

# Fluidized Bed Combustion Fly Ash Leaching: A Column Study

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**Keywords:** Fluidized Bed Combustion, fly ash, leaching, column study, SEM/EDS, QXRD

**Abstract:** Pennsylvania is a leader in utilizing coal wastes such as anthracite culm and bituminous coal gob in fluidized bed combustion (FBC) boilers. Alkaline FBC fly ash could contain large proportions of calcium and sulfur bearing minerals. Although FBC ashes typically do not meet ASTM C618 due to high SO<sub>3</sub> and loss on ignition (LOI), they have the potential to be beneficially used in filling and reclamation of mine land features. However, leaching of harmful elements from ash deposits and consequent groundwater contamination is an environmental concern. This study assessed the leaching behavior of Schuylkill FBC ash monofills exposed to neutral water flow in a column scale. The assessment included characterizing the coal ash (via scanning electron microscopy and X-ray diffraction), and flow-through leaching test in a 30cm column. In comparison with pulverized coal ash, the studied FBC ash contained higher clay and quartz and lower glass content, with 8% LOI. Within 8 days, alkalinity of fly ash raised the pH from 5 to 11. Under neutral inflow condition, Si, Al, and Fe concentrations slowly decreased, while Mg content was almost constant. Calcium and Sulfur contents decreased by a factor of 2 and Na and K contents dropped by a factor of 3 to 5. Among trace elements, molybdenum, associated with S and Ca, decreased from 1.30 to 0.08 mg/L, while Se, and As contents (associated with clay particles) were almost constant during 8 days. It was found that all detected trace elements were below Pennsylvania maximum acceptable leaching limits.

## Introduction

Fluidized bed combustion (FBC) in Pennsylvania has been considered a primary technology to combust mining refuse from anthracite and bituminous coals. FBC boilers burn low BTU anthracite culm and bituminous coal gob, which contain higher concentrations of pyrite (FeS<sub>2</sub>). To compensate for the high sulfur content in the fuel and lower the SO<sub>x</sub> emissions, limestone is added to the bed as a sulfur capture mechanism. The rock in the coal mining refuse mainly contains clay minerals with minor amounts of quartz and carbonates. The combustion temperature in FBC system is set

high enough to decompose the added limestone at approximately 800°C. At this temperature, clays lose pore water and hydroxyl groups [1]. Although such temperature is not high enough to fully melt the clay structure into a glass, in lack of crystallinity, the dehydroxylated clays become chemically reactive and x-ray amorphous.

In addition to the decomposition of clays, limestone is decomposed to lime and carbon dioxide. Amount of limestone is usually much greater than stoichiometric requirements for formation of alkaline sulfates. Therefore, FBC ash consists of spent and unspent amounts of sorbent [2]. The excess lime can act as an activator in presence of water to produce alkaline pH. At pH about 12.45, dehydroxylated aluminosilicate network begins to dissolve, and hydrates into C-H-S gel giving cementitious properties to FBC ash. Anhydrite is formed as the desulfurization byproduct is removed from the flue gas stream. It reacts with moisture to form gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). Formation of ettringite from gypsum and dissolving clay is a second reaction, which cause expansion if it is formed in high amounts. Ettringite is an important component in the cement formulation process. High amounts of sulfur promotes formation of ettringite, which can sequester heavy metals such as arsenic.

FBC ash does not typically meet ASTM C618 requirements for use in concrete mainly due to high level of sulfate and free lime [3]. However, it has been extensively used to reclaim active and abandoned mine lands. The growth in use of coal ash in mine reclamation was a result of regulatory enhancement from 1986 through 2004. Regulations for coal ash analysis parameters, concentration limits, surface and groundwater monitoring, and geologic and hydrologic evaluations were defined by Pennsylvania Department of Environmental Protection (DEP)[4]. Acid mine drainage (AMD) is the most serious stream pollution problem in Pennsylvania. Waste coal can leach highly concentrated AMD and may include elevated levels of trace elements such as arsenic, lead, copper, and chromium. FBC facilities remove abandoned coal refuse banks, mix the culm with limestone, and produce an alkaline ash, which can be used to reclaim active and abandoned mine lands. Several environmental benefits are associated with this process including removal of acid-producing culm banks, backfilling of abandoned mine lands (AMLs), and reduction of acid mine drainage. However, for use of coal on mine sites, it must meet maximum acceptable leachate limits for components such as aluminum, iron, arsenic, cadmium, calcium, chloride, barium, boron, chromium, copper, lead, magnesium, molybdenum, mercury, nickel, potassium, selenium, sodium and zinc in groundwater monitoring wells.

Pennsylvania defines four uses of coal ash on active mines as beneficial uses: alkaline addition, low permeability material, soil substitute/additive, and placement. To qualify for use as an alkaline addition agent, the ash should have a neutralization potential of at least 100 parts per thousand and a pH between 7.0 and 12.5. In coal ash monofills, a laboratory proctor density test must be performed to define the theoretical maximum dry density and optimum moisture content, which allows adequate on-site compaction. Chemical characteristics of coal ash leachate, and groundwater quality are evaluated by

DEP through Synthetic Precipitation Leaching Procedure (SPLP) test and groundwater monitoring points in the placement sites [5]. Batch leaching tests (e.g., TCLP, SPLP, and leaching test as ASTM D4793) used to assess the general leaching risk of elements from fly ash, are performed over a short durations (e.g.,  $18 \pm 2$  hr for TCLP), and their chemical conditions do not adequately mimic that of compacted fly ash deposits in mine sites. They attempt to consider the worst-case situation (e.g., pH of  $2.88 \pm 0.05$  in TCLP) to simulate acidic conditions of mine sites, but low pH is not the worst condition for leaching of all elements. For example, As leaches out more significantly at higher pH [6]. Also, aggressive stirring or agitation and using low volumetric solid to liquid ratios (e.g., 1:20 in TCLP) during the test are far from in-situ mine site conditions, where ash layers with high solid to liquid+void ratio (e.g., 1:0.7) are in contact with water flow under gravity. Column leaching tests have been considered to better simulate the water passing through compacted layers of ash in a mine field [7].

The objective of this study is to evaluate the leaching behavior of environmentally important elements from FBC ash using a flow-through column test. Such system can simulate the exposure of ash monofills to rain water flow in a more realistic condition. The chemical composition of FBC ash (e.g., mineralogy and alkalinity) and how it may affect the release of major, minor, and trace elements from ash deposits is evaluated. The results of this study will contribute to the environmental assessment of mine land reclamation using coal ash byproducts.

## **Materials**

FBC fly ash from Schuylkill Energy Resources' power station in Shenandoah, PA, was analyzed in this study. This ash is a product of burning anthracite culm and bituminous gob. A bulk sample of approximately  $0.2 \text{ m}^3$  was acquired from the power plant. After thoroughly mixing the as-received fly ash, test specimens were acquired using a cone and quarter method to obtain a representative sample from the bulk. Figure 1 depicts the huge ash deposits in Mahanoy Township, Schuylkill County.

## **Methods**

The fly ash was characterized for its physical properties, including moisture content, particle size distribution (using laser diffraction), density (using helium pycnometry), and surface area (using  $\text{N}_2$  gas adsorption). Organic carbon content was measured using loss on ignition (LOI) according to ASTM D7348, and two-atmosphere thermogravimetric analysis technique developed in a separate study [8]. Bulk chemistry of FBC ash was quantified using X-ray fluorescence (XRF), and also by total digestion of the ash, followed by performing inductively coupled plasma atomic emission spectroscopy (ICP-AES). Total sulfur content was determined through ASTM D5016 using infrared (IR) detectors to measure the concentration of emitted  $\text{SO}_2$  gas [9].



Figure 1- FBC ash deposits in Mahanoy Township, Schuylkill County, PA

Total content of trace elements was determined using ASTM D6357 method [10]. This method is used specifically for combustion residues from coal utilization processes. Environmentally available content of trace elements in fly ash was determined using EPA 3050b method, which is an acid digestion technique followed by ICP-AES or ICP-MS to determine the composition of sediments and soil. This procedure is not considered as a total digestion technique for fly ash. It can dissolve almost all elements that could become environmentally available. Therefore, elements bound in silicate structures are not totally dissolved by this procedure.

To characterize the morphology and elemental distribution within fly ash particles, FEI Quanta 200 Environmental Scanning Electron Microscopy (ESEM) was used. To prepare the polished samples, the fly ash was mixed with a low viscosity epoxy resin and cast inside 5mm diameter holes that were drilled into an epoxy puck. After the epoxy had set, the surface was polished with abrasive grits and diamond paste down to 0.25  $\mu\text{m}$ . The polished samples were carbon coated prior to inserting inside ESEM chamber. The ESEM was equipped with Energy-Dispersive X-ray Spectroscopy (EDS)

to allow quantitative compositional analysis. To determine the mineralogy of the fly ash sample, quantitative X-ray diffraction (QXRD) analysis was performed using PANalytical Empryeon  $\theta$ - $\theta$  powder diffractometer with para-focusing optics and  $\text{CuK}\alpha$  radiation. The measurements were performed over the angular range of  $5$ - $70^\circ$  ( $2\theta$ ). Data evaluation was performed using the software MDI-Jade 2010.

Finally, flow-through column test was performed according to ASTM D4874 to assess the leaching behavior of major, minor and trace elements [11]. The continuous operation was performed in a saturated up-flow mode in a transparent cylindrical column with the diameter of  $11\text{cm}$  and height of  $30\text{cm}$ . The optimum moisture content and maximum dry density of the fly ash were determined using ASTM D698 method [12]. Fly ash was compacted at optimum moisture content in 5 layers inside the column, and saturated according to the suggested method in ASTM D2434 using a vacuum pump [13]. The flow rate was set to collect one pore volume in  $24\pm 3$  hr. The influent solution was considered as distilled water with pH in the range  $5.1$  to  $6.5$  to simulate the rainfall conditions. A peristaltic pump was used to push the influent water passing through the column, and the leachate was collected inside a nitrogen chamber to minimize the exposure to air and prevent carbonation. Finally, the leachate was filtered through a  $0.45\ \mu\text{m}$  filter, and stored at  $4^\circ\text{C}$  to be later analyzed using ICP-AES method. The test was performed for 8 days under neutral condition. Figure 2 shows the column leaching set up.



Figure 2 – Schematic of flow-through leaching column apparatus

Before running the leaching test, acid neutralization potential (ANP) of FBC ash was measured according to EPA 600/2-78-054 method, which determines the tons of CaCO<sub>3</sub> equivalent per 1000 tons of fly ash. The pH of solid fly ash was also measured by EPA SW-846 test method 9045D, which is a common method to measure the pH of soil and solid wastes. A benchtop multi parameter meter was used to measure the pH.

## Results and Discussions

### Characterization

The physical characteristics of FBC ash were determined as presented in Table 1. The as-received ash contained very low moisture content. Density of Pennsylvania FBC ash is similar to PC ash (2.5-2.6 g/cm<sup>3</sup>), but the surface area is significantly higher for FBC ash. Loss on ignition (including organic and inorganic carbon, water, and volatile matter) was measured as 8.10%, which does not meet ASTM C618 requirement to be used in concrete (LOI<6%). However using the two-atmosphere TGA method [8], organic carbon was more precisely determined as 2.6%. FBC ash particles are larger than average reported size for PC fly ash.

Table 1- Physical properties of FBC fly ash

Physical characterization	Measure
Moisture (% wt)	0.20
Density (g/cm <sup>3</sup> )	2.60
Surface area (m <sup>2</sup> /g)	12.8
LOI% at 750°C	8.10
Organic carbon by 2A-TGA (% wt)	2.60
Particle size D10 (µm)	1.51
Particle size D50 (µm)	10.0
Particle size D90 (µm)	50.9

Bulk chemical composition of ash in terms of major and minor elements is summarized in Table 2. It was found that FBC ash mainly contained Si and Al with significantly lower amount of iron in comparison with Pennsylvania PC ash from First Energy power plant [14]. Schuylkill FBC ash does not contain the typical high amount of Ca expected for this type of ash. The reason is probably the lower sulfur concentration in the anthracite culm, which needs less limestone to capture the sulfur emissions, which also results in lower sulfur content in the ash. The ash is also rich in alkali and alkali-earth metals (Na, K, Mg, Ca, total 7.4%); these elements give fly ash its alkaline property in contact with water.

Table 2- Bulk chemical content of FBC ash measured by XRF and ICP-AES techniques

Oxides	FBC fly ash (wt. %)	
	XRF	ICP-AES
SiO <sub>2</sub>	59.1	54.04
Al <sub>2</sub> O <sub>3</sub>	23.0	21.0
Fe <sub>2</sub> O <sub>3</sub>	4.46	4.11
CaO	2.50	3.03
MgO	0.89	0.83
Na <sub>2</sub> O	0.36	0.36
K <sub>2</sub> O	3.55	3.31
SO <sub>3</sub>	0.99	1.55*
TiO <sub>2</sub>	1.41	1.24
P <sub>2</sub> O <sub>5</sub>	0.08	0.11

\*measured by LECO sulfur analyzer

Table 3- Total and environmentally available contents of trace elements in FBC ash

Element	Total (mg/Kg)	Env. Available (mg/Kg)
Ba	733	250
B	<250	<100
Mn	165	146
V	96	<25
Cr	89	27
Zn	64	37.6
Ni	31	14
Cu	41	87*
Pb	30	22.5
Co	8	5.3
Mo	3	2
As	9	13.8*
Tl	1	<1
Sb	1	<1
Se	<12.5	<5
Cd	<2.5	<1
Ag	<12.5	<5

Table 3 presents environmentally available and total content of trace elements in FBC ash. Barium, boron, manganese, vanadium, chromium, and zinc have concentrations

above 50 mg/Kg, which can be partially available to leach out. Among those elements with higher concentrations, Mn has the highest level of environmentally available content followed by Pb and Zn. Using stronger acids in total digestion typically results in higher total content, but for arsenic and copper, less acidic condition to measure environmentally available content indicated higher concentration.

### SEM/EDS Analysis

FBC ash contains mostly sub-angular particles due to lower combustion temperature in the boiler as SEM images in Figure 3 depict. The quantitative EDS results (average oxide composition) based on several area and point by point measurements, are presented in Table 4. For majority of elements, EDS results are consistent with the bulk chemistry measured by XRF and ICP-AES.

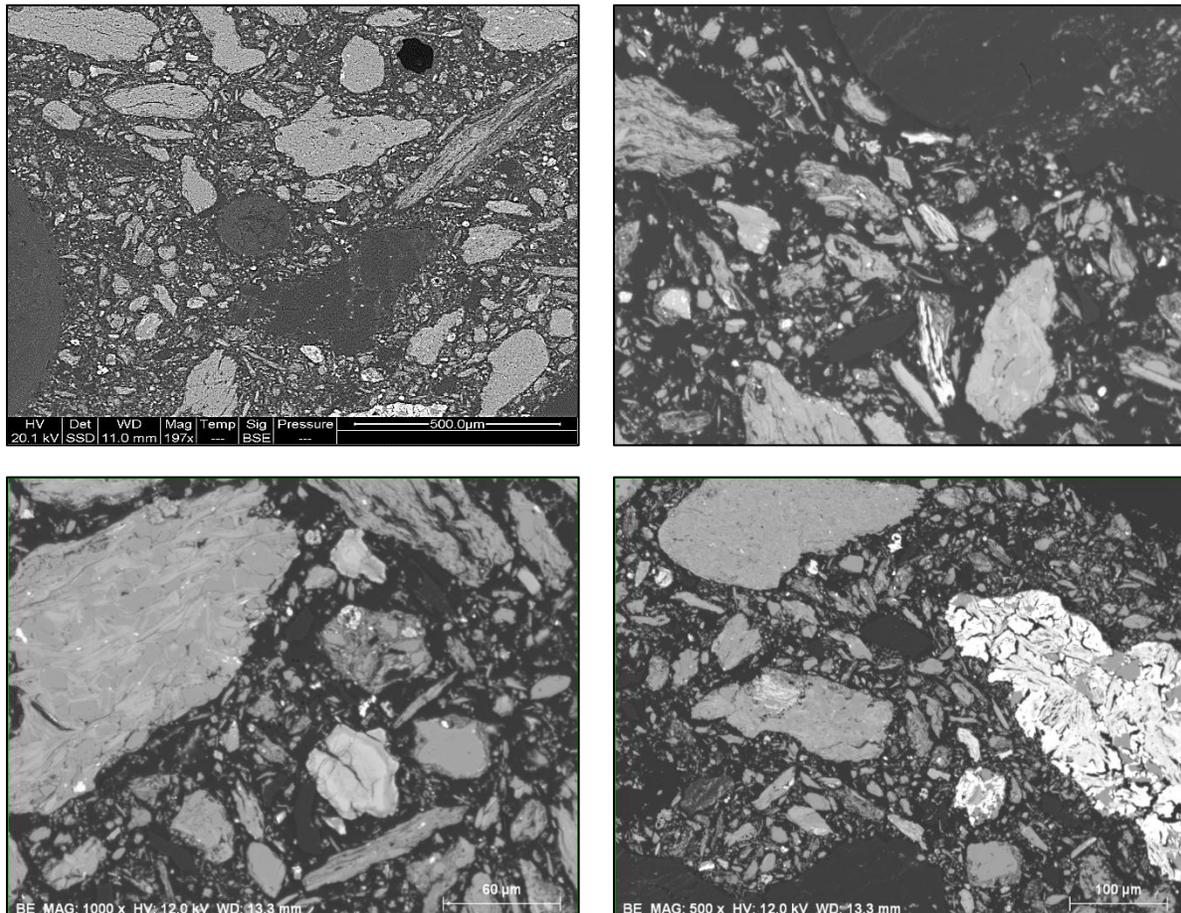


Figure 3- Backscattered SEM images of Schuylkill FBC ash

Table 4- EDS analysis results for oxide composition of FBC ash

Oxide	Wt%		
	EDS	XRF	ICP-AES
SiO <sub>2</sub>	61.3	59.1	54.04
Al <sub>2</sub> O <sub>3</sub>	22.5	23.0	21.0
Fe <sub>2</sub> O <sub>3</sub>	7.29	4.46	4.11
CaO	1.85	2.50	3.03
MgO	0.65	0.89	0.83
Na <sub>2</sub> O	0.96	0.36	0.36
K <sub>2</sub> O	2.82	3.55	3.31
SO <sub>3</sub>	1.13	0.99	1.55
TiO <sub>2</sub>	1.21	1.41	1.24
P <sub>2</sub> O <sub>5</sub>	0.25	0.08	0.11

Elemental maps for major, minor, and trace elements (As, Mo, and Se) from SEM/EDS analysis are shown in Figure 4. Analyzing elemental maps for more SEM images revealed that the sub-granular ash particles contain significant amount of Al and Si, with lower amount of Mg and alkalis such as K, and very low Na content. Al, Si, and K are overlapped in the same areas throughout the maps. Some areas with pure Si oxides were found. Iron-rich particles contain Mg and As. The similar maps for Ca, S, and Mo, shows the association of molybdenum with calcium sulfate such as anhydrite and gypsum. Selenium and arsenic are associated with Al-Si containing particles.

### QXRD Analysis

Mineralogy of FBC ash was analyzed and quantified using QXRD technique and Jade Software. Results in Table 5 show Schuylkill ash contains high clay and quartz content. Illite was the predominant clay found in the ash as EDS maps with overlapping Al, Si, and K areas also showed. Areas with pure Si in the elemental map contained the quartz particles. The amount of unspent calcite in the ash suggests low amount of sulfur in the flue gas. Moreover, no significant amount of gypsum was detected. As the temperature in FBC boiler is not high enough to fully melt the clay particles, no amorphous hump was identified in the XRD pattern, and 11% amorphous phase is representative of dehydroxylated clays in the ash.

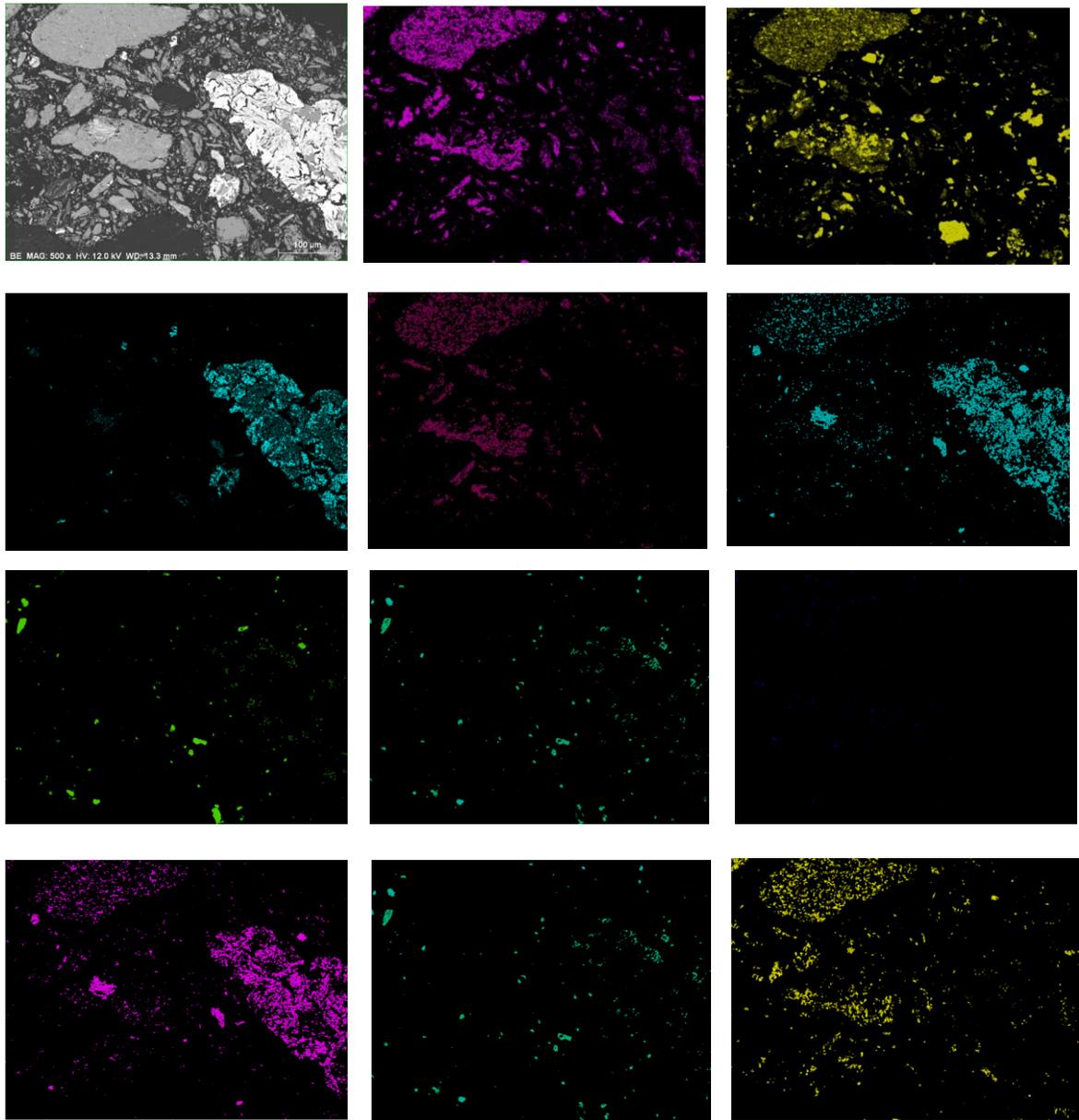


Figure4- Elemental maps for SEM/EDS analysis of Schuylkill FBC ash  
 (Top-left to bottom-right: Backscattered SEM, Al, Si, Fe, K, Mg, Ca, S, Na, As, Mo, and Se)

Table 5- Mineralogy of Schuylkill FBC ash

Mineral Phase	Wt %
Illite	48.0
Quartz	33.0
Calcite	5.15
Hematite	2.10
Albite	0.21
Amorphous phase	11.1

### Leaching Properties

Leaching behavior of Schuylkill ash was analyzed during eight days (void volumes) of water flow through compacted layers of ash in a 30cm column. The influent water was neutral (pH of 5.12 to 6.46) to simulate the rain water flow. Figure 5 shows how the pH of flow water has significantly raised up to 11.30 in contact with alkaline fly ash. The pH of solid ash was 12.44 and acid neutralization potential was measured as 22. Although the ash is qualified for use as an alkaline addition agent in terms of pH, but it does not meet the neutralization potential of at least 100 parts per thousand.

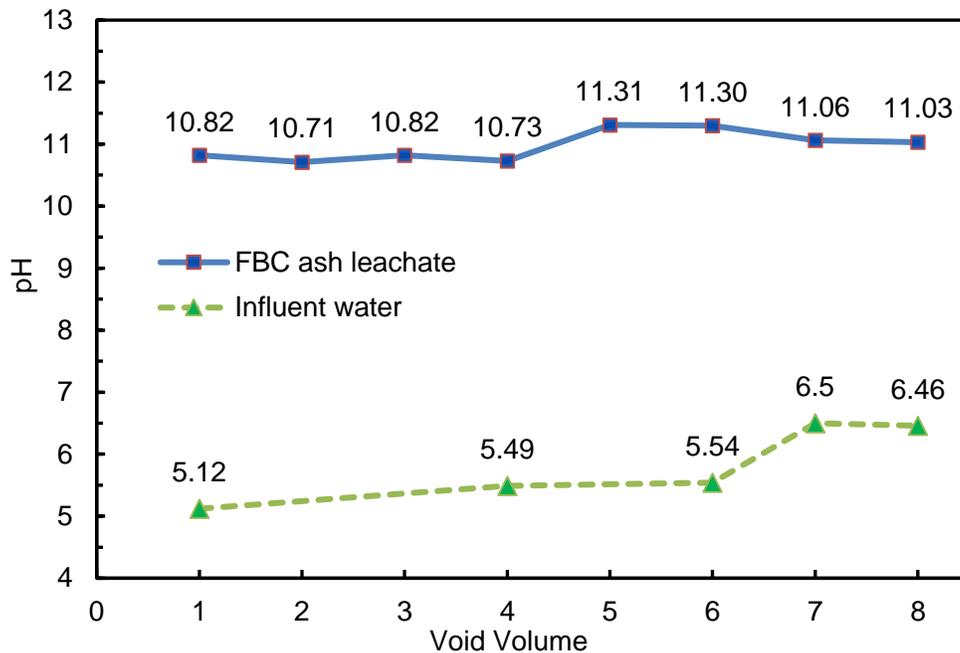


Figure 5- pH evolution during eight days of leaching for FBC ash

Leachate composition of eight void volumes are presented in Table 6. Concentration of sulfate and calcium starts to decrease gradually during the eight days of leaching. It

could be possibly due to dissolution of low amount of gypsum (detected in EDS maps but not detected in XRD analysis) and dissolving limestone, which raised the pH. Molybdenum was found to be associated with Ca and S in EDS maps, so dissolution of gypsum possibly releases this trace element to the effluent solution. Aluminum and silica dissolved from reactive dehydroxylated illite particles releasing As and Se to the leachate. Mg, Na, and K had much lower concentration (x10) in comparison with PC ash leaching, and no wash-out step was observed at the early stages comparing to PC ash [14]. Potassium is mainly trapped inside clay structure, and Na is associated with surface salts or albite minerals. Dissolution of iron oxides minerals (e.g., hematite) is in the minimum range at pH of 6-9.50, and consequently low concentration of iron was detected in the leachate at early stages of leaching process. Comparing the leachate composition with maximum acceptable leaching limits by Pennsylvania DEP [15], it was concluded that all major, minor, and trace elements concentrations were below the threshold limits. Trace elements with higher content in the solid ash (listed in Table 3) were also measured in the leachate, and compared to PA DEP leaching limits. All concentrations were below the limits, examples include Ba (0.03-0.07, Limit=50mg/L), B (0.3-0.7, limit= 15mg/L), Mn (<0.005, Limit=2.5mg/L), V (0.16-0.03, Limit= 6.12), and Cr (0.19-0.05, Limit= 2.5).

Table 6- Major, minor, and trace elements concentrations in the FBC ash leachate

VV (day)	Leachate (mg/L)										
	S	Si	Ca	Mg	Na	K	Al	Fe	Se	As	Mo
1	581	11.2	414	0.38	86	87	0.410	0.183	0.021	0.005	1.35
2	535	11.9	504	0.54	60	71	0.223	0.177	0.013	0.004	0.63
3	530	10.4	507	0.53	50	61	0.233	0.157	0.010	0.004	0.36
4	419	9.8	472	0.46	35	45	0.127	0.160	0.009	0.004	0.23
5	366	9.2	412	0.45	26	36	0.213	0.140	0.008	0.005	0.13
6	315	9.0	419	0.41	22	33	0.137	0.088	0.008	0.006	0.10
7	285	8.5	345	0.36	18	30	0.250	0.063	0.008	0.006	0.11
8	257	8.6	298	0.32	15	25	0.250	0.047	0.007	0.007	0.08
PA DEP Limit (mg/L)	2500	*	*	*	*	*	5.0	7.50	0.5	0.25	4.375

\*Limit not established

## Conclusions

Leaching behavior of Pennsylvania FBC ash from Schuylkill resources was analyzed through material characterization and column scale leaching test. The findings include:

- Dissolution of FBC ash resulted in significant change in pH, from 5 to 11, which is the result of dissolution of unspent limestone in the ash. Such high alkalinity could be used to neutralize acid mine drainage in mind lands.

- FBC ash mainly contained sub-granular clay particles (e.g., illite) with high Al and Si content, which are reactive and gradually dissolve in neutral-to-alkaline pH range.
- Concentration of all environmentally important elements in the FBC ash leachate was lower than Pennsylvania leaching limits. It could be attributed to high alkalinity, which decelerate the dissolution of some elements.
- Due to low sulfur content in PA anthracite culms, Schuylkill ash did not contain high content of gypsum.

### **Acknowledgment**

Funding provided by the Pennsylvania Coal Ash Research Group (PCARG) is greatly appreciated. We are especially thankful to Randy Lindenmuth (Schuylkill Energy) and Paul Kish (First Energy) for their insights and expertise that greatly enhanced this research.

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