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REMOVAL OF LEAD FROM CONTAMINATED WATER

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ABSTRACT

Over the past 25 years, about 750,000 sites with potential contamination have been reported to federal or state authorities. Heavy metals are the largest class of contaminants and also the most difficult to treat. This work focuses on the removal of lead from aqueous solution.

Lead pollution impacts all the systems of the human body. Many of the methods utilized to remove lead from wastewater have shortcomings. Alkali Ash Material Permeable Reactive Barrier (AAM-PRB) is a newly developed novel material made with fly ash alkali activating solution and filler material (sand and coarse aggregates). AAM-PRB was used to remove lead (Pb) from contaminated water. AAM-PRB materials with high permeability were created. Permeability must be high for a reactive barrier material, in the range of 10^{-2} cm/sec to 10^{-1} cm/sec. AAM - PRB from three ash sources with permeability of 10^{-1} cm/sec were produced and crushed into pellets. Effectiveness of the various barriers was determined by batch and column tests. Laboratory experiments indicate lead ion reduces from 1000 ppm to less than 2 ppm with 10 liters of solution and Lead ion from 10 ppm to less than 0.01 ppm. Analyses were performed by Atomic adsorption techniques.

Key Words: Contaminated water, lead, AAM, Fly Ash, Permeability, Reactive Barrier

1. INTRODUCTION

Essentially all industrial processes generate by-products that become waste materials. These waste materials have the potential of contaminating the environment. Heavy metals, solvents organic compounds, and petroleum products account for most of the contaminants.

The adverse impact of heavy metals, especially lead, on the environment and living organisms have been well documented by many researchers. The main focus of this

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paper is to show effectiveness of AAM in removal of lead from aqueous solution. Elements having atomic weights between 63 and 200 are heavy metals and have a specific gravity exceeding 4.0 (Freeman, 1998). Living organisms need trace amounts of some heavy metals, including cobalt, copper, iron, manganese, molybdenum, vanadium, strontium, and zinc. However, there is a limit beyond which it can be damaging to the organism. Non-essential heavy metals that are harmful to living organisms and have been encountered in both underground water and surface water systems are cadmium, chromium, mercury, lead, arsenic, and antimony (U.S. EPA, 1996).

Heavy metals have been used in a variety of ways for many centuries. For the past three centuries, the production of heavy metals such as lead, copper, and zinc has increased exponentially. Plumbing and insect control (as lead arsenate in apple orchards) are a couple of common examples of lead application. Exposure to heavy metals leads to developmental retardation, various cancers, kidney damage, and even death in some instances. Exposure to high levels of lead has also been associated with the development of autoimmunity.

Generally, all heavy metals that exist in underground and surface waters are in low concentration and in colloidal, particulate, and dissolved phases (Freeman, 1998). Heavy metals in the colloidal and particulate states are usually detected in hydroxides, oxides, silicates, sulfides; adsorbed to clay, silica, or organic matter. Metals in ions or un-ionized organometallic complexes are generally observed in soluble forms. The level of solubility of trace metals in surface waters is a function of concentration of ligands and level of pH (Freeman, 1998; Hongling et al., 2009).

Heavy metals in surface water or underground water systems can be from natural or synthetic sources. Heavy metals are present in more than half of Department of Defense (DOD), superfund, Department of Energy (DOE) and Resource Conservation and Recovery Act (RCRA) sites. The primary heavy metals of concern are arsenic, lead, cadmium, chromium, and mercury. Various forms of these metals may enter the environment from sources including abandoned dumping sites, wastes from metal plating and metal smelting, by-products from refining operations, and from manufacturing of computer components.

According to EPA there are over 200,000 contaminated sites in the United States alone that requires remediation (U.S. EPA, 1996). The estimated expense for this massive undertaking is at least $750 billion and can reach $1 trillion. This paper focuses on lead which is one of the most problematic heavy metals. Lead is dangerous to human health and the ecosystem because (Verstraeten et al, 2008):

a) Lead is in elemental form, and hence cannot be degraded or transformed into a less dangerous material, b) Lead is highly harmful to living organisms even in very low concentration, c) Lead contamination is already widespread in the ecosystem. Lead exposure in humans can have severe neurological and physiological repercussions.

1.1 Health Impact of Lead Exposure

Impact of lead exposure in humans has been known to cause wide variety of health problems such as (Navas-Acien et al, 2007; Woodruff et al, 2008; Iqbal et al, 2008):

- Various forms of blood disorders and Anemia
- Rapid deterioration of brain and the nervous system
- Reduced fertility both in men and women
- Failure of the kidney
- Alzheimer disease

In 2000, the allowable level of lead in drinking water was reduced from 0.6 ppm to 0.05 ppm. However, no level of lead is considered to be actually safe today.

1.2 Methods of Removing Lead from Contaminated Water

Removal of lead from industrial processes can be classified into several categories. Some of the most effective methods to remove lead from contaminated water are precipitation stabilization, ion exchange, and adsorption (Leighton et al, 2003; Von Lindern et al, 2003; Hadioui et al, 2008; Kaur et al, 2008; Kurniawan et al, 2006; Zhang et al, 2009, Dole, 2004; Babel and Kurniawan, 2003; Yabe and De Oliveira, 2003).

1.3 Background on In-Situ Permeable Reactive Barrier

Permeable Reactive Barriers or treatment walls, is a relatively new method of contaminated water remediation. The concept was first forwarded in 1989 at the University of Waterloo in Canada. The investigators conducted bench scale investigations using zero valent iron to breakdown chlorinated hydrocarbons. The first in situ demonstration was conducted in Borden, Canada. In 1992, the permeable reactive barrier technology, “funnel and gate system”, was first presented in scientific literature. In 1994, the first commercial application of permeable reactive barrier technology was installed in Sunnyvale, CA. This installation is still operating and has proven itself in terms of cost effectiveness compared to pump and treat remediation (US EPA RTDF, Sept., 1997). The prospects for Permeable Reactive Barriers as a cost effective alternative to pump and treat remediation are excellent. A considerable amount of effort is underway to investigate new cost effective materials which can be used to create new types of permeable reactive barriers with new functionalities. A promising new material called AAM barrier material has demonstrated the ability to remove heavy metals from highly contaminated water (Cd, Cr).

AAM material can have permeability ranging from 10^{-2} cm/sec up to 10^{-1} cm/sec. AAM contamination barrier materials utilize high permeability of 10^{-1} cm/sec. There exists a large body of literature on the various mechanisms of heavy metal removal and materials used for permeable barrier technology (Hilts, 2003; Schirnding et al, 2003;

Matlock et al, 2001; Dole, 2004; Al-Haj Ali and El-Bishtawi, 1997; Vaca Mier et al, 2001; Curkovic et al, 1997; Babel and Kurniawan, 2003; Yabe and De Oliveira, 2003). Materials range from clay minerals to synthetic zeolite, from fly ash to zero valent iron and metal oxides.

1.4 Background on Coal Combustion Fly Ash

In 2006, the United States consumed 1050 million tons of coal for electric generation. From this operation, about 108 million tons of coal ash was produced. Of this 130 million tons, 85 million tons are fly ash, 25 million tons are flue gas desulfurization (FGD), 15 million tons are bottom ash and the rest are boiler slag (U.S. EPA, 1996). Fly ash is by far the largest by-product produced. Only 37% of this huge quantity of fly ash (35 million tons) generated are reused or recycled, the remaining 73% is landfilled (U.S. EPA, 1996). The leading fly ash applications are shown in Table 1.

Table 1. Leading applications of fly ash

Applications	**% used**
Concrete	50
Structural Fill	14
Waste Stabilization	12
Mining applications	5
Road base	3
other	16

ASTM C-618 classifies the fly ash into two groups, class F (low CaO), and class C (high CaO). The current work focuses on the utilization of class F fly ash. The percentage composition of class F fly ash is shown below in Table 2.

Table 2. Range of composition of class F fly ash

Oxides	**% Content**
SiO2	45-65
Al2O3	20-45
Fe2O3	3-12
CaO	3-10
MgO	1-3
Alkali	<1.5
SO3	1-5
LOI(%C)	0.1-6

The conventional approach of using fly ash has been as filler in concrete. Addition of fly ash to concrete mixtures has been practiced for decades. However, the level of utilization of fly ash without having an effect on its properties is limited. Recently, conversion of fly ash to zeolites has been reported by many researchers (Nascimento et

al, 2009; Roman-Zamorano et al, 2009; Qiu and Zheng, 2009). Zeolites possess uniform molecular pore and large surface areas, which makes it an ideal material for molecular sieve.

1.5 Background on Alkali Ash Material

A considerable effort is underway to investigate cost-effective materials, which can be used to create new types of permeable reactive barriers with new functionalities. It has been shown in our laboratory that a promising material called Alkali Fly Ash Permeable Reactive Barrier (AAM-PRB) material has the ability to remove heavy metals such as lead from highly contaminated water. The authors of this paper have published several papers on the subject of chemically activated fly ash cementitious material (Rostami et al, 2009; Rostami et al, 2006; Rostami et al, 2005; Rostami, 2004; Rostami, 2004; Rostami and Brendley, 2003; Jahanian and Rostami, 2001; Rostami et al, 2001; Rostami, 2004). AAM-PRB material is created from an alkali-activation process, in which a highly caustic sodium hydroxide solution, mixed with concentrated sodium silicate (aq), reacts with fly ash to create a material with cementitious characteristics and a unique microstructure. Alkali Fly Ash (AAM) material can be created with a permeability ranging from 10^{-9} cm/sec to 10^{-1} cm/sec. Preliminary work has shown that the AAM-PRB material can efficiently (99%) remove lead from highly contaminated water (1000 ppm).

Along with the sodium silicate and sodium hydroxide, AAM consists of class F fly ash and sand. Fly ash, the by-product of coal burning operations, has properties and chemical compositions that vary according to the coal source and power plant operations. This variability makes AAM barrier material extremely versatile. By varying the ash type, different contaminants can be removed.

2. MATERIALS AND METHODS

2.1 Preparation of Samples

Four different types of class F fly ash will be employed in construction of AAAM material. Selected fly ashes for this experiment represent a wide range of ash generated by coal burning operation. XRF was used to obtain the composition of fly ashes (provided by ash supplier). These fly ashes have compositions as shown in Table 3. Unlisted trace amounts of heavy metals are known to be present.

To make the AFA-PRB material, fly ash ASTM C -33 sand, sodium hydroxide 50%, and type N sodium silicate will be used. Mix design of a typical AAM-PRB is shown in Table 4.

AAM-PRB mixture consists of approximately 90% by weight solid and the rest is liquid. The solid portion consists of class F fly ash, fine aggregate and aluminum powder. The liquid part contains sodium hydroxide, sodium silicate and water. The mixing

Table 3: Ash Composition

Oxides	Source 1	Source 2	Source 3	Source 4
SiO_2	47.0	63.2	53.8	59.9
Al_2O_3	23.0	19.4	23.4	15.0
Fe_2O_3	21.4	5.4	6.0	5.9
CaO	2.1	4.3	8.9	10.7
MgO	0.8	1.3	1.9	2.4
Alkali	1.4	1.1	1.0	0.2
SO_3	0.05	1.4	0.9	1.0
LOI (%C)	12.0	2.1	0.6	3.3
Heavy metals	Trace	Trace	Trace	Trace

LOI refers to loss on ignition and provides % C

Table 4: Mix Design for AAM-PRB

Material	Weight (gm)	%Weight
Sodium Hydroxide	50	1.78
Sodium Silicate	50	1.78
H_2O	200	7.14
Fly Ash	1250	44.60
Fine Agg.	1250	44.60
Al Powder.	3	0.11

procedure consists of a) thoroughly mixing fly ash, fine aggregates and aluminum powder, b) mix sodium hydroxide, sodium silicate and water together, which represent the activating chemicals, c) add the activating chemicals to the solids and mix them well until uniformity is reached. Well-mixed AAM-PRB materials are allowed to cure at room temperature for at least 12 hours. Addition of aluminum powder, which reacts rapidly with high alkaline solution resulting in release of hydrogen gas along with proper-mix design, can produce materials with wide range of permeability. The process of creation of AAM-PRB will convert fly ash from powder form to pelletized form. Various pellet size ranges can be obtained by compaction and changing the mix design. AAM-PRB economy is very attractive. Cost of components of AAM-PRB is summarized in Table 5.

Table 5: Cost of Materials

Sodium silicate	Sodium hydroxide	Fly ash	Sand	Al powder
\$100-\$160 per ton	\$120-\$190 per ton	\$10-\$30 per ton	\$10-\$20 per ton	\$40-\$60 per kg

Based on the cost of material in Table 5, a typical AAM-PRB mixture can cost from \$35 to \$60 per ton. The cost of natural and synthetic zeolites is much higher than that of AAM-PRB and the cost of ion exchange resins are far greater than the cost of zeolites.

2.2 Experimental Work

The experimental work was divided into the following tasks.

2.2.1 Task 1: Fly ash contaminants

To ensure that any heavy metal contaminants already part of the fly ash are not leachable from the material under conditions of expected use. Leaching experiment using uncontaminated water with a pH on the order of natural rainwater, pH~5.6, and pH encountered in contaminated effluents were considered. One thousand bed volume of natural rain water passed through the AAM barrier, and from every 100 bed volume a sample was taken out and heavy metals (lead, mercury, cadmium, chromium) concentration and pH level was measured. After 300 bed volume of natural water, the pH was stabilized to 6.8 and remained constant throughout. Testing of effluent for heavy metals after 1000 bed volume revealed that no heavy metals are present in effluent and the AAM barrier does not release heavy metals to the environment.

2.2.2 Task 2: Permeability control and measurement

To measure the permeability of AAM-PRB material, the Constant Head Permeameter Apparatus from ASTM D-2434 was utilized. A 7.5 cm diameter AAM-PRB material sample is placed in the permeameter according to ASTM D-2434 procedures. After opening the inlet valve, flow of water through the specimen is allowed until a steady state is achieved with no change in hydraulic gradient. Two approaches were used to produce the controlled permeability materials:

a) Controlled gradation of aggregates, binder volume and compaction effort
b) Inclusion of aluminum powder as gassing agents

AAM-PRB materials were prepared from a mixture of fly ash, sodium silicate, water, and sand. Various methods of processing and materials were utilized. AAM-PRB materials utilized in the creation of the Permeable Reactive Barrier material are:

a) Ambient cured, low calcium fly ash
b) Ambient cured, high calcium fly ash

The results of this work can be seen in Table 6.

Creation of high permeability material using both gassing agents and controlled gradation/ compaction methods were possible, however, using gassing agents proved to be more repeatable.

2.2.3 Task 3: Pb Removal

To evaluate the effectiveness of AAAM as a barrier, two testing schemes will be utilized. The batch test is a quick screening procedure. The column test involves flow and is comparable to real conditions. The result obtained from the column test will be used to construct a bench scale permeable reactive barrier.

Table 6: Comparison of AAM Materials formed from Different Ash, Different Curing, Foaming Agents, and Controlled Gradation/Compaction

AAM-PRB Barrier Materials	Permeability (cm/sec)	Strength (psi)	Repeatability
Gassing Agents			
Source 1	10^{-2}-10^{-1}	50-200	poor
Source 2	10^{-2}-10^{-1}	50-200	very good
Source 3	10^{-3}-10^{-1}	50-400	poor
Source 4	10^{-3}-10^{-1}	50-250	good
Controlled Gradation/Compaction			
Source 1	10^{-5}-10^{-2}	100-900	average
Source 2	10^{-5}-10^{-1}	100-400	good
Source 3	10^{-7}-10^{-5}	100-700	average
Source 4	10^{-5}-10^{-3}	100-400	good

2.2.3.1 Batch Test

Batch tests are normally carried out to choose a reactant (and the proper amount) to remove the heavy metal. Batch tests are quick methods that allow rapid screening of potential reactants. Batch studies were performed by placing 5 to 25 g of crushed barrier material in a test container, and then adding (by weight) 20 to 100 times of lead contaminated water, and sealing the sample container. The sealed sample is then rotated at 30 rpm, thoroughly mixing contaminated water and crushed barrier material. The water is periodically withdrawn for analysis by Atomic Adsorption (AA) and pH measurement. Since batch testing does not involve the flow of water, it does not simulate an actual situation.

2.2.3.2 Column Test

After selection of barrier material from the batch test, a column test was conducted. The optimum column diameter should be at least 40 times greater than that of the average crushed barrier particle material of 15 to 40 mesh. Therefore the column should be at least 2.5 cm in diameter or greater. The optimum length of the column should be greater than or equal to four times the column diameter, indicating that the barrier column should be at least 10 cm in length. The volume of barrier material in the bed would be 50 cc. AAM-PRB barrier material has a wet bulk density of 0.85 g/cc, thus the amount of material required to fill the optimum size column is approximately 95 g. To minimize the waste, a 2.5 cm x 10 cm column will be used. This will result in 95 g of barrier material. Samples of laboratory solutions with varying Pb concentrations of 10, 100, and 1000 ppm were passed through columns containing barrier material samples. Water samples were withdrawn every 0.5 liter for analysis by atomic adsorption (AA) and for pH measurement.

3. RESULTS ON HEAVY METAL REMOVAL ABILITY OF AAM

AAM barrier material was used to remove Pb from contaminated water. Fly ash from four different sources was used to produce reactive barrier of different permeability. AAM materials with permeability ranging from 10^{-2} to 10^{-1} were created. Only the highest permeability proved to be viable for reactive barrier material.

3.1 Batch test

In this work, 10 grams of barrier materials was added to 500 gram of 1000 ppm solution of Pb. The removal ability of AAM from source 1 and 4 was less than source 2 and 3, while AAM from source 2 and source 3 had similar removal efficiency. AAM from source 2 had more work time during processing, thus AAM from source 2 was selected for column test. Table 7 below shows the result of batch test of AAM for Pb removal ability.

Table 7. Results of batch testing

AAM Barrier	1000 ppm Pb
Source 1	142 ppm
Source 2	0.21 ppm
Source 3	0.23 ppm
Source 4	94 ppm

3.2 Column test

AAM reactive barrier constructed from fly ash obtained from source 2 was used in the column test. In this work, 10 liters of 10, 100, 1000 ppm Pb with increments of 0.5 liter were passed through 95 gm of barrier materials. Figure 1 shows the ability of AAM to remove Pb from 10 ppm contaminated water.

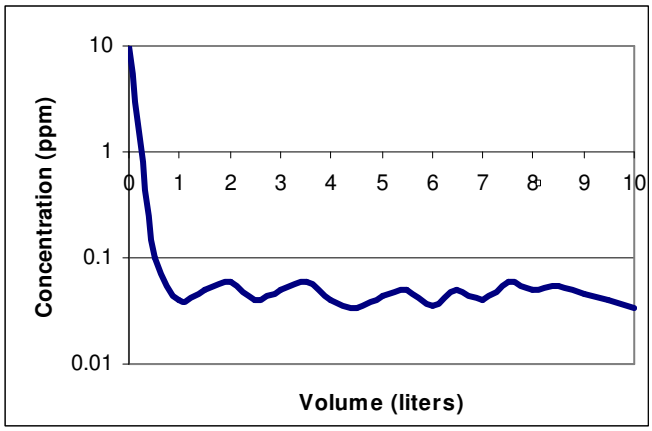

Figure 1. Influence of AAM barrier on removal of lead (10 ppm) from volume of contaminated water.

After permeation of 10 liters of 10 ppm lead solution through the column containing 95 g of AAM, effluent of solution containing 10 ppm lead was reduced to about .05 ppm. Figure 2 shows the ability of AAM to remove Pb from 100 ppm contaminated water.

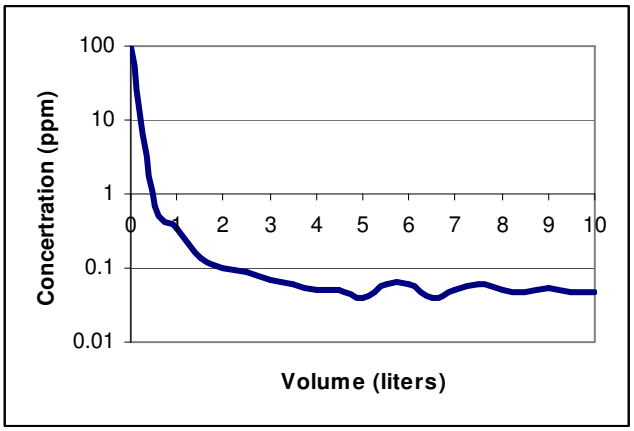

Figure 2. Influence of AAM barrier on removal of lead (100 ppm) from volume of contaminated water.

After permeation of 10 liters of 100 ppm lead solution through the column containing 95 g of AAM, effluent of solution containing 100 ppm lead was reduced to about .06 ppm. Figure 3 shows the ability of AAM to remove Pb from 1000 ppm contaminated water.

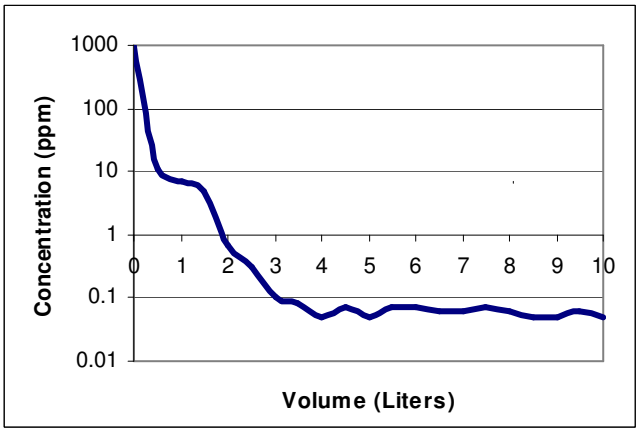

Figure 3. Influence of AAM barrier on removal of lead (1000 ppm) from volume of contaminated water.

After permeation of 10 liters of 1000 ppm lead solution through the column containing 95 g of AAM, effluent of solution containing 1000 ppm lead was reduced to about .08 ppm. Figure 4 shows the ability of AAM to remove Pb from 10000 ppm contaminated water. 10,000 ppm lead experiment was conducted to see whether the breakthrough would be achieved.

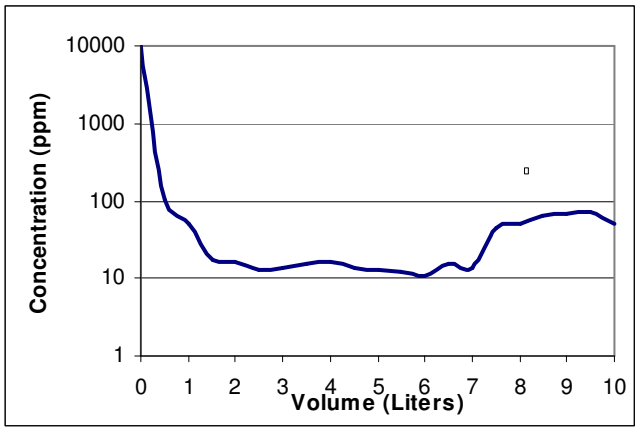

Figure 4. Influence of AAM barrier on removal of lead (10000 ppm) from volume of contaminated water.

After permeation of 7 liters of 10000 ppm lead solution through the column containing 95 g of AAM, it appears that breakthrough was achieved.

From the result obtained in column test, AAM has the ability to remove Pb very effectively. AAM contaminant barriers are very economical. The cost of materials will be similar to the cost of a Portland cement system. The basic materials to create AAM are fly ash, sodium silicate, and sodium hydroxide. These materials are available all over the United States in bulk volume for commodity prices. The manufacturing cost will be similar to that of precast concrete products for manufactured material and ready mix concrete for ambient temperature applications. The cost of zeolites will be similar to the cost of currently available ion exchange materials. This represents the most significant cost. If AAM is viable as a contaminant barrier without the addition of ion exchange material, this will be the most economical option. Typical cost of materials for AMM compositions without stone are $40 to $50 per ton. The manufacturing operation for elevated temperature curing should increase this price to about $100 per ton.

4. CONCLUSIONS

Since the Industrial Revolution, the production of heavy metals such as lead, copper, and zinc has increased exponentially. Heavy metals have been used in a variety of ways for many centuries. For example, lead has been used in plumbing, and lead arsenate has been used to control insects in apple orchards. Exposure to heavy metals has been linked with developmental retardation, various cancers, kidney damage, and even death in some instances of exposure to very high concentrations. Exposure to high levels lead has also been associated with the development of autoimmunity, in which the immune system starts to attack its own cells, mistaking them for foreign invaders.

In this paper, the removal mechanism of lead from contaminated water varies based on the pH of the solution. At high pH the removal is mainly precipitation of heavy metal to the surface of the barrier materials. The adsorption is removal mechanism as the pH

level drops below 9 while the pH is lower than 6, the removal process is the ion exchange.

Removal of heavy metals such as lead from industrial processes can be grouped in several known techniques. Some of the most effective methods to remove lead from contaminated waters are precipitation stabilization, ion exchange and adsorption. Investigation of AAM in the past 3 years has demonstrated that fly ash can be transformed into an effective sorbent for heavy metals. AAM has the ability to remove heavy metals such as Pb from contaminated solution. 1000 ppm of Pb solution was reduced to about 0.6 ppm after passing through the optimized Controlled Permeability AAM contamination barrier. The process has been successful with class F fly ash from many sources. Further work is needed on the methods of production of material in larger scales.

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