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An Introductory Study of Coal Fly Ash Enhancement for Heavy Metal Removal in Wastewater
Treatment

By

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B.A. University of Louisville, 2020

A Thesis

Submitted to the Faculty of the

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in Chemical Engineering

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Removal in Wastewater Treatment

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DEDICATION

This thesis is dedicated to my parents

Mr. Timothy Romes

and

Mrs. Janelle Romes

who have always supported me and given me the opportunity to become the best version of myself.

ACKNOWLEDGMENTS

Thank you to the Gupta Research Labs team especially: Dr. Gautam Gupta, Dr. Mahyar Ghorbanian, Joe Cahill, Luis Moreno, Adam Flynn, Amanda Arts, Evan Grimm, and many others.

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Finally, thank you to my parents for their support. Achieving this level of education would not have been possible without them.

ABSTRACT

An Introductory Study of Coal Fly Ash Enhancement for Heavy Metal Removal in Wastewater Treatment

Samuel T. Romes

September 21, 2021

Coal Combustion Products are the materials that remain after pulverized coal is burned to generate electricity. The fine portion, fly ash, has many re-use capabilities across various industries. Most recent data shows that approximately 60% of fly ash produced in the United States was used with the largest re-use being concrete additives. This work aims to enhance fly ash via hydrothermal treatment in order to expand the re-use capabilities of the material. These materials were tested as adsorbents for heavy metal removal from wastewater samples. The metals tested for removal were Copper (II) Acetate, Cadmium Chloride, Boric Acid, Sodium Selenite, and Mercury (II) Chloride.

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1 Introduction

In 2019, over 29 million short tons of fly ash were produced in the United States. Forty percent, or approximately 12 million tons of fly ash were left to waste and be landfilled [1]. The two main types of fly ash are Class C and Class F fly ash. Class F Fly ash is composed of greater than 70% SiO_2 , Al_2O_3 , and Fe_2O_3 and is the focus of this study.

Coal Fly Ash recycle is important to eliminate a waste stream resulting in less usage of the landfill space and can provide value-added products which have environmental impacts of their own. One major re-use path for fly ash is concrete. Not only does coal fly ash provide decreased permeability, increased long term strength, reduced damage from heat of hydration, and increased resistance to sulfate and other chemical attacks; it also replaces materials which would be mined to manufacture cement resulting in more CO_2 and Greenhouse Gas Emissions. Therefore “for each ton of fly ash used in place of traditional cement a reduction of slightly less than one ton of carbon dioxide is achieved.” [2]

Zeolites are natural or synthesized crystalline aluminum-silicates which have various applications in the fields of Chemistry and Chemical Engineering such as adsorption and catalysis [3]. Much like the way fly ash has become a sustainable supplemental cementitious material, it is possible that fly ash can be enhanced to become a sustainable (and cost-effective) adsorbent. Fly Ash is a promising path to zeolite creation because of their naturally porous bodies and much lower raw material cost. This is an interesting proposal because coal power plants could recycle their waste fly ash for treatment of their wastewater – both eliminating landfill space and cleaning their effluent streams in the same act.

Hydrothermally treating coal fly ash has been studied extensively. Querol et al. studied the synthesis of thirteen different zeolites from eleven types of ash. His work focuses upon reaction conditions used in this paper such as molarity, solid to liquid ratio, and reaction temperature. It also

acknowledges zeolites as having a high potential use in water decontamination [4]. Wang et al. studied the metal adsorption capacity of class F coal fly ash. Through performing batch titrations, it was determined that metal uptake of Nickel, Chromium, Copper, Lead, and Cadmium increases as pH increases [5]. Margeta et al. studied natural zeolites as well as chemically treated zeolites and their efficacy in water treatment. She found that chemical pretreatment resulted in significant increases in adsorption capability, up to 71% increase in some cases. She also reported adsorption efficiency for zeolites across different methods of wastewater treatment [6]. What do you get when you put these studies together? A strong case for a cost-effective, waste eliminating product that has the potential to make a large impact in wastewater treatment.

The Environmental Protection Agency (EPA) is constantly updating the Code of Federal Regulations and Priority Pollutants list for industrial wastewaters. For coal fired power plants in particular, the main regulations of interest are the safe disposal of Coal Combustion Residuals (CCR) and meeting the treatment goals of Effluent Limitation Guidelines (ELG) [7, 8]. CCR regulations address the risks associated with coal ash disposal such as contaminants leaking into ground water [9]. ELG set forth by the EPA highlight priority pollutants such as mercury, selenium and total suspended solids including copper, cadmium, chromium, boron, etc [10]. Clearly, both the disposal of coal combustion products and the treatment of effluent wastewaters are challenging. Coal fly ash has the potential to solve both problems at once.

We address these challenges by studying the impact of coal fly ash as a starting material which can be used for effective wastewater treatment of common plant effluent solutions such as mercury, selenium, copper, cadmium, and boron. The results were promising, and we present them in this paper.

2 Materials and Methods

2.1 Laboratory Reagents

The hydrochloric acid utilized was ACS Grade (36.5-38%) from VWR Chemicals. Water was twice deionized to a resistance of 18.2 MΩ-cm. Sodium hydroxide pellets were reagent grade from VWR Chemicals. All metals utilized to create mock wastewater solutions (Sodium Selenite, Boric Acid, Copper (II) Acetate, Cadmium Chloride, Mercury (II) Chloride) were high purity grade chemicals from VWR Chemicals.

2.2 Coal Combustion Products

The coal combustion product utilized was coal fly ash. Coal fly ash was received from a Southeastern United States powerplant burning Illinois Basin Fuel. The coal is typified as being a high sulfur and chloride fuel. The ash was classified as Class F Fly Ash. It is possible that additives such as hydrated lime and activated carbon were present in trace amounts, but not enough information was given by the supplier to make a determination on this subject.

2.3 Mock Wastewater Solutions

Wastewater mock solutions were created in lab. Stoichiometric amounts of heavy metal solids were added and mixed with one liter of twice deionized water under constant stirring to create the following solutions: 15 ppm Boric Acid, 15 ppm Copper (II) Acetate, 15 ppm Sodium Selenite, 15 ppm Cadmium Chloride, 5 metal solution – 15 ppm: Sodium Selenite, Boric Acid, Potassium Dichromate, Copper (II) Acetate, Cadmium Chloride, and finally 6-metal solution – 15 ppm: Mercury (II), Sodium Selenite, Boric Acid, Potassium Dichromate, Copper (II) Acetate, Cadmium Chloride

2.4 Equipment

The incubator used for drying was a Fisher Scientific Isotemp Incubator and the temperature of the incubator was held at 50 degrees Celsius. The centrifuge used to separate solution from sample was a Fisher Scientific Marathon 3200R Benchtop Centrifuge. The centrifuge was run at 4500 RPM for 30 minutes and the temperature was set at 25 degrees Celsius. The pH and ORP meter used was a VWR Traceable pH/ORP meter. This instrument was listed as ideal for wastewater applications.

2.5 Fly Ash Hydrothermal Treatment

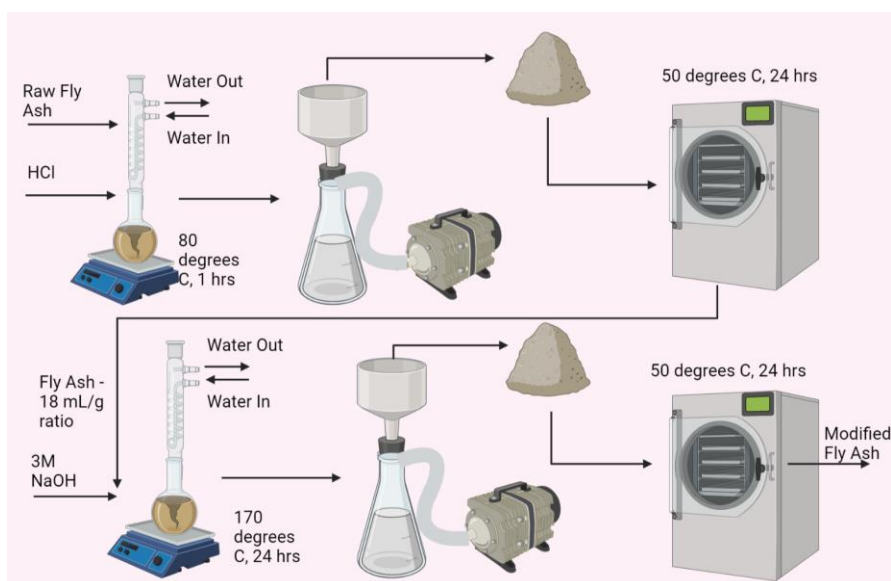


Figure 1. Process outline for the modification of fly ash through two step hydrothermal treatment.

Coal Fly Ash was first washed by 36% HCl at 80 degrees Celsius for 1 hour. The ratio of acid solution to ash was 25:1 [11]. The ash was then vacuum filtered from solution and washed with deionized water. The ash sample was dried at 50 degrees Celsius for 24 hours. The dry ash was then added to 3M NaOH solution and refluxed at 170 degrees Celsius for 24 hours. The ratio of alkaline solution to ash was 18 mL/g [4]. After reflux, the ash was filtered from solution, washed with deionized water, and

dried at 50 degrees Celsius for 24 hours. The dried product was then used for testing in heavy metal removal of wastewater solutions.

An additional point of discussion related to the hydrothermal treatment method, is related to waste. High solution to ash ratios would potentially create a large amount of difficult waste. To combat this, the Gupta Research Labs team has begun research centered around ferric chloride recovery from the first step of the hydrothermal treatment process. Additionally, this pathway could lead to a value-added product which improve the economic viability of this project on a large scale. Ferric Chloride has a current market value near \$320 per ton and can be used as flocculants, coagulants, odor sequestration, and catalysts for the production of vinyl chloride.

2.6 Adsorbent Testing

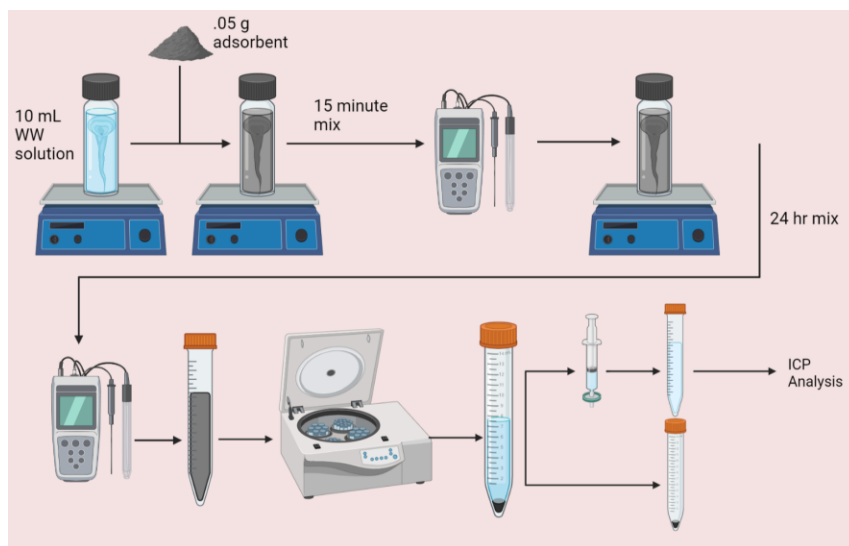


Figure 2. The lab-scale batch process used to test adsorbent efficacy.

Adsorbent efficacy was tested through a batch method. Mock wastewater solutions (10 mL) and adsorbents (.05 grams) in a were mixed for fifteen minutes before pH and ORP readings were recorded. The batch mixture was then allowed to mix for 24 hours. After the 24-hour mix, a second pH and ORP reading was recorded. The mixture was finally centrifuged, syringe filtered, and stored for ICP analysis. These experiments were performed in triplicates.

2.7 Analytical Methods

Material characterization was performed using three main analytical techniques: Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), and BET Surface Analysis. Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) was performed to measure the percent removal of heavy metal content within mock wastewater solutions.

2.8 Scanning Electron Microscopy

Scanning electron microscopy was a technique used to analyze the surface topography and crystalline or porous structure of the materials [12]. SEM comparisons between raw fly ash and enhanced fly ash gave a visual indication of whether or not the process was successful. The equipment used for SEM imaging was a TESCAN Vega3 SEM.

2.9 X-Ray Diffraction

X-Ray Diffraction was utilized to identify the formation of crystalline materials [13]. The equipment used was a BRUKER Discovery D8 HR-XRD. Samples were scanned through the 2θ range of 10 to 80 at a scan speed .5/second and an increment of .02/second.

2.10 BET

Brunauer-Emmett-Teller theory was utilized to analyze the surface area, pore volume, and pore size of each sample [14]. The equipment used was a Micromeritics TriStar 3000 – automated gas adsorption analyzer using BET. Approximately 0.2-0.3 grams of sample were degassed at 120 °C for 120 minutes before undergoing BET analysis.

2.11 ICP-AES

Heavy metal adsorption was analyzed using an IRIS Intrepid II XSP Inductively Coupled Plasma Atomic Emission Spectrometer, ICPAES. Mercury detection required the use of an HGX-200 advanced membrane Cold Vapor (CV) attachment which enables the reduction of Hg^{2+} (liquid phase) in the sample to ground-state mercury Hg^0 (gas phase). The sample and Sn(II)Cl are pulled by the peristaltic pump into the mixing block where the reduction takes place. The mixture then travels to the gas liquid separator where the liquid drains while the gas is pushed through the separator by the carrier gas, Argon, into the ICPAES for the quantitative analysis. Percent removal was determined by comparing the signals of blank-corrected untreated mock wastewater samples to mock wastewater samples which had been treated by adsorbents.

3 Results and Discussion

3.1 SEM

Scanning electron microscopy was used to highlight physical differences in the surface structure of coal fly ash before and after hydrothermal treatment. Figure 3 highlights surface differences between Raw Fly Ash and Fly Ash which has undergone hydrothermal treatment. Figure 3A shows a relatively uniform sample with circular-like structures occupying the space. Figures 3B and 3C show samples with little to no uniform structure; rather, they look to have an amorphous, porous nature to them.

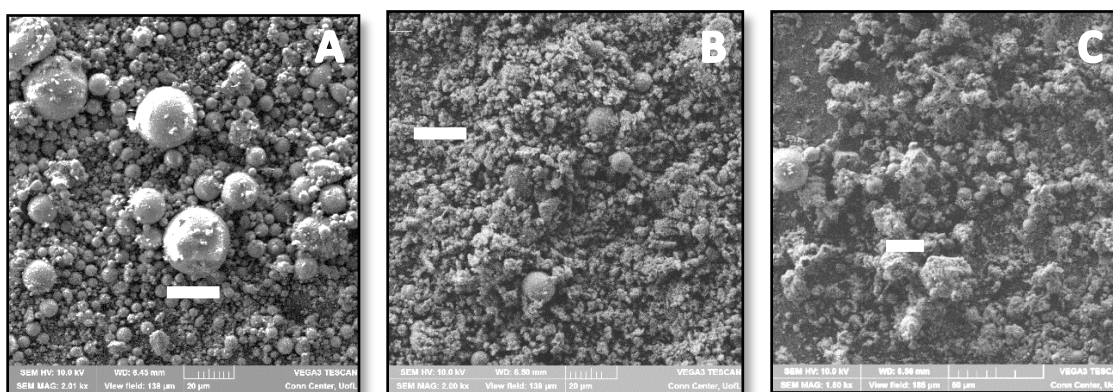


Figure 3. SEM Image comparisons of Raw Fly Ash (A), NaOH Reflux Fly Ash (B), and HCl Wash + NaOH Reflux Fly Ash(C).

3.2 XRD

All three adsorbents were analyzed using XRD. Figure 4 shows the results of the XRD analysis. Raw fly ash is composed mainly of quartz, magnetite, hematite, and mullite. Hydrothermal treatment introduces peaks indicative of zeolite X for both fly ash which has undergone an NaOH reflux and fly ash which has undergone a HCl wash and NaOH reflux [15] [16] [17].

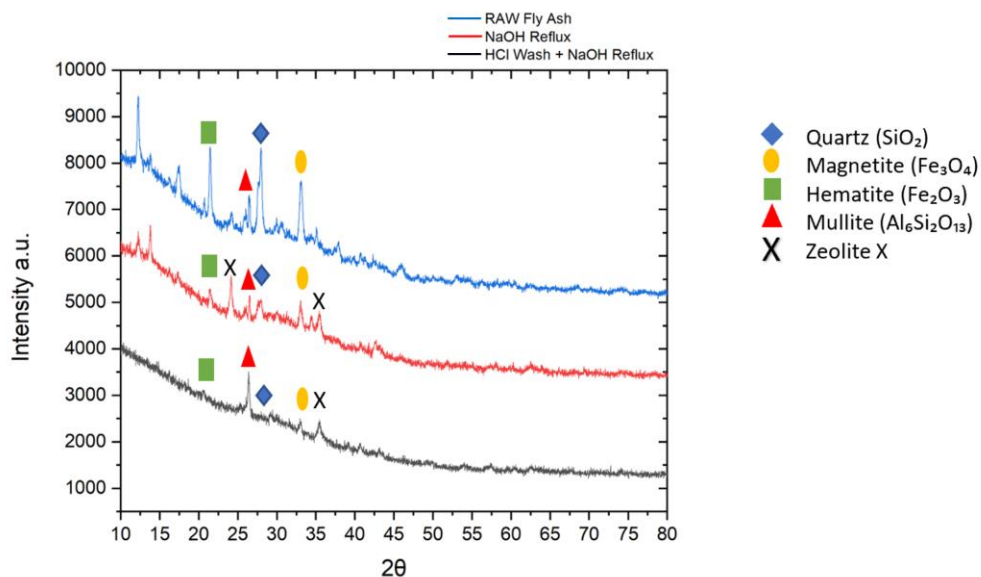


Figure 4. XRD Comparisons of Raw Fly Ash, NaOH Refluxed Fly Ash, and HCl Wash + NaOH Refluxed Fly Ash.

3.3 BET

BET analysis provided a quantitative measurement for three metrics: surface area, pore volume, and pore size. The working theory was that a higher specific surface area would facilitate a higher level of heavy metal adsorption due to an increase in adsorption sites. Pore volume and pore size were two factors which were considered as well.

Table 1 – BET data for all adsorbents used in batch experimentation.

Adsorbent	Surface Area $\frac{m^2}{g}$	Pore Volume $\frac{cm^3}{g}$	Pore Size <i>nm</i>
Raw Fly Ash	2.7613	.007305	10.582
NaOH Reflux	19.3511	.070936	14.663
HCl Wash + NaOH Reflux	23.9589	.020743	3.461

3.4 Heavy Metal Adsorption

Cadmium Chloride, Sodium Selenite, Mercury (II) Chloride, Boric Acid and Copper (II) Acetate solutions were treated with the adsorbents created in lab. To test the heavy metal removal efficacy of the adsorbents, experiments were run as described in the Materials and Methods section. These samples were then tested through ICP Analysis. Percent heavy metal removal was calculated by comparing blank or “untreated” solutions” against those which had adsorbents added.

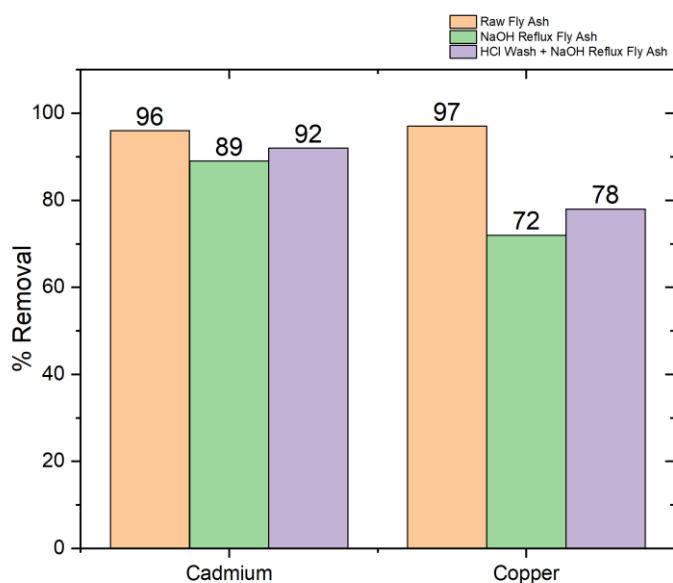


Figure 5. Cadmium and Copper removal as a function of adsorbent.

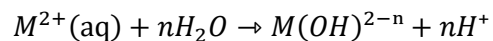
Cadmium and Copper ions were successfully removed by fly ash. Cadmium and Copper are both a 2+ heavy metal ion. The operating pH range for Cadmium and Copper removal was between 8-9. Alkaline conditions are favorable for Cadmium and Copper removal because an increase in pH causes the surface of the fly ash particle to become more negatively charged and therefore cationic ion adsorption increases [18].

At an elevated pH, Cadmium exists in the form of $\text{Cd}(\text{OH})^+$, $\text{Cd}(\text{OH})_2$ and $\text{Cd}(\text{OH})_3^-$. In this state, the cadmium ion is stable and can be chemically adsorbed at SiO_2 sites [19]. At an acidic pH, cadmium exists as the much less favorable Cd^{2+} and Cadmium removal is not favorable.

Copper can be removed from solution through electrostatic adsorption or precipitation. Electrostatic adsorption is the most common and occurs when the surface of the fly ash adsorbent carries a negative charge and attracts the surface of the positively charged copper ion. Precipitation occurs when the hydrated silicon oxide of the coal fly ash complexes with the copper ions via chemical bonding [20]. At alkaline pH, Copper exists as CuO and CuOH^+ . The pH of all three solutions was greater than 8 after 24 hours of mixing and therefore Copper was successfully removed from solution.

Harja et al. studied copper removal by low cost adsorbents obtained from ash [21]. They achieved greater than 97% removal and found that the low adsorption capacity at pH values less than 4 could be attributed to hydrogen ions that compete with metal ions for adsorption sites. At pH values greater than 6, they found copper ions precipitate as its hydroxide and could be both adsorbed and precipitate out of solution.

Cho et al. studied both copper and cadmium removal [18]. They showed over 95% removal of both elements at, or above a pH of 8. The following equation describes the manner in which metal cations hydrolyze in aqueous solutions.



Cho also found that copper removal above pH 6 was a function of both precipitation and adsorption, but adsorption dominated. Finally, general trends from Cho's work showed increased removal at higher pH's which were achieved by increasing heavy metal concentration or decreasing fly ash dosage.

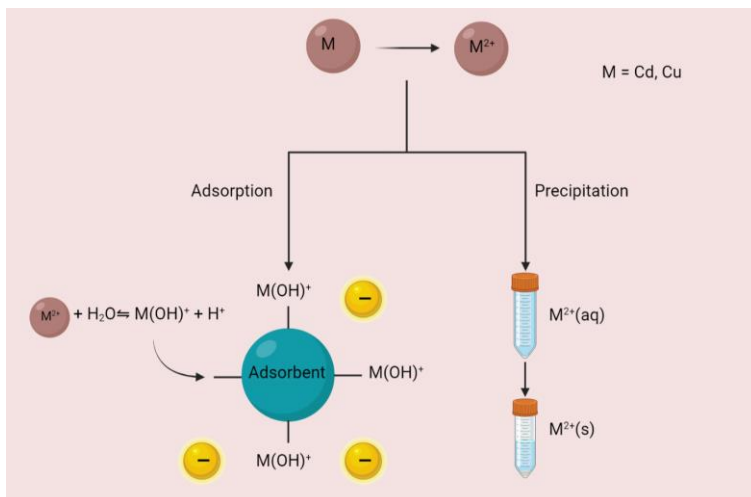
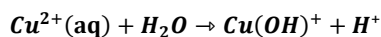
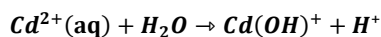


Figure 6. Visual representation of the mechanisms, adsorption and precipitation, of removal for Cadmium and Copper following equations of the form:



Selenium, Mercury, and Boron were also investigated. Heavy metal removal for each adsorbent can be seen in Figure 7.

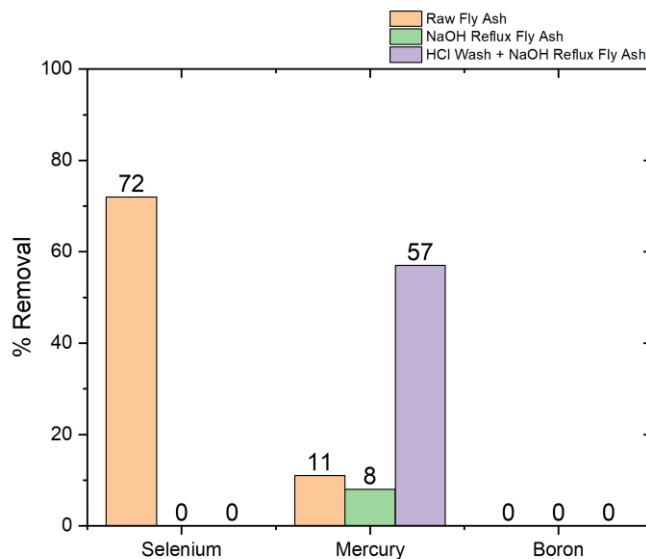
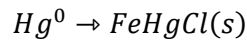


Figure 7. Selenium, Mercury, and Boron Removal as a function of adsorbent.

Sodium Selenite (Na₂SeO₃) mock wastewater solution contains Selenium as an ion of charge 2+. Maximum removal of selenium was found in the pH range of 2-3 where Selenium mainly exists in the

form HSeO_3^- and H_2SeO_3 [22]. In alkaline conditions above pH of 9, selenium samples exist as SeO_3^{2-} , where selenium removal is less favorable. Raw fly ash, operating a pH near 4, was able to successfully remove 72% of the selenium in solution. Both “NaOH Reflux” fly ash and “HCl Wash + NaOH Reflux” fly ash operated at a pH range near 9 and were unsuccessful in removing Selenium from solution.

For mercury (II) ions, fly ash treated by “HCl Wash + NaOH Reflux” performed the best achieving a removal of almost 60%. This occurred at a pH of 8.96. Mercury removal appears to be favorable at lower pH. Using Coal Fly Ash Zeolite A, Attari et al. achieves upwards of 90% removal [23]. This reaction is carried out near a pH of 2.5 and 25 degrees Celsius. One reason the HCl Wash + NaOH reflux was so successful at an alkaline pH may be due to the catalytic activity of Fe_2O_3 in the presence of hydrochloric acid. Kuncoro et. al. reported the following mechanism for Mercury removal [24].



It is likely that significant removal (upwards of 90%) could be achieved using “HCl Wash + NaOH Reflux” fly ash at an acidic pH.

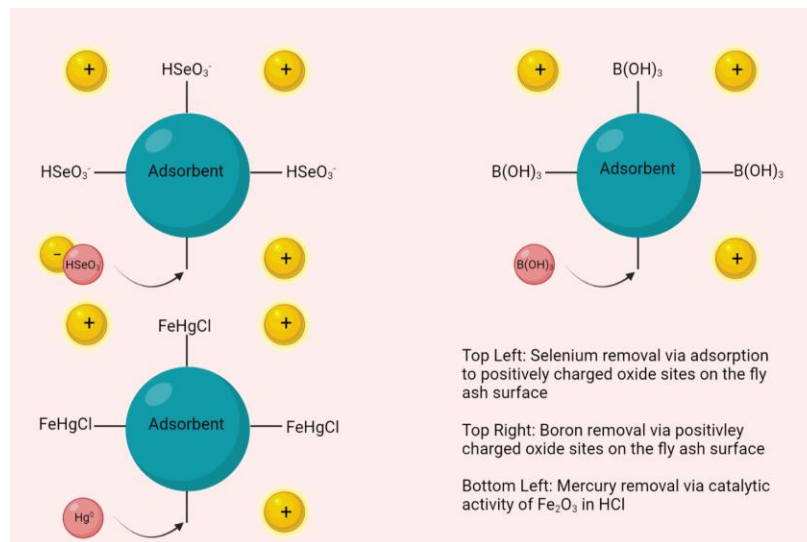


Figure 8. Visual representation of the mechanism of removal for Selenium, Mercury, and Boron.

Boron was not successfully removed. Kavak et. al. found that the borate anion was absorbed best at a highly acidic pH [25]. The mechanism of adsorption relates to oxides in the fly ash forming aqua complexes in solution and hosting positively charged surfaces. These positively charged surfaces are suitable for Borate Removal. If hydrochloric acid was used to adjust pH, the surfaces become even more favorable as chloride ions on the fly ash surface can be exchanged for borate. It is likely that “HCl Wash + NaOH Reflux” would achieve the highest boron removal if run in solution with a highly acidic pH.

4 Conclusions

Fly ash was hydrothermally treated via sodium hydroxide reflux as well as hydrochloric acid wash and a sodium hydroxide reflux in order to enhance fly ash adsorption capabilities. Raw Fly Ash was tested against both adsorbents. Successful heavy metal removal across all three adsorbents was seen for Cadmium and Copper. Mercury and Selenium, while not totally removed across all three adsorbents, showed trends which may allow for future research to optimize removal. Surprisingly, the raw fly ash performed the best of the three adsorbents which does not make a strong case for the additional time and energy used. These samples all saw successful removal at their natural, high pH. Selenium, Mercury, and Boron removal should be investigated in solution with highly acidic conditions.

This introductory study identified and investigated a potential recycle path for the 12 million tons of coal fly ash that go to landfill each year. Raw fly ash and hydrothermally treated fly ash both successfully removed cadmium and copper ions from mock wastewater solutions. Selenium, Mercury, and Boron all show potential for success and should be investigated further. If optimized, fly ash has the potential to become a cheaper alternative to current wastewater adsorption technology.

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