

Using Calcium Stearate as a Modifier for fly ash stabilization

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Abstract

Heavy metal leaching from improperly disposed fly ash remains a concern to human health and the environment as a result of their toxicity. The objective of this experiment is to evaluate the performance of calcium stearate to encapsulate heavy metals in fly ash to prevent chemical leaching. To assess the ability of the additive to solidify heavy metals, batch leaching tests and column leaching tests were performed. The methodology involved testing fly ash with various amounts of additive. The fly ash with the additive was evaluated by measuring the concentration of heavy metals such as Chromium, Lead and Cadmium that leached from the fly ash. Batch leaching results indicated that the concentration of chromium was $65 \frac{\mu g}{L}$ for fly ash alone. However, with 1% calcium stearate the concentration dropped to $45.5 \frac{\mu g}{L}$. Column leaching results revealed that the concentration of cadmium was $166 \frac{\mu g}{L}$ for 3% calcium stearate. On the other hand, with 1% calcium stearate the concentration fell to $93.4 \frac{\mu g}{L}$. Further testing revealed that 1% calcium stearate reduced the amount of heavy metal leaching from fly ash. It is hypothesized that the calcium stearate attacks the transition metals rendering the metal essentially inactive due to the free lime hydration.

Keywords: Heavy metal leaching, Fly ash, Water-repellant

1. Introduction

Fly ash is a common by-product of the coal combustion process that has been studied for reuse in the construction industry for many years. Yao et al. (2014) explain that the world generates approximately 750 million tons of coal-based fly ash each year. Coal fly ash is hazardous both to human health and the environment if disposed of improperly. Due to the pozzolanic properties of fly ash which increases the durability, workability, and strength of concrete products, the global demand for increased usage of fly ash has significantly increased in growing construction industries. However, there is a tremendous difference between the amount of fly ash produced globally and the amount disposed of properly through recycling and other means.

According to the Environmental Protection Agency, (EPA 2015) in the United States alone, over 110 million tons of Coal Combustion Residuals (CCRs) have been generated. However, there are less than 25% of the total quantity of fly ash produced

in the world has been recycled (Zentar et al., 2012). Given the realities that up to 75% of fly ash are disposed of by other means such as placed in embankments, landfills, and ash ponds, there is a high probability of heavy metal leaching into groundwater. The issue is the presence of trace metals in the fly ash which leach out of the combined materials and cause detrimental effects to the environment as well as humans. Hence, the removal or solidification of these metals is of utmost importance attempting to recycle fly ash and creating a sustainable final product. Water dissolution, phosphating and adding sodium carbonate have been reviewed and tested as possible methods to remove these metals, but not all elements have become effectively stabilized (Aubert et al., 2006). In terms of solidification, most solutions lead to mixing the by-product with cement, which has been proven effective but fails to control the leaching of various metals. Yao et al. (2014) states currently, 20% of coal-fired fly ash is used in concrete production with it also being used in road base construction, and

soil amendment. More recently, a form of organic modification that uses organosilanes to irreversibly bond with fly ash has been introduced as water-repellant technology (Daniels et al., 2009). With this idea in mind, CALSANTM 50, a product made by BASF which is a calcium stearate dispersion in water was believed to be a suitable replacement (BASF 2009). The objectives of this paper are to (1) review previous work regarding the use of waterproofing substances as an option for the solidification of fly ash, (2) evaluate the performance of calcium stearate through chemical leaching testing in various conditions and (3) provide commentary on whether this is a viable option for attenuation of fly ash metals.

2. Background

Many methods have been tested to explore the possibility of recycling fly ash in order to reduce the volume of waste while providing a comparable and safe alternative to landfills. Among these methods are the inclusion of slag, alkali-activation, metakaolin, Portland cement and nano-silica (Rashad, 2014). For this experiment, the process of encapsulation will be explored as an option to mitigate leaching levels to EPA regulatory standards.

In order to begin comprehending how to prevent fly ash from leaching into the ground, a basic understanding of what fly ash is must be acquired. As stated previously, fly ash is a common by-product of the coal combustion process. Depending on how the coal is processed, a wide range of properties could be seen when analyzing different ashes. Cho et al. (2005) state coal fly ash is extremely varied in its mineral makeup, and its characteristics depend heavily on the kind of coal used and the way in which it was burned as well as several other factors. On average, fly ash occurs as fine particles with a spherical shape and an average size of fewer than twenty micrometers, has low to medium bulk density and a high surface area for its small size. The pH values range from 1.2 to 12.5, but the trend is usually towards alkalinity.

The common heavy metals found in fly ash include Chromium, Lead, Cadmium, Nickel, Barium, Strontium, Vanadium and Zinc (Yao et al., 2015). Since there are hazardous metals in fly ash a method must be created in order to encapsulate the contaminants eliminating the chance for leaching to occur.

There have been many tests performed in order to determine the best method of containing the toxic metals that are present in fly ash. The leaching of toxic metals from fly ash is the primary inhibitor to the widespread use of fly ash in construction projects. Van der Sloot speaks how leaching is a function of the surface exposed to the leaching fluid. The ratio of the particle surface area to the volume occupied by the particles, the average particle size, and internal pore structures in the material all control the surface area where dissolution from the solid to the liquid can occur. Smaller particle sizes produce larger surface area, allowing for increased contact between the solid material and the leaching fluid, resulting in increased contact between

leaching fluid and leachable constituents (Van der Sloot, et al. 1997).

Kerkhoff shows that fly ash can be used as liming material on acid soils or acid mine soils or alkali soils for improving the pH of the soils depending on the nature of soil and ash. Increases in pH induced by alkaline fly ash addition is a desirable property and could be used for detoxifying elements like Cd, Al, and Mn. Due to the fine nature of fly ash, it improves the WHC of sandy soils removing the compaction of clay soils. It improves the physical and chemical properties of soil as well as the biological properties of problematic soils. Application of fly ash, particularly unweathered ones, shows a tendency of accumulating elements like B, Mo, Se, and Al, whose toxic levels are responsible for a bad influence on animal and human health, (Kerkhoff 2001).

Cinquepalmi et al. investigated the use of fly ash as an artificial aggregate in Portland cement mortars and found that the release of each metal increased with an increasing amount of artificial aggregate in the mortar specimens. Also, it was found that pH has a great influence on the leachability of heavy metals from the cement mixture. The greatest effect was shown at pH levels below 6-8 and as the pH rose, the release of metals decreased to a point where the amount of artificial aggregate in the mixture had nearly no effect at all (Cinquepalmi et al., 2008).

The ionic strength of the leaching fluid also influences solubility and leaching behavior. Ionic strength is the relationship between the concentration of ions in solution and the charges of those ions. Ionic strength impacts reaction rates as well as the solubility of ionic species, with solubility, generally increasing as ionic strength increases, (Lowenbach, 1978).

Becker, et al., (2013) determined that after several experiments using the Environmental Protection Agency's testing guidelines for fly ash leaching, fly ash leached toxic metals at levels exceeding the regulatory limits. It was noted that pH levels can directly affect the rate and amount of leached metals, and each batch test conducted should be adjusted to accurately reflect site-specific conditions. There is typically a non-linear relationship between leachate metal concentrations and fly ash content, thus requiring full testing be done to better estimate the amount of leached material. Additionally, column leach testing was found to not accurately represent long term leaching risks, and therefore should not be conducted with that purpose in mind.

Singh, et al., compared the chloride resistivity of nanoparticles to fly ash and concluded that in general, fly ash does not prevent chloride permeability as well as silica nanoparticles. A silica nanoparticle content of 3% was shown to improve chloride ion resistance up to nearly 40% when compared to plain cement. Adding fly ash alone reduces the strength of the concrete compared to not adding any, but adding the fly ash and a small amount of silica increases the compressive strength of the concrete. Park et al, (2006) found that adding small amounts of silica, 4% by weight, increased strength in concrete bricks up to nearly 20.8MPa. Commercial

bricks made with natural sands had compressive strengths of around 11.9MPa. Most notably, it was found that the silica helped reduce the leaching of the hazardous substances from the solidified ash.

“It was observed that the efficiency of nanoparticles such as nano-silicon oxide depends on their morphology and genesis, as well as on the application of superplasticizer and additional treatment options such as thermal treatment and ultra-sonification. The method studied here is capable of manufacturing a wide range of nanoparticles with engineered parameters such as particle size, porosity and surface conditions. It was demonstrated that all synthesized nano-silicon oxides improve the early compressive strength of Portland cement mortars, but at later stages of hardening, strength was adversely affected by these additives. The major problem of nano-silicon oxide application and such strength loss is related to the agglomeration of nanoparticles at the final drying stage. High-temperature treatment affects the performance of these additives and should be avoided. Further research is required to modify the sol-gel method in order to avoid the formation of agglomerates and to achieve better dispersion of developed nano silicon oxides” (Drexler 1991).

(Malhotra 1988) explains that “Concrete containing Class F fly ash exhibited higher long-term resistance to chloride-ion penetration compared to Class C fly ash concrete. The best long-term performance was recorded for both of the 53 percent and the 67 percent Class F fly ash and 70 percent of Class C fly ash concrete mixtures as they were found to be relatively impermeable to chloride ions in accordance with ASTM C 1202. Except for control mixture C-4, the differences in the Coulomb values of the high-volume fly ash mixtures are not significant. All fly ash concrete mixtures showed excellent performance with respect to chloride-ion penetration resistance.”

Al-Saadoun, et al., (1993) determined that fly ash blends of cement typically outperform plain cement in corrosion resistance of reinforcing steel. Fly ash blends significantly affected the initiation time for corrosion and in some cases tripled the time required for corrosion to begin. Notably, lignite fly ash performed best when compared to bituminous and sub-bituminous fly ash. Since the corrosion of steel is an electrochemical process, it was shown that having a 25% fly ash mixture can increase the electrical resistivity of cement threefold.

Fly ash has a tendency of not acting as a pozzolanic material until at least one week after being mixed. Fraay, et al., (1989) determined that fly ash created as a byproduct of bituminous coal (class F) contains crystalline inclusions which are primarily comprised of alumina-silica glass. This crystalline structure is the cause of the delayed hydration when mixed with Portland cement. The pozzolanic reaction is explained by the dependency of breaking down the glass on the alkalinity of the pore water. High alkalinity in conjunction with the precipitation of reaction by-products can inhibit the breaking down phase, ultimately delaying the pozzolanic reaction from the fly ash. Additionally,

the pH and temperature of the pore water can affect the reactivity of the fly ash as well as the solubility of the glass. In most cases, lower pH levels create less reactive fly ash which, in turn, delays the pozzolanic state from occurring. Cao., (2010) determined that by influencing the micro-gradation of cement the delayed pozzolanic reaction of fly ash could be counteracted and early strength could be improved. Since fly ash slows the early strength generated by concrete, slag was incorporated into the design to improve early strength. Since slag on its own can produce harmful air voids, the fly ash will be used to counteract this by filling the air voids. Cao found that the optimal fly ash to slag ratio was 4:1 and performed best when an activator was used. When the activator content was at 2%, the day one compressive strength and flexural strength was approximately 21% and 10% higher, respectively, when compared to concrete with just fly ash. These numbers rose when the activator was increased to 3%. The activator significantly speeds up the hydration process and increases early strength in cement with fly ash and slag additives. This same method could potentially be used in speeding up the hydration of fly ash cement mixes to help move the pozzolanic reaction along.

Rashad reviewed many experiments that tested various admixtures to fly ash and the effect that occurred. One experiment studied the compressive strength with various amounts of fly ash added to Portland cement. From the experiment, it was found that a ratio of 60 percent fly ash to 40 percent of Portland cement yielded the best results for mechanical performance. This is good to keep in mind because for the proposed experiment a comparable product must be made in terms of strength and not just reduced leachability (Rashad, 2014).

Most of these procedures were completed using one or more of the following testing methods: batch, column or block. For the purposes of this paper, it was found sufficient that batch testing and column testing be performed.

"Batch extraction tests typically involve mixing a sample of waste or other fill material with a specific amount of leaching solution without renewal of the leaching solution. The mixing is performed over a short period with the aim of trying to reach equilibrium with some type of rotary agitation. The mixing is followed by filtration and analysis of the filtered liquid phase" (Washington State Department of Ecology 2003).

“A column leaching procedure is used to classify waste as hazardous or not and to determine the effectiveness of a waste treatment process. A column leaching procedure using a leaching fluid being pumped into a sample material in a column. One should then use a tube coming out of the sample column and into an effluent to sample the extracts” (Washington State Department of Ecology 2003).

By using calcium stearate technology, an effective way of encapsulating heavy metals within a cement matrix is expected.

3. MATERIALS AND METHODS

The fly ash studied was donated from Headwaters Resources and came from Brayton Point. Headwaters Resources lists the material density at 2.38 grams per centimeter cubed.

The calcium stearate (Calsan™ 50) was procured from BASF Corporation in New Jersey. It is a part of the metal stearate family and is used primarily as a coating lubricant.

The columns used for the column leaching tests had dimensions of 4.5 inches in diameter by nine inches in height for a total volume of 143.14 cubic inches. They were connected to a gravity head system to conduct the column tests.

A spectrophotometer was used to determine the amount of light that a sample of the leachate could absorb. The intensity of light that reaches the detector would then be used to measure the quantity of heavy metals that have leached into solution.

The batch leaching was performed using varying fly ash and calcium stearate mixtures and adding the leachate of nitric acid and distilled water at a pH of 4.2 to 4.4. The pH was set at this level to mimic the worst-case scenario and cause the most leaching to occur from the fly ash mixtures. The amount of leachate was kept at a constant 50 mL and the liquid to solid ratio was altered using various amounts of fly ash and calcium stearate mixtures. After creating ratios of 100:1, 50:1 and 10:1, each with a different quantity of calcium stearate (0%, 1%, 2% and 3% by weight), the mixtures were shaken for 48 hours. Once the shaking was complete, the leachate was extracted from the surface with pipettes. Ten mL of the solution was mixed with cadmium, chromium, and lead reagents in a test tube and shaken for three minutes each to ensure a complete mixture. Each test tube was then placed in the spectrophotometer to measure the amount of heavy metal leaching that occurred. This process was repeated twice for duplicate testing.

The column leaching test was performed by first mixing the fly ash and calcium stearate mixtures with a specified amount of water to achieve optimum moisture content and maximum dry density. Once this was complete, the columns were then filled with each mixture in five equal lifts, compacting each with approximately 25 drops of a compaction hammer. The columns were then attached to a gravity head system as seen in Figure 1, with one inlet tube feeding leachate from a holding tank and one outlet tube to allow for the collection of the passed leachate. Based on the pore volume that was calculated to be one liter (Source needed), it was determined that each time one liter of solution passed through the column, it would be tested for the selected heavy metals. The data would then be input into the Yalcin Leaching Model in order to predict the leaching behavior for an extended period of time. Each pore volume was tested twice to provide duplicate results.

“A Yalcin leaching model was formulated to capture the observed experimental leaching behavior of the contaminant exhibiting an initial increase in concentration followed by a decrease in concentration with further leaching until it reaches a low steady-state concentration. The model is as follows.

$$C(t) = C_s - C_s \cdot e^{-Kk_{bt}} + C_0 \cdot e^{-Kk_{bt}}$$

Where k_b is the dissolution rate coefficient (min^{-1}), $K = (S/S_0)^a$, S = Solid phase concentration (mg/g), S_0 = Initial solid phase concentration (mg/g), C_s is the effective saturation concentration (solubility) of contaminant (mg/L), t is the time (min), and a is a dimensionless empirical constant” (Das 2007).

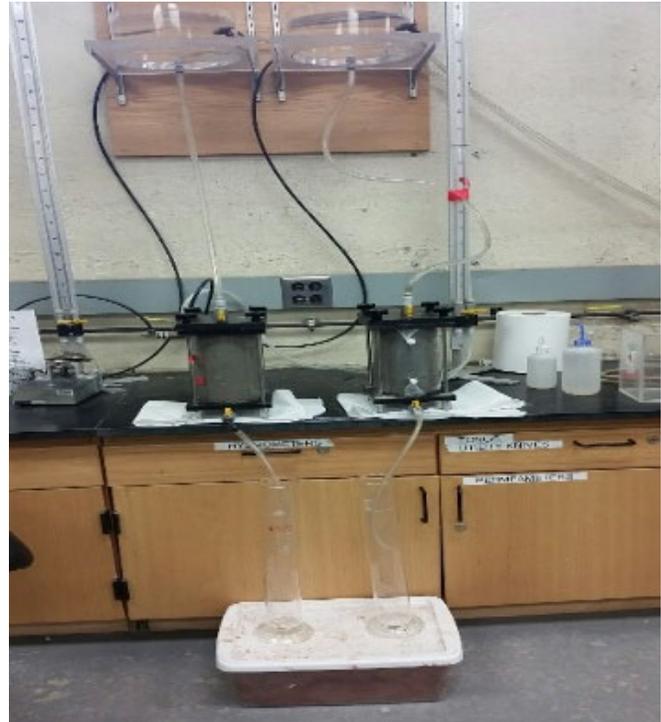


Figure 1. Gravity Head Column Leaching

4. RESULTS AND DISCUSSION

Tables 1 through 3 show the results of the batch leaching tests that were conducted on each L:S ratio containing 0-3% calcium stearate. For each metal, a general trend could be seen that the 1% calcium stearate mixture produces a constant value or a decrease in heavy metal concentration when compared to the plain fly ash solution, most notably for chromium. The same results could be seen in the graphical representation in Figures 2 through 4. The same general trend could be seen in this instance as well.

Table 1. Batch Leaching Average Concentration of Cadmium

Cadmium		
L:S Ratio	Calcium Stearate (% by weight)	Average Concentration (mg/L)
100:1	0	0.069
	1	0.072
	2	0.0697
	3	0.0655
50:1	0	0.0635
	1	0.068
	2	0.0715
	3	0.0815
10:1	0	0.0805
	1	0.0673
	2	0.0805
	3	0.1425

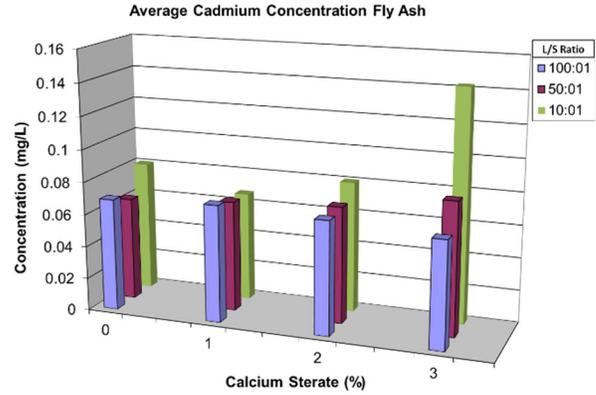


Figure 2. Batch Leaching Average Concentration of Cadmium

Table 2. Batch Leaching Average Concentration of Chromium

Chromium		
L:S Ratio	Calcium Stearate (% by weight)	Average Concentration (mg/L)
100:1	0	0.065
	1	0.0455
	2	0.0475
	3	0.0495
50:1	0	0.0685
	1	0.081
	2	0.0585
	3	0.051
10:1	0	0.119
	1	0.0495
	2	0.0835
	3	0.0925

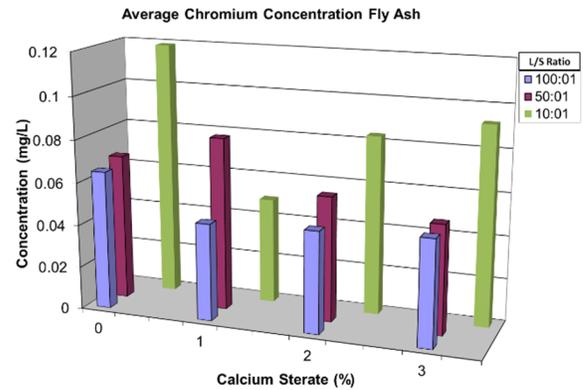


Figure 3 Batch Leaching Average Concentration of Chromium

Table 3. Batch Leaching Average Concentration of Lead

Lead		
L:S Ratio	Calcium Stearate (% by weight)	Average Concentration (mg/L)
100:1	0	0.1495
	1	0.155
	2	0.166
	3	0.155
50:1	0	0.1775
	1	0.1555
	2	0.1765
	3	0.181
10:1	0	0.135
	1	0.1545
	2	0.190
	3	0.3275

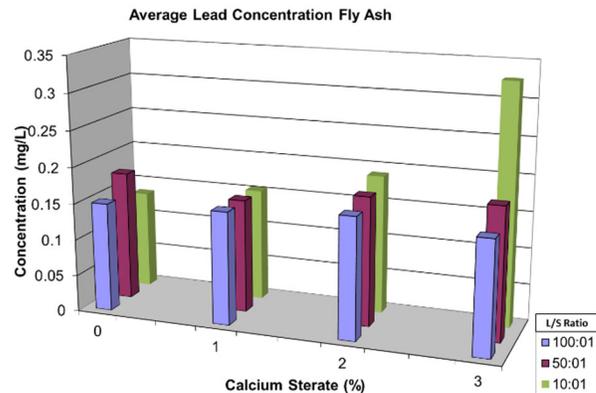


Figure 4. Batch Leaching Average Concentration of Lead

Table 4 shows the results of the column leaching tests based on the average values from each pore volume collected. These values vary widely due to the lack of information for the succeeding pore volumes which could not be collected due to time restraints. From the information gathered, as seen in Figures 5-16, it has been shown that the data collected will fit the predicted concentrations, but more data would be needed to create more accurate results.

Table 4. Column Leaching Average Heavy Metal Concentrations

Column Leaching Data			
Heavy Metal	Calcium Stearate (% by weight)	Average Concentration on PV1 (µg/L)	Average Concentration on PV2 (µg/L)
Chromium	0	197	259.5
	1	540.5	143.5
	2	478.5	144
	3	514.5	153
Cadmium	0	78.4	60.1
	1	93.4	63.25
	2	131	133.8
	3	166	137.8
Lead	0	189	138.5
	1	222	141
	2	309	310
	3	413	307

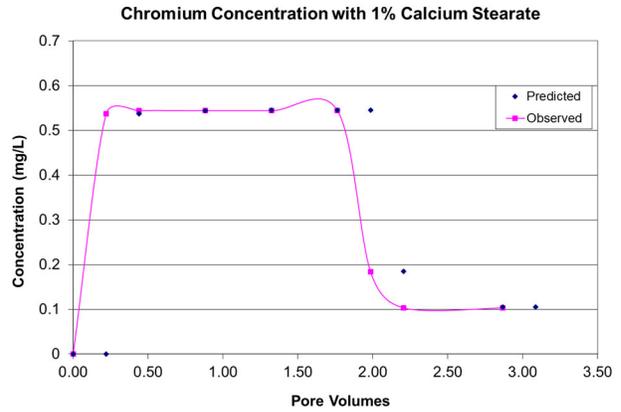


Figure 6 - Yalcin Leaching Model (1% C.S. - Chromium)

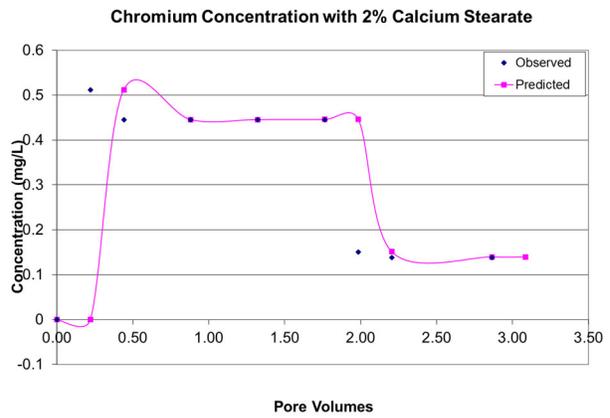


Figure 7 - Yalcin Leaching Model (2% C.S. - Chromium)

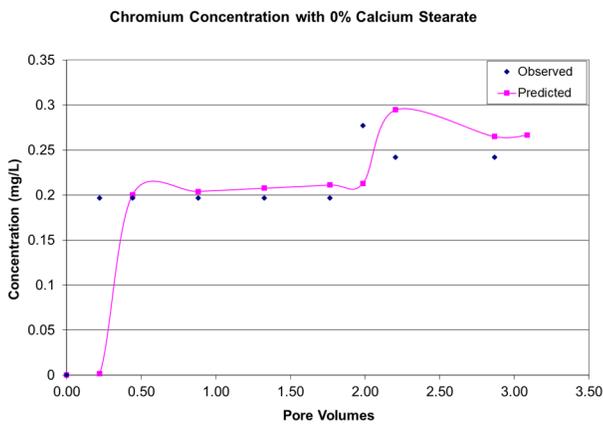


Figure 5 - Yalcin Leaching Model (0% C.S. - Chromium)

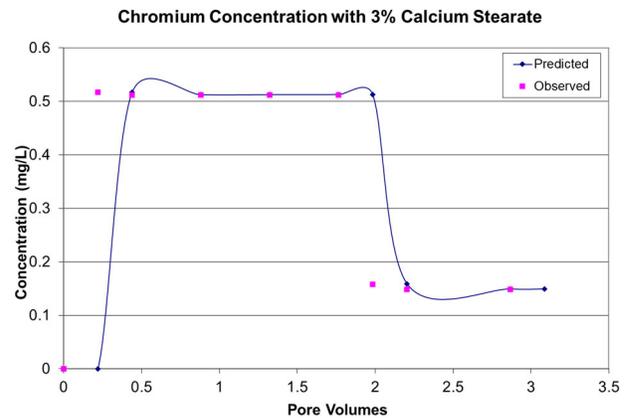


Figure 8 - Yalcin Leaching Model (3% C.S. - Chromium)

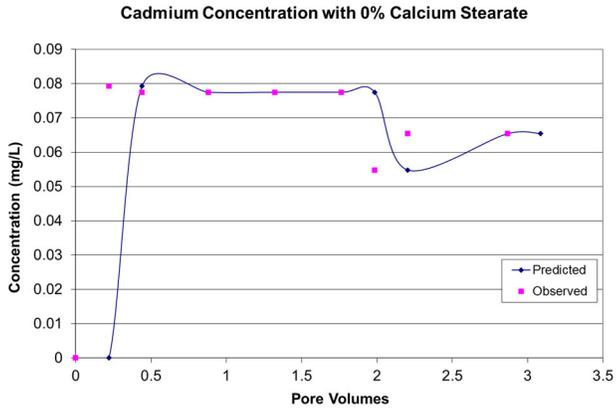


Figure 9 - Yalcin Leaching Model (0% C.S. - Cadmium)

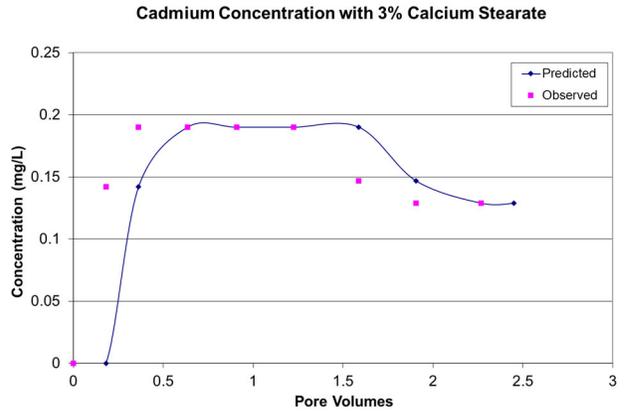


Figure 12 - Yalcin Leaching Model (3% C.S. - Cadmium)

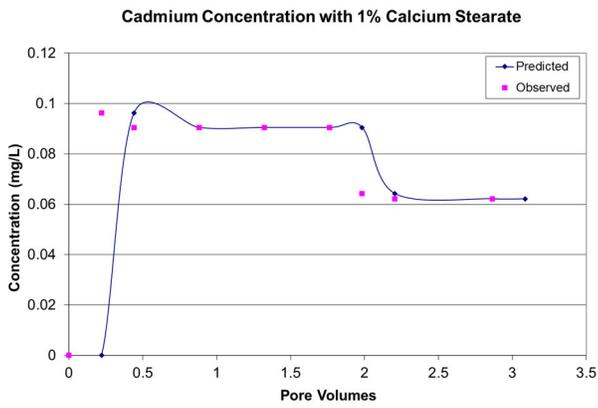


Figure 10 - Yalcin Leaching Model (1% C.S. - Cadmium)

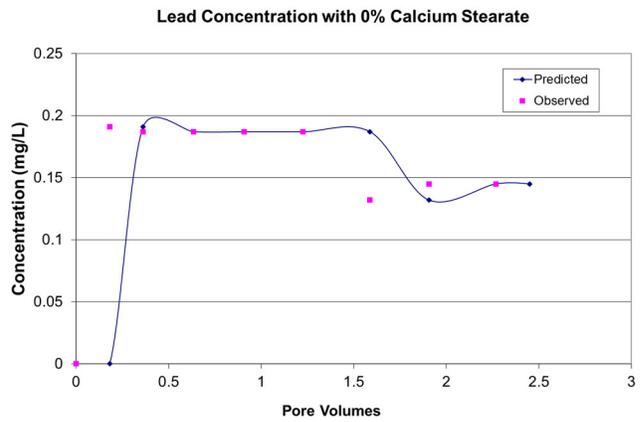


Figure 13 - Yalcin Leaching Model (0% C.S. - Lead)

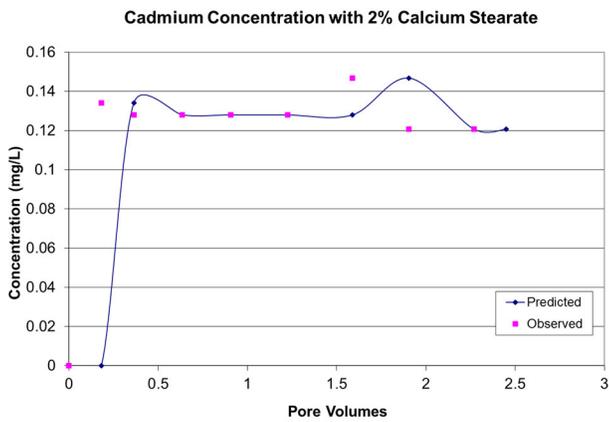


Figure 11 - Yalcin Leaching Model (2% C.S. - Cadmium)

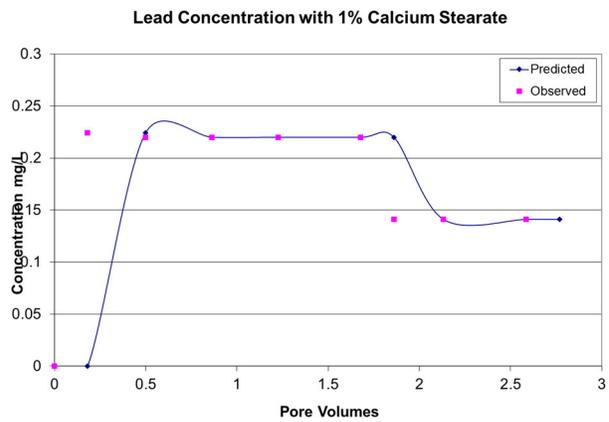


Figure 14 - Yalcin Leaching Model (1% C.S. - Lead)

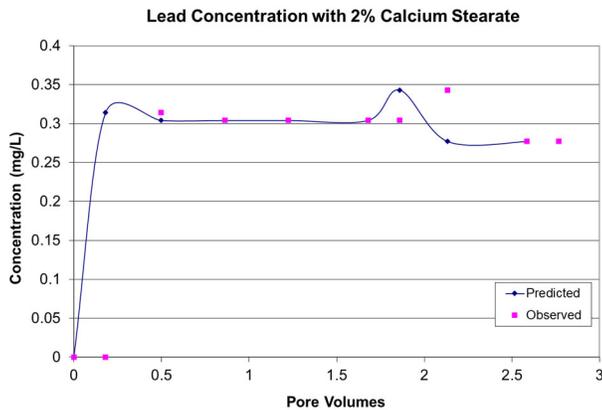


Figure 15 - Yalcin Leaching Model (2% C.S. - Lead)

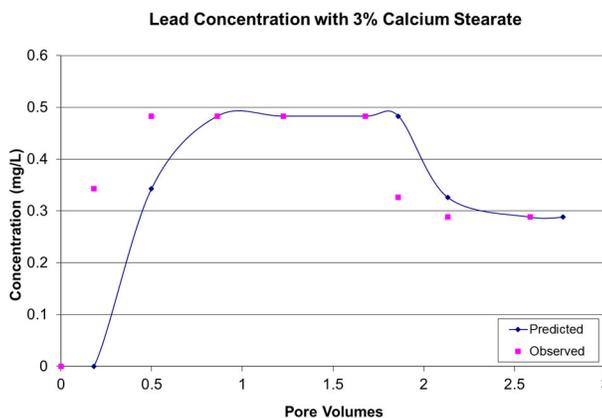


Figure 16 - Yalcin Leaching Model (3% C.S. - Lead)

After final review of all the compiled data, the batch leaching and column leaching data generally indicate that by adding 1% calcium stearate to fly ash, the overall concentration of leached heavy metals, Chromium, Cadmium, and Lead, was reduced by the greatest amount when compared to the 0%, 2%, and 3% mixtures. At higher concentrations of calcium stearate, a trend of increasing concentrations of leached metals was observed in both testing methods.

The Yalcin leaching model was used as a numerical modeling method rather than an analytical method due to time constraints and the limited pore volumes that we're able to be obtained. This does not discredit the data obtained, instead, it shows that the recorded results follow a predicted trend. This trend could become more accurately depicted if subsequent pore volumes were collected and tested allowing for precise analytical modeling to predict leaching behavior for months or even years in the future.

Since calcium stearate is a molecule consisting of a central calcium ion with two stearate groups attached, it could be considered a scavenger additive. The stearate group is essentially a long-chain carbon molecule, making it similar in nature to polymer chains. Using the chain, calcium stearate will attack the transition metals, found in the form of metal chlorides by the following reaction



where $\text{Ca}(\text{O}_2\text{C}_{18}\text{H}_{37})_2$ is the calcium stearate, MCl_2 is the metal chlorides (Cr, Cd, and Pb), CaCl_2 is calcium chloride and MSt_2 which is the new metal stearate chain (Equistar n.d.). The calcium chloride is an inactive chemical and the new metal stearate is the heavy metal that combines with the long stearate chains, effectively encapsulating the contaminant.

During chemical bonding, there is only a certain amount of bonds that could be made and electrons that could be transferred. This being the case, it was determined that the 1% calcium stearate addition provided the optimum conditions in terms of bonds available. When additional quantities of calcium stearate were added, there was an excess of electrons that were available to bond creating a poorer performance of the additive.

Due to limited data, future research to be conducted will include triplicate testing for both the column and batch leaching tests. Furthermore, numerous additional pore volumes would be collected to more accurately predict the contaminant leaching behavior using the Yalcin leaching model. Other options include testing for more heavy metals or using different leaching models such as the Van Genuchten leaching model.

5. ACKNOWLEDGMENTS

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