source of drinking and cooking water for most of its population is groundwater extracted from shallow aquifers through community network drinking. Since groundwater is a main contribution to Bangladesh's water supply, 75 million people are at risk and 24 million are potentially exposed to arsenic contamination in drinking water (Safiuddin, 2001). With a total population of approximately 140 million, the Government of Bangladesh considers the impact of groundwater arsenic contamination to be a national disaster (Ali, 2006). Other parts of the world have also suffered from this groundwater disaster. High concentrations of arsenic in groundwater has had toxic effects in local populations of India, Taiwan, Mongolia, China, Japan, Poland, Hungary, Belgium, Chile, Argentina, and North Mexico (Kundu, 2004). Although arsenic contaminated water in these countries is mostly due to naturally occurring arsenic in sediments and/or volcanic rocks, other parts of the world, including Thailand, Ghana, and the western United States, have developed arsenic contaminated drinking water from mining activities or gopher eradication efforts (Kundu, 2004). These activities include leaching of mine tailings or deposition of arsenic released to the atmosphere during the smelting process (Kundu, 2004).

- Not available

^a Estimated to be drinking water with arsenic $> 50 \mu gL^{-1}$,

From Smedley 2003 and data sources therein

b Before mitigation

c United Nations Childrens's Fund (UNICEF) estimate

^d Maximum

Table 1: Summary of the Distribution, Nature, and Scale of Documented Arsenic Problems ($>50 \mu g L^{-1}$) in Aquifers in South and East Asia

(Source: World Bank. Towards a More Effective Operational Response. Vol 1. Policy Report. 2005 p. 26)

 Arsenic is a toxic metalloid element. It occurs in different oxidation states in nature: +3 (As (III), also called arsenite) and +5 (As (V), also called arsenate). The speciation of arsenic is important in determining the chemistry and biochemistry behind aqueous arsenic solution. The actual distribution of arsenate (As (V)) and arsenite (As (III)), as well as the redox conditions, varies in nature but is influenced by pH, as shown by the arsenic speciation diagram. Arsenic speciation diagrams (Figures 1 and 2) represent the predominant forms of As (V) and As (III) present at a given value of pH. As (V) exists in four forms in aqueous solution: H_3AsO_4 (pH 0-2), H_2AsO_4 ⁻ (pH 2-7), $HAsO₄⁻²$ (pH 7-12) and As $O₄⁻³$ (pH 12-14). As (III) is found in three forms in aqueous solution: H₃AsO₃ (pH 0-9), H₂AsO₃⁻ (pH 9-12) and HAsO₃⁻² (pH 12-14). The primary As (V) species found in groundwater (pH values from 6-9) are $H_2AsO_4^-$ and $HAsO_4^{-2}$. In contrast, it is the uncharged form of As (III) (H_3AsO_3) that is found at typical groundwater pH values. Since drinking water pH typically ranges from 5.5 to 8.3, any method of removing arsenic can be determined by using the predominant species present. However, the method must be robust enough to remove all species. For example, at pH 7, arsenate is nearly an equal mixture of anions; whereas arsenite is virtually all neutral. Many remediation technologies are more efficient at removing arsenate than arsenite, due to charge electrostatic interactions with arsenate.

Figure 1: Arsenate Speciation Diagram (+5 oxidation state)

Figure 2: Arsenite Speciation Diagram (+3 oxidation state)

 Arsenic can have adverse effects on human health. Arsenite is about sixty times more toxic than arsenate and inorganic arsenic compounds are about 100 times more toxic than organic arsenic compounds (Jain and Ali, 2000). The ingestion of inorganic arsenic in drinking water could cause kidney, lung, bladder, skin, and liver cancer (Smith, 2003). Additionally, higher doses of inorganic arsenic compounds in the human body can lead to a disease called arsenocosis (Kundu, 2004). The symptoms of arsenocosis include thickening and discoloration of the skin, lesions, stomach pain, nausea, vomiting, diarrhea, loss of limbs, and hearing impairment (Gilles, 2000). Most of these symptoms only appear within five to ten years of exposure and can lead to death after fifteen to twenty years (Gilles, 2000). This is why the impact of the human crisis is just now apparent.

Due to arsenic's links to cancer and arsenocosis, the Environmental Protection Agency (EPA) has changed the drinking water standard in the United States for arsenic from 50 parts per billion (ppb) to 10 parts per billion (EPA, 2006). Additionally, the provisional guideline value set by the World Health Organization (WHO) for arsenic has been set at 10 ppb (WHO, 2001).

The lowered standard creates unique challenges for small rural water treatment facilities in the United States, including economic pressure on small rural communities with high levels of arsenic in their drinking water. Current remediation technologies, including coagulation/filtration and ion exchange, are quite expensive and are typically designed for the large water treatment facilities rather than small rural water treatment facilities. Not only are small rural systems at a disadvantage because of the high operating cost, but also due to the difficulty in obtaining well-trained operators and in

maintaining optimum operating conditions needed to successfully meet drinking water standards.

 The effects of ingested arsenic contaminated water is great and maintaining the arsenic standard for drinking water is difficult; therefore, there is an urgency to develop a low-cost remediation technology for arsenic removal that can be easily adapted to rural supply systems. This study is characterizing a robust new media for the removal of arsenic in drinking water using limestone as the base material. Limestone-based material provides several benefits to the drinking water community including reasonable removal efficiency as compared to material cost, compatibility with other water treatment processes, ease of use, and low-cost disposal in landfills (Webb et al., 2006). Our most recent media uses iron impregnated limestone and has been found to be comparable and efficient to the best available technologies, such as granulate ferric hydroxide. The type of limestone that is used in our technology is Minnekahta. Part of our research focus is on evaluating waste stabilization of this material. More specifically, we are exploring waste stabilization by incorporating the iron- limestone waste into cement.

Our *in situ* research focuses on removing arsenic from groundwater at the source. Our newest technological approach involves the integration of the iron impregnated limestone with pervious cement. The key characteristic of pervious cement is that it has an open pore structure that allows high rates of water transmission. The preparation of pervious concrete involves carefully controlled amounts of water and cement materials to create a paste that forms a thick coating around aggregate particles. Little or no sand is used in a mixture of pervious concrete to create a substantial void content. The use of the paste to coat and bind the aggregate particles together creates a system of highly permeable,

interconnected voids that drains quickly (Tennis, 2004). During our research, with the help of Mr. Mike Young, College Engineer- Supervisor of the Research Engineering and Support Shops, we prepared a column with pervious cement, hereafter known as a pervious cement reaction barrier (PCRB). Figure 3 illustrates the PCRB. This study will show how PCRB will be a cost effective technique to remove arsenic at the source of contamination.

Figure 3: Pervious Cement Reaction Barrier (PCRB)

Methods

Waste Stabilization Method:

Waste stabilization is an important criterion for any arsenic remediation technology. The technology must not only remove arsenic, but also be disposed in a safe and cost effective manner. This project examines the potential for arsenic rich iron-limestone waste to be disposed either in a normal landfill or recycled in cement. The first method for this project involved the preparation of cement cubes with iron-coated limestone waste. The iron coating was approximately 10 to 40 microns thick. Four mixes of arsenic laden iron-coated limestone were prepared with cement, sand, varied amounts of arsenic waste material, and water. The water-to-cement ratio of each mix was fixed at 0.45. Mixes 2-5 were mixed with 5%, 10%, 20%, and 30% of the proprietary limestone waste material provided by Rohm and Haas, respectively. Mix 1 was used as the control mix and contained 0% waste material. Table 2 illustrates the amounts of each substance used in preparing the mixes. After each mix was prepared, ten 2 by 2 inch cubes were made in accordance with the ASTM C 109 procedure. A mixture was filled into a 2 by 2 inch molding by three layers. Each layer was patted down 20 times with a tamper. After the cube was filled, the excess material was scraped off. Two cubes were made for each mix. The cubes were then cured for fourteen days in a humidity room at $73^{\circ} \pm 2$ F.

After fourteen days of curing, which is the process of hardening Portland cement, compressive strength tests were performed by a Forney 400K compression machine. The machine squeezed each cement cube until broken. The compressive strength of each cube is calculated by dividing the maximum load at failure (peak load) by the sample cross sectional area (Ozdemir, 2007):

$$
\sigma_c = \frac{F}{A}
$$

Where:

σ**c** = Unconfined Compressive Strength (psi)

 $$

 $A = Cross-sectional area of the core sample$

 (in^2)

Component	Mix ₁	Mix ₂	Mix 3	Mix 4	Mix 5
Cement (g)	250	238	225	200	175
Sand (g)	688	688	688	688	688
Fe-Ls Waste	$\overline{0}$	12.5	25	50	75
Material (g)					
Water (g)	113	113	113	113	113

Table 2: The Amount of Components for Each Concrete Mix.

 Samples of the concrete mixes were sent to Mid-Continent Laboratory in South Dakota to perform TCLP (Toxicity Characteristic Leaching Procedure) and Ca WET (California Waste Extraction Test). The TCLP is designed to determine the mobility of both organic and inorganic analytes present in either liquid, solid, or multiphasic wastes (EPA, 1992). If the leachate is less than 5 ppm arsenic then it may be disposed in a normal landfill; however, if it is greater than 5 ppm then the material is considered hazardous and the material must be sent to a certified hazardous waste disposal facility at a substantially increased cost. The CA WET is a more aggressive extraction procedure than the TCLP. The main differences between TCLP and CA WET are found in the rigor

of laboratory leaching solutions and the types of materials that are tested. Table 3 shows the primary differences between TCLP and CA WET.

Table 3: Differences between TCLP and Ca Wet (Source: California Environmental Protection Agency: Department of Toxic Substances Control)

Pervious Cement Reactive Barrier Method:

The second method for this project involved the preparation of pervious concrete. Two mixes of pervious concrete were prepared. The first mix consisted of 2.929 lbs of Type I Portland cement, 20 lbs of #9 limestone, and 1.098 lbs of water. The second mix consisted of 2.26 lbs of Portland cement, 16.86 lbs of #9 limestone, and .926 lbs of water. The water/cement ratio of each batch was fixed at 0.30. Each batch of cement was poured into 6 x 12 inch cylinders and then cured for 48 hours in a humidity room at $73^{\circ} \pm 2$ F.

After the cement was cured, the cylinders containing the pervious cement mixes were each placed in columns (Figure 3) and flow tests were performed. Two flow tests were performed with a stop watch for each PCRB. The preliminary testing for the flow rate (seconds/liter) was to establish the pervious cement's level of permeability and to characterize the flow rate of each column prior to testing. The flow rate was conducted by noting the amount of time each sequential liter took to dispense out of a 20 liter carboy.

 Two 20 liter carboys were used to prepare two batches of 210 ppm arsenic with 20 liters of de-ionized water by shaking the carboy. The third 20 liter carboy was used to dispense one batch of solution through the PCRB. The fourth 20 liter carboy was used to collect the solution leaving the PCRB (leachate). Tygon™ tubing was used to connect both the dispensing carboy and leachate carboy to the PCRB. The leachate was filtered using a 0.45 micron cellulose nitrate membrane filter in a syringe filter apparatus and collected in a 6mL vial. Additionally, a sample of the initial solution was collected. The pH of both the leachate and standard solution and the conductivity of the leachate were

measured using digital pH and conductivity meters. Overall, 14 standards and 18 leachates were collected over a four week period.

With the help of Mr. Rick Fowler at Western Kentucky University's WATERS analytical laboratory, graphite furnace atomic absorption spectroscopy (GF-AAS) was used to measure the amount of arsenic in the collected standard and leachate samples. GF-AAS involves the atomization of a sample by use of a graphite furnace. Atomization is the process which converts molecules of a solid or liquid substance to gas-phase free atoms, resulting in free atoms undergoing electric transitions from their ground state to excited electronic states (Petrucci, 2002). Additionally, GF-AAS involves the absorption of radiation from a light source by free atoms. The light source used during this analysis was a hollow-cathode lamp where emission was specific for the detection of arsenic.

The GF-AAS instrument is calibrated using five standards, all of known concentrations, and a blank sample to set up a calibration curve. After the five standards and their corresponding absorbencies are recorded, absorbance verses concentration is plotted and a line of best fit is drawn to determine the concentration of a sample based on the read out of the GF-AAS.

Results

Waste Stabilization:

 The compressive strength test and peak load values for each cement cube are shown in Table 4. The compressive strength was calculated by dividing the peak load by the cross sectional area. The control had an average strength of 2984 psi. The cement cubes containing 5%, 10%, 20% and 30% of the iron limestone arsenic waste presented average strengths of 3114, 2756.5, 1734, and 1474 psi, respectively. There is clearly a break around 10% with a range from 3130 to 2383 psi. The average compressive strength between 5% and 10% was 3119 ± 75 psi and the average compressive strength between 10% and 30% was 1760 \pm 390 psi. The average strength between 5% and 10% is much greater than the average strength between 10% and 30%; therefore, the cement cubes containing less than 10% Fe-LS arsenic waste can withstand great strength. This shows that cement with arsenic waste has the potential to be recycled as cement for normal use such as roadways and parking lots.

$%$ Fe-LS Arsenic Waste	Mix	Peak Load (lbs)	Area (in^2)	Compressive Strength (psi)
0%	1	12,600	$\overline{4}$	3150
0%	$\mathbf{1}$	11,270	$\overline{4}$	2818
5%	$\overline{2}$	12,820	$\overline{4}$	3205
5%	$\overline{2}$	12,090	$\overline{4}$	3023
10%	3	12,520	$\overline{4}$	3130
10%	3	9,530	$\overline{4}$	2383
20%	$\overline{4}$	8,180	$\overline{4}$	2045
20%	$\overline{4}$	5,710	$\overline{4}$	1423
30%	5	6,130	$\overline{4}$	1533
30%	5	5,660	$\overline{4}$	1415

Table 4: Compressive Strength of Concrete with Iron- coated Limestone Arsenic Waste as Aggregate

The TCLP and CA WET test results for each cement cube are shown in Table 5. The average arsenic level in the leachate for the TCLP tests, which were conducted at a pH of 2.88 due to the high alkalinity of the limestone based material, is 0.007 ppm. This value is slightly lower than the CA WET's result of .46 ppm arsenic because the CA WET procedure is a more rigorous extraction. CA WET's protocol requires an extraction fluid with a higher pH and a longer extraction time. It was expected that the level of arsenic in the leachate of the CA WET would be higher than that from TCLP due to the more rigorous protocol. However, all of the samples showed arsenic levels substantially less than 5 ppm, which is the regulatory limit. Since the samples were below the regulatory limit, the samples are suitable to be disposed in a normal landfill. This means that disposal costs will be minimized, which is a key advantage for this technology. Additionally, the final leachate concentration of arsenic for all components was well below the regulatory limit of 5 ppm as seen in Table 6.

Sample Test	Sample Description	Arsenic (ppm)	Regulatory Limit (ppm)
TCLP	Mix 1 - 0% waste	< 0.005	5
TCLP	Mix $2 - 5\%$ waste	< 0.005	5
TCLP	Mix $3 - 10\%$ waste	< 0.005	5
TCLP	Mix 4 - 20% waste	< 0.005	5
TCLP	Mix $5 - 30\%$ waste	< 0.005	5
CA WET	Mix 2 - 5% waste	0.081	5

Table 5: Results of TCLP and CA WET test for Concrete with Fe-LS Arsenic Waste

Table 6: TCLP and CA WET Test Results for Media Components

Flow Rate for Pervious Cement Reaction Barrier:

Before dispensing 216 ppb arsenic into the PCRB column, two flow tests were performed with a stop watch for each PCRB. The preliminary testing for the flow rate (seconds/liter) was to establish the pervious cement's level of permeability and consistency to characterize the flow rate of each column prior to testing. The flow rate was conducted by noting the amount of time each liter took to dispense out of a 20 liter carboy. Figures 4-7 illustrate the flow rates in each PCRB column with 11 liters flowing through. The flow rates were consistent throughout the tests. Column one flow tests resulted in an average of 42.6 ± 4.7 sec/L and 42.8 ± 7.3 sec/L, respectively. Column two flow tests resulted in an average of 40.1 ± 5.0 sec/L and 34.9 ± 4 sec/L, respectively. These results show that both PCRB columns had good flow capability and could continue to be used for the upcoming experiment.

Figure 4: First Flow Test for PCRB Column One

Figure 5: Second Flow Test for PCRB Column One

Figure 6: First Flow Test for PCRB Column Two

Figures 7: Second Flow Test for PCRB Column Two

Pervious Concrete Reaction Barrier:

After performing flow tests for both PCRB columns, PCRB column one was chosen to conduct initial experiments examining possible removal of arsenic. There were 14 standard arsenic samples and 18 leachate samples collected during the experiment.

The 14 standard samples displayed an average concentration of 216 ppb \pm 7 with an average pH of $4.3 \pm .2$. The leachate samples had an average pH of $11.2 \pm .1$ and an average conductivity of 240 ± 40 . The 18 leachate samples showed an average concentration of 168.6 ppb \pm 4.2. Since 216 ppb of arsenic was in the initial solution and 168.6 ppb of arsenic was in the leachate, 47.4 ppb of arsenic was removed by the PCRB column. This results in 21.9% of the initial solution of arsenic being removed. Figure 8 shows the result of the GF-AA analysis of both the standard and leachate samples for arsenic.

Figure 8: Arsenic concentrations of both

standard (blue-above) and leachate (pink-below) samples.

Discussion

Arsenic contaminated groundwater continues to be a major concern in the world; and small rural water treatment facilities still face challenges to meet EPA's arsenic standard for drinking water. The studies in this research have provided evidence that there is a possibility that these small rural water treatment facilities can overcome this obstacle by removing arsenic with a limestone based material and safely disposing it either in a normal landfill or recycling it in cement.

 For disposal in cement, there are two primary characteristics that prove that the combination of iron-limestone arsenic waste and cement is an effective waste disposal method. First, the cement cubes with iron-limestone arsenic waste illustrated that the mixture can maintain good consistent strength at an average of 3119 psi ± 75 with between 5% and 10% iron-limestone arsenic waste. Cement with iron-limestone arsenic waste has a characteristic of high strength enables this to be a waste disposal method by being recycled and used for roadways and parking lots since it would be able to withstand great weight such as with vehicles.

The second characteristic that allows iron-limestone in cement to be an effective disposal media is that it will generate a non-hazardous leachate under typical landfill conditions. The results from TCLP and CA WET were .007 ppm and .46 ppm, respectively. Both are well below the regulatory limit of 5 ppm arsenic; therefore, the leachate is considered safe and the cement waste can be safely disposed in a normal landfill.

Not only did this study focus on waste disposal, but also on removing arsenic at the source. Before beginning the experiment for arsenic removal, flow tests were

performed on both PCRB columns. Preliminary testing for the flow rate was performed to characterize the PCRB columns' permeability. There was no significant change in the flow rate from the beginning to end of the experiment as illustrated in figures 2-4. With an average of 42.7 sec/L and 37.5 sec/L in column one and two, respectively; the PCRB columns showed good flow capability.

Column one was selected to examine the possibility of arsenic removal. As seen in Figure 4, there was an average of 216 ppb of arsenic in the initial standard solution and an average of 168.6 ppb of arsenic in the leachate. The PCRB removed an average concentration of 47.4 ppm of arsenic from the initial standard solution. This results in an average of 22% of the initial standard solution being removed by the PCRB column. Arsenic removal was observed because pervious cement is made with limestone. Limestone is an excellent media for arsenic removal because of the presence of Ca 2+ ions and its buffering capacity. The presence of Ca 2+ ions in limestone will react with arsenate and precipitate as a calcium arsenate mineral, thereby removing arsenate from water (Webb, 2007).

Conclusion

The iron coated limestone met an important criterion: waste stabilization. The cement cubes containing limestone waste material are classified as non-hazardous and suitable for disposal in municipal landfills, as verified by TCLP and CA WET tests. In addition, the compressive strength tests indicate that the formation of concrete remains a viable option for either disposal or recycling of the limestone waste material at around 5% levels.

The pervious cement proved to be permeable and an innovative method to remove arsenic at the source. The PCRB had good flow capability with an average of 42.7 sec/L and 37.5 sec/L in column one and two, respectively. Additionally, with an average 22% being removed from the initial solution, the PCRB column one established the ability to remove arsenic.

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