Separation of Phosphorus- and Nitrogen-nutrients from Agriculturally Degraded Waters Using Pervious Filter Material Developed from Industrial By-products

Final Report

Submitted to:

Ohio Water Resources Center

Submitted by:

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ABSTRACT

End-of-tail filtration has been suggested as a more aggressive and effective approach to reduce losses of nutrients from crop lands compared to current best management practices (BMPs) focusing on source reduction and minimizing transportation. In this study, a number of industrial by-products, coal fly ash, stabilized flue gas desulfurization (FGD) materials, and bauxite leaching residual (red mud) were used in the preparation of pervious filter materials owning both reactivity to nutrients and adequate hydraulic properties for the end-of-tail filtration approach. By modifying the composition, the pervious materials derived from these industrial by-products are expected to have selective nutrient-adsorbing capabilities, which can be used to separate and recycle phosphorus- and nitrogen-nutrients from agricultural drainage waters (ADWs). A series of batch and column tests were carried out in this study to investigate the effectiveness of using selected pervious materials on the reductions of nitrate and phosphate in ADWs. The goal of this study is to demonstrate the feasibility of applying a low-cost and environmentally-sustainable approach to ADW handling and treatment. Results obtained from this study suggest that the filter material containing red mud did not have the expected adsorption effect on nitrate. However, the pervious filter material made from the coal combustion by-products, i.e., fly ash and stabilized FGD material, was found to be able to effectively remove phosphate and potentially nitrate from ADWs. The reduction of nitrate is unlikely through an adsorption mechanism. Other chemical processes, such as biological reaction, might have contributed to the observed nitrate reduction. This study suggests an end-of-tail filtration approach using agricultural and industrial wastes can be developed as an alternative to current BMPs to reduce nutrient discharges from crop lands and produce value-added products containing concentrated phosphate.

1. **Problem and Research Objectives**

Problem

  Eutrophication of water bodies, a result of release of excessive phosphorous (P) and nitrogen (N) from soil to drainages[1], has been an increasing environmental issue in the US, especially in the Midwest, northeast, and Gulf coast area where the watersheds of major freshwater bodies involve rapid growth and intensification of crop and livestock farming[2]. Not only eutrophication posts unpleasant aesthetic characteristics to water bodies, accumulation of toxic, volatile chemicals produced by algae can cause neurological damage in people and animals being exposed to them. Consequently, eutrophication of water resources results in losses of biodiversity, as well as their amenities and services[3]. For example, the recent outbreaks of Cyanobacteria, or blue-green algae, in the Grand Lake at St. Mary's area in Ohio has led to state officials to issue water contact and fish consumption advisories.

  The major cause of many eutrophication incidents can be directly correlated to fertilizer application[4]. To prevent accumulation of nutrients in surface waters, reduction of nutrients present in the agricultural degraded waters (ADW, i.e., livestock wastewater overflow, subsurface drainages, and surface runoffs from cropland) is perceived as necessary approach[5]. Although many best management practices (BMPs) focusing on source reduction and minimizing transportation have been implemented to reduce losses of nutrients from crop lands, these approaches have shown no control on dissolved phosphorus losses[6,7], which is the most readily available form of phosphorus to aquatic organisms[8]. Instead, end-of-tail filtration has been suggested as a more aggressive and effective approach[6]. However, the application is limited. Ideal filter materials, i.e., material with both favorable nutrient-sequestrating capability and hydraulic property, have yet been identified[9].

  In this study, low-cost pervious sorption materials prepared from a self-geopolymerization process using agricultural wastes and industrial by-products are tested for their potential as an alternative to current BMPs. The self-geopolymerization process enchains agricultural wastes with chemically-effective, nutrient-sorbing industrial by-products (e.g., coal ash, flue gas desulfurization materials, and bauxite residual) and forms pervious materials. By modifying the composition, the pervious materials are expected to have selective sorption capabilities to nitrogen (N-) and phosphorus (P-) nutrients with adjustable hydraulic properties, which can be used to separate and recycle nutrients from ADWs.

Objectives

In this study, a geopolymerization procedure is developed to convert coal combustion by-products (i.e., fly ash and flue gas desulfurization (FGD) material) and alkaline bauxite leaching residual (bauxite red mud) to pervious filter materials. The materials are tested in a bench-scale setting for their effectiveness and capacity on removing nutrients from simulated agricultural drainage waters. The main objective of this study is to assess the effectiveness of using the industrial by-product-derived pervious filter materials on the reduction of nutrients in ADWs.

2. Methodology

Pervious Filter Material Preparation and Characterization

Coal combustion by-products (i.e., fly ash and stabilized FGD materials) and bauxite leaching residue (i.e., red mud) are used in the preparation of the nutrient-selective pervious filtration materials (Figure 1). Two different types of pervious filtration materials (i.e., P- and N-types) are prepared using a method modified from Cheng et al.[10] and Jin[11].

P-Type Filter Material

Class F fly ash and sulfite-rich stabilized FGD material provided by coal combustion power plants located in eastern Ohio are used to prepare the phosphorous-capture (P-type) filtration materials. Quick lime (Carmeus USA, Pittsburg, PA), CaO, is added to provide required alkalinity.

N-Type Filter Material

The nitrogen-capture materials are prepared from red mud, fly ash, and stabilized FGD material. No quick lime is used in the preparation of N-type filter materials. The bauxite red mud provided by a bauxite processing plant located at southeast Texas is oven-dried before use. Woodchip is used in the preparation of both N and P-type filter mixtures to modify the hydraulic properties. The prepared mixtures are then cured in a humidity chamber.
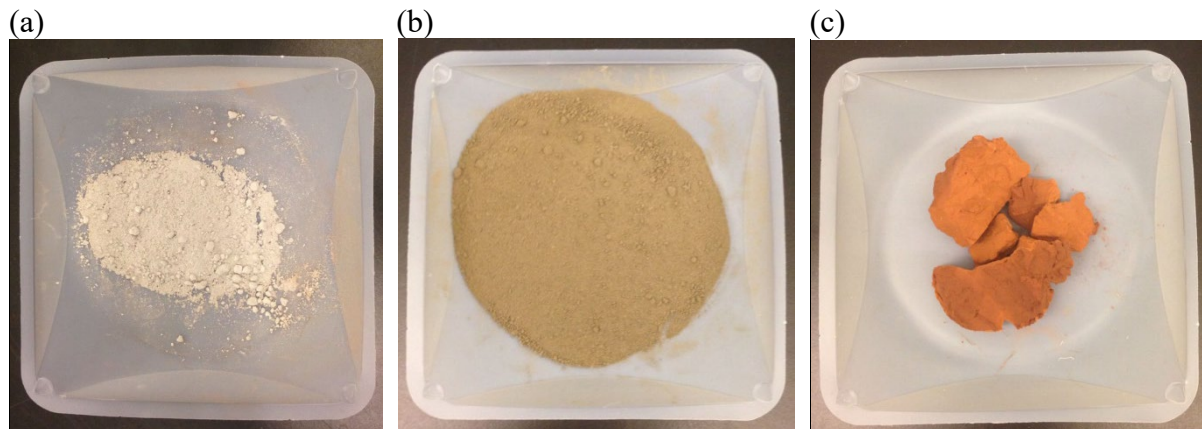
Figure 1. (a) Stabilized FGD material, (b) fly ash, and (c) bauxite red mud used in the preparation of pervious filtration materials.

These two types (i.e., P- and N-types) of pervious materials are expected to have selective sorption capacity, which can be used to sequentially separate and recover soluble phosphorous and nitrogen in agricultural drainage waters. In practice, two different pervious filter materials can be used in series. The dissolved phosphorous is expected to be selectively retained in the first pervious material (P-type) containing only fly ash and FGD material while allowing nitrate to pass through. Nitrate is captured in the second pervious material (N-Type) containing bauxite red mud, fly ash, and stabilized FGD material.

Details on the chemical and physical characterizations of the filter materials are described in the "*Physical and Chemical properties Integrity Evaluation*" section.

Bench-Scale Column Test

A series of column tests are carried out to measure the adsorption capacity and efficiency of prepared pervious materials for P- and N-nutrients with a simulated ADW. The setup of the column test is illustrated in Figure 2. A solution with simple matrix containing $Na_2PO_4 \cdot 12H_2O$ and $NaNO_3$ was used in the column tests. The column tests were carried out either in a close-loop or flow-through mode. For a given column test, a peristaltic pump delivers the solution to the inlet of a series of two vertically-oriented columns at a constant feed rate (Figure 2). The ADW sequentially passes through the column containing P-type filter material (P-type column) and then the N-Type column. For a given set of filter materials, the column test is carried out under a saturation condition demonstrated in Figure 2.
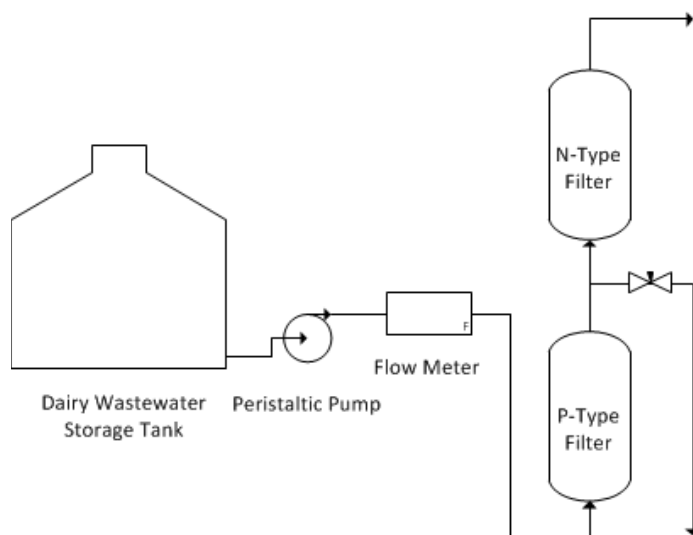
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Figure 2. Setup of bench-scale column test

Effluent samples are collected periodically from the outlets of P-type and N-type columns for a list of chemical analyses for Cl-, SO_4^{-2}, PO_4^{-3}, And NO_3^-.

3. Principal Findings and Results

*Characterizations of Industrial By-products*

The chemical compositions of fly ash, stabilized FGD material, and bauxite red mud are first characterized and the results are summarized in Table 1. As shown in the table, calcium (Ca) and sulfur (S) are the two most abundant elements in the stabilized FGD material, which is associated with the presence of hannebachite ($CaSO_3 \cdot 0.5H_2O$), portlandite ($Ca(OH)_2$), and enttringite ($Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$) in the material. The X-ray diffractogram and mineral composition of stabilized FGD material can be seen in Figure 3. Iron (Fe), aluminum (Al), sulfur (S), and silicon (Si) are the major elements in fly ash. Based on XRD analysis, the fly ash used in this study is comprised of amorphous glass, aluminum silicates (e.g., mullite), and iron oxides (hematitem, magnetite, and maghemite). Bauxite red mud is consisted of Al, Fe, and Ca. The X-ray diffractograms of fly ash and red mud are not shown.

By properly coalescing fly ash, stabilized FGD material, and red mud under high alkaline environment, fly ash acts as an inorganic polymer binder to enchain active ingredients through a geopolymerization process. After being alkali-activated, the Si-O-Si or Al-O-Si bonds in fly ash and stabilized FGD material are disassociated and subsequently form network-like crystalline

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and/or amorphous alkaline aluminosilicates with structural framework similar to zeolite[12]. In a previous project, it has been demonstrated that a geotextile material derived from the geopolymerization process with a mixture of fly ash and stabilized FGD material, has effective phosphorus sorption capability by forming Ca- and Fe-precipitates[10,13,14]. However, the fly ash/stabilized FGD material mixture did not show observable effect on nitrate mitigation[10].

Bauxite red mud was added to enhance the nitrogen-nutrients adsorption capability of the fly ash/FGD mixture. Bauxite red mud contains minerals, e.g., iron (III) (hydr)oxides and hydrous aluminum oxides, that have high affinities for nitrate[15]. As a result, the material has been shown to be an effective nutrient sorbent[16]. Cengeloglu et al[16] used original and acid-treated bauxite red mud to remove nitrate from aqueous solution and reported 70% and over 90% of removal, respectively. They found the alkaline property of bauxite red mud hindered the adsorption performance.

In this study, bauxite red mud is used as the sole alkalinity source in the geopolymerization process, which might promote the nitrate adsorption capacity. During geopolymerization, the OH- ions from bauxite red mud is consumed (eq. 1) and redistribute the electron density around the silicon atom in fly ash, which weaken the strength of Si-O-Si bond[17] and progress the polymerization process. The reaction neutralizes the negative surface charge of red mud particles, and therefore, might promote the nitrate sorption.

$$\equiv Si - O - Si \equiv + OH^- \Rightarrow \equiv Si - OH + {}^-O - Si \equiv \tag{1}$$
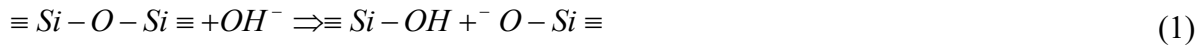
Preparation of P- and N-type pervious filtration

A series of P- and N-type pervious filtration materials have been prepared based on the formulas listed in Tables 2 and 3. Currently, the prepared materials are undergoing a 21-day curing process. The images of two selected prepared materials can be seen in Figure 4. The hydraulic property of the filtration materials are adjusted by the addition of woodchip. Two different sizes of woodchip, i.e., <2.3mm and 2.3-3.6mm, are used. The addition of woodchip creates larger capillary routes for water to pass through. During the geopolymerization process, active ingredients are coated on the surface of woodchip, which allows the nutrients in ADW to react with the active ingredients while passing through the void space.

Table 1. Chemical compositions of fly ash, stabilized FGD material and bauxite red mud used in this study

		Fly Ash	Stabilized FGD material	Red Mud
Phosphorus	P	531	177	1054
Potassium	K	2986	1307	310
Calcium	Ca	9836	172906	33055
Magnesium	Mg	1528	10026	227
sulfur	S	11827	85746	2867
Aluminum	Al	27050	9705	62817
Boron	B	531	313	<3
Copper	Cu	42	<0.4	<0.8
Iron	Fe	59824	18855	240960
Manganese	Mn	85	73	139
Molybdenum	Mo	22	<13	<0.5
Sodium	Na	18851	5296	32412
Zinc	Zn	109	40	22
Arsenic	As	143	36	28
Barium	Ba	177	137	61
Beryllium	Be	<0.18	<0.11	<0.18
Cadmium	Cd	2	6	5
Cobalt	Co	23	4	15
Chromium	Cr	74	25	1397
Lithium	Li	167	106	55
Nickel	Ni	48	7	6
Lead	Pb	28	8	46
Antimony	Sb	<1.5	17	<1.5
Selenium	Se	20	18	1
Silicon	Si	4771	1481	184
Strontium	Sr	229	212	117
Thallium	Tl	129	38	871
Vanadium	V	2	<1.1	<0.6
Mercury	Hg	NA	0.318	NA

NA:Not Available
Unit: mg/kg

Table 2. Formulas of Prepared P-type Filtration Materials

	P-type
Fly Ash	10.0
Stabilized FGD material	6.0
Quick Lime (CaO)	1.2
Deionized Water	10.5
Wood Chip (<2.3 mm)	2.5

Unit: g

Table 3. Formulas of Prepared N-type Filtration Materials

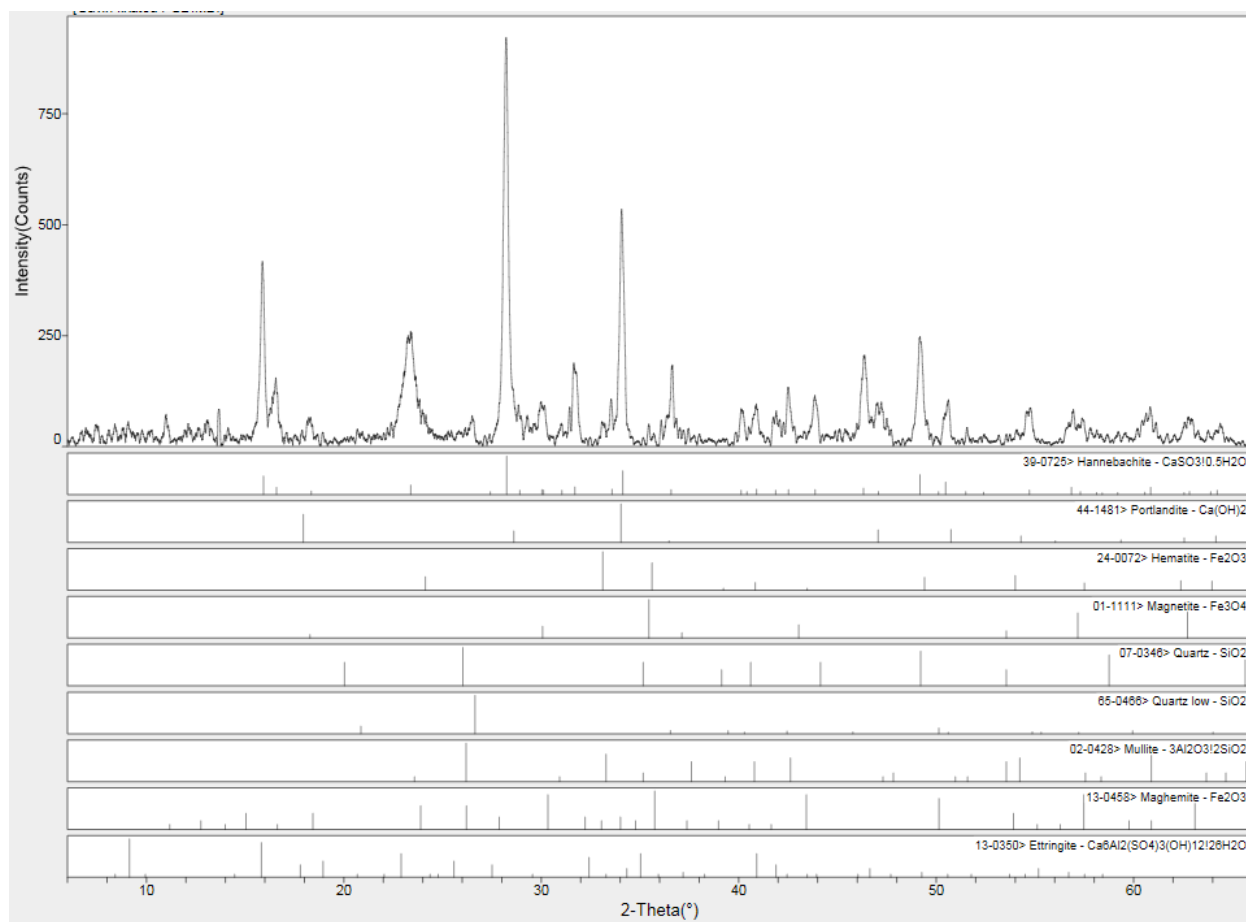|	N-type
Fly Ash	10.0
Stabilized FGD material	6.0
Red Mud (dried weight)	8
Deionized Water	10.5
Wood Chip (<2.3 mm)	2.0

Unit: g

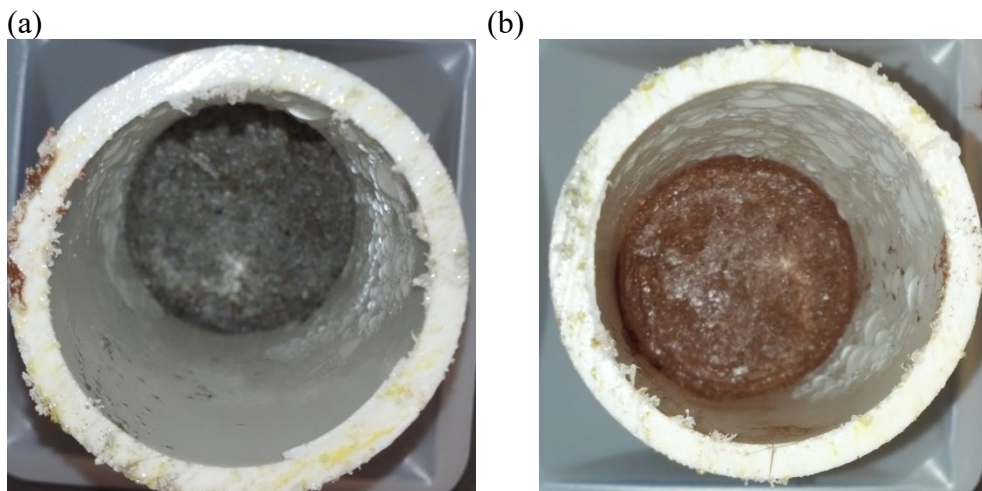Figure 3. Mineral composition of stabilized FGD material

Figure 4. Prepared Pervious filtration materials. (a) P-type and (b) N-type.

Adsorption Capacities

The nutrient adsorption capacities of P- and N-type materials were evaluated using the materials prepared from the formulas listed in Tables 2 and 3 for the P-Control and N-Control materials. For either type of the material, the adsorption experiment was carried out by adding six different amounts of the prepared solid, ranging from 0 to 1 gram, into six separate 125-mL HDPE bottles. Each bottle contains 100mL of either 250 mg/L of phosphate or 100 mg/L of nitrate solution. The bottles were then mixing by a tumbler for 24 hours at a rotating speed of 18 rpm. After mixing, the solution collected from each bottle was filtrated with 0.45mm filter and analyzed for NO_3^- or PO_4^{-3}.

The equilibrium concentrations of phosphate and nitrate in the solution after mixing as a function of material dosage are shown in Figure 5. As shown in the figure, over 97% of phosphate was removed by the P-type material with a solid-to liquid (L/S) ratio of 100. With the same L/S ratio, no significant nitrate was adsorbed by the N-type material.
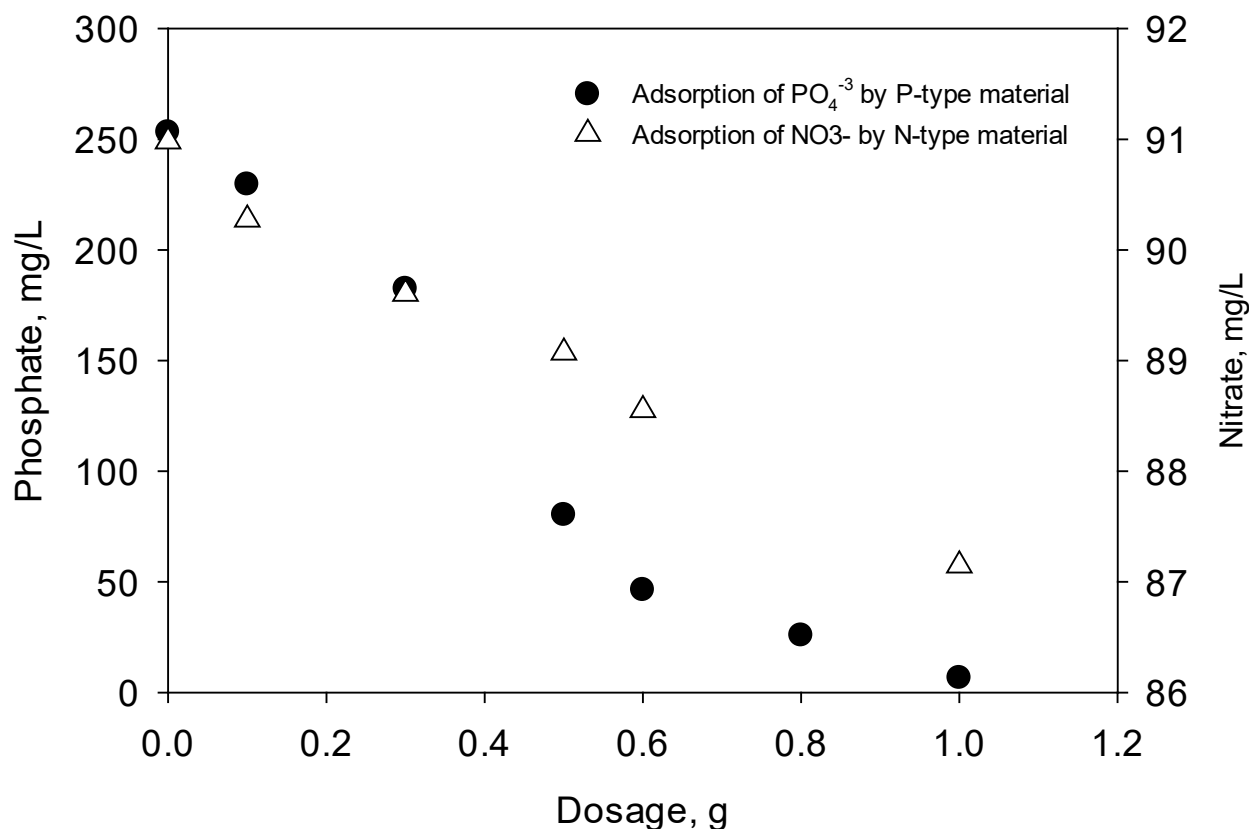

Figure 5. The equilibrium concentrations of phosphate and nitrate in the solution as a function of material dosage

The adsorption isotherms of phosphate on P-type material and nitrate on N-type material are illustrated in Figure 6. As shown in the figure, the adsorption isotherms of phosphate and nitrate can be expressed as Langmuir isotherm. The Langmuir isotherm equation is written as

$$\frac{C_e}{q_e} = \frac{1}{K \cdot Q_a^0} + \frac{C_e}{Q_a^0}$$

Eq. 1

where q_e is mass of material adsorbed (at equilibrium) per mass of adsorbent; Q_a^0 represents the maximum adsorption capacity (monolayer coverage); C_e is the equilibrium concentration in solution when amount adsorbed equals q_e; K is constant (L/mg).
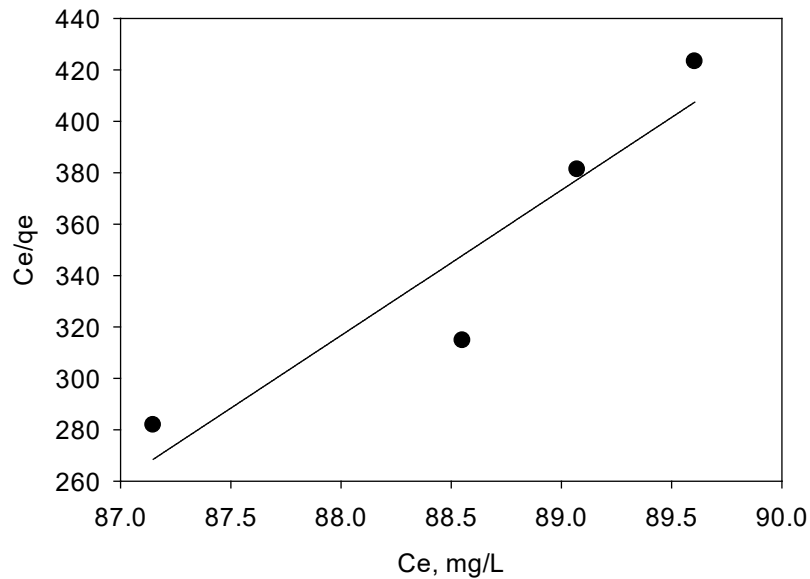
(a)
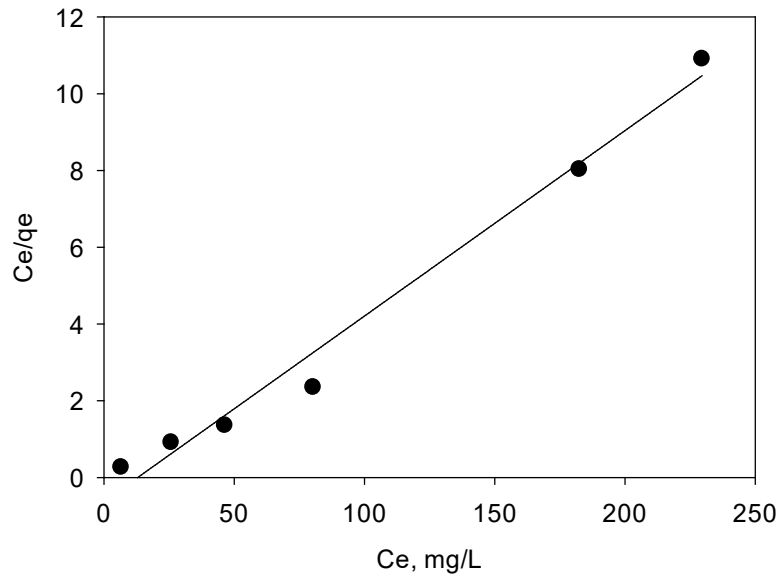
(b)

Figure 6. Langmuir isotherms for (a) phosphate and (b) nitrate

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It is estimated that the maximum phosphate adsorption capacity of P-type material is 20.7 mg/g. For the N-type material, the adsorption capacity was approximately 0.18 mg/g, which is much less than the expected adsorption capacity.

Column Test

Close-loop Column System

Two series of close-loop column tests were carried out to simulate the removal of phosphate and nitrates when passing ADW through an unlimited length of pervious sorbent column. In the first series, the solution was first introduced into P-type column and then N-type column. In the second series, only N-type column was used. The flow rate was kept at 1.13±0.17 mL/sec for both series. A simplified agriculturally degraded solution prepared with NaH_2PO_4 and $NaNO_3$ was used. A collection schedule was then setup to collect a series of eluent fractions based on pre-determined time interval. During each sampling event, eluents were collected from the inlet and outlet of the first column, as well as the outlet of the second column in the first series, for nitrate and phosphate analyses.

The temporal trends of nitrate and phosphate at the inlet of the first column can be seen in Figure 7, which represent the concentrations in the storage tank. It was found that the concentration of nitrate in the first series decreased over 68.5% (from the original 47.1 mg/L to 14.8 mg/L) after 30 hours of circulation. In the second series, a similar removal efficiency (60.1%) was observed during the first 26 hours when only N-type column was used. However, the concentration of nitrate decreased to a level lower than the detection limit after 146 hours of circulation. In the case of phosphate, over 95% of the phosphate in the solution was removed within 30 hours of circulation in both close-loop series.

Results observed from the two close-loop series of column tests demonstrate that the pervious filter materials prepared in this study can effectively decrease the concentrations of nitrate and phosphate. Although the concentrations of both nitrate and phosphate showed a decreasing trend throughout the testing period in both testing series, for a given sampling interval, no significant changes were observed between the samples collected before and after the columns. It suggests that the time for the solution to travel through the lengths of these columns was not long enough to show any changes.

The decreases of nitration concentration observed in both column tests were unlikely due to adsorption. Results obtained from the adsorption isotherm experiment suggest that the adsorption of nitrate on the N-type material is very limited. Other mechanisms, such as biological

reduction, might have involved in nitrate removal. Also, it seems the addition of red mud did not have significant effect on the reduction of nitration concentration.
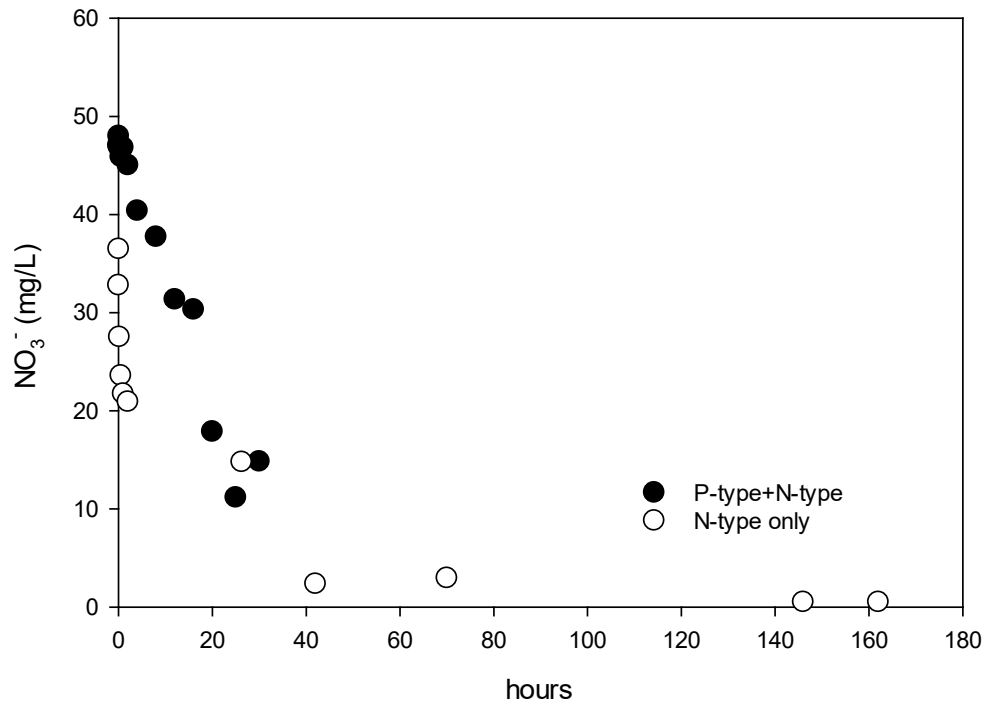
Flow through Column System

A flow-through column test was setup to further investigate the removal of nitrate and phosphate under the condition that is similar to real application. Since no significant difference in the removal of phosphate and nitrate between the P- and N-types sorbents, only the P-type material was used in the test.   The material was packed into a 2.5 ft long acrylic column with a diameter of 6 inches. The flow rate of the simulated agriculturally degraded solution, prepared from the same formula used in the close-loop column test, was controlled at 0.46 mL/min. As a result, the retention time of the solution in the column was maintained at 20 hours.

Results obtained from the test can be seen in Figure 8. As shown in the figure, over 77% of nitrate removal was achieved short after one pore volume passing though the column, which increased to 98% after approximately 168 hours. Compared to the results obtained from the close-loop system, which is also shown in Figure 8, the temporal trends of nitrate removal are very similar between the two systems.

In the case of phosphate, over 99% of removal was achieved after about 560 hours or 28 pore volumes, which increased from the 82.5% observed after about one pore volume. The removal of phosphate kept increasing as more solution passing through the column. It suggests that the adsorption of phosphate of was likely controlled by the release of sulfate and the complexation of phosphate on the pore surface of the pervious material. As more solution passing through the column, more sulfate was released from the matrix of the pervious material, which allowed more phosphate to be retained within the pervious material.

Results obtained from the flow through column test confirmed the potential of using the pervious material derived from stabilized FGD material (P-type) to remove both nitrate and phosphate from agriculturally degraded solution.
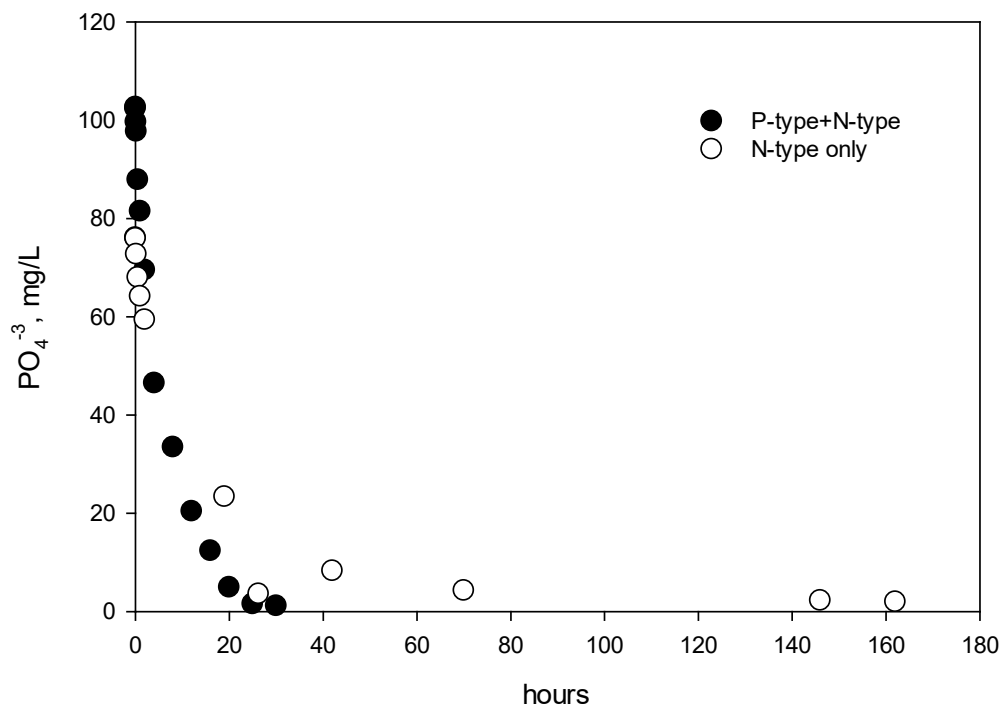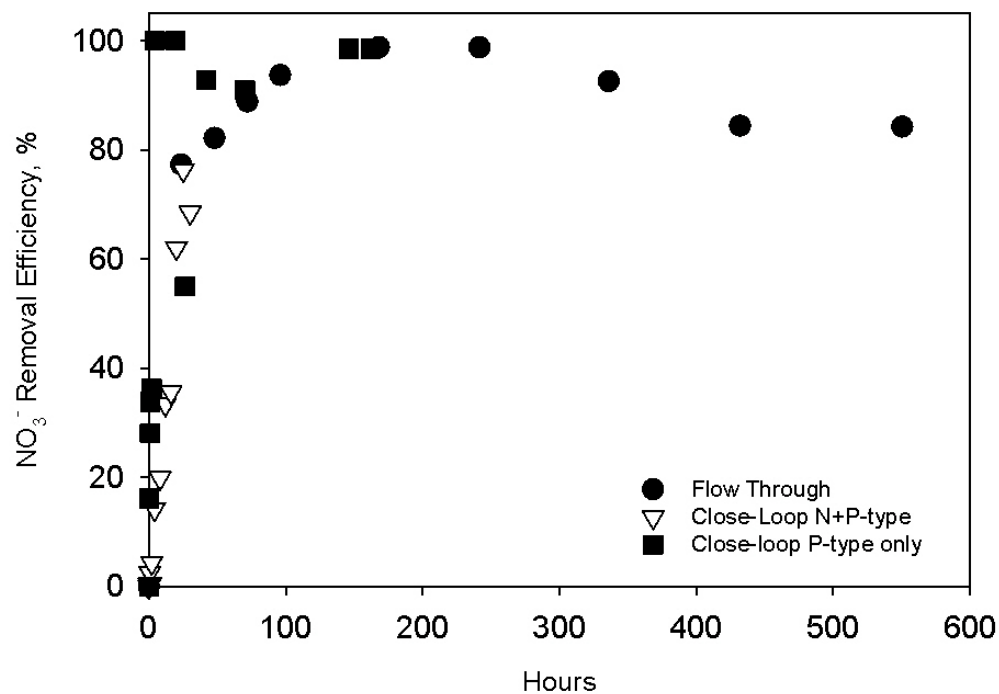
(a)

(b)

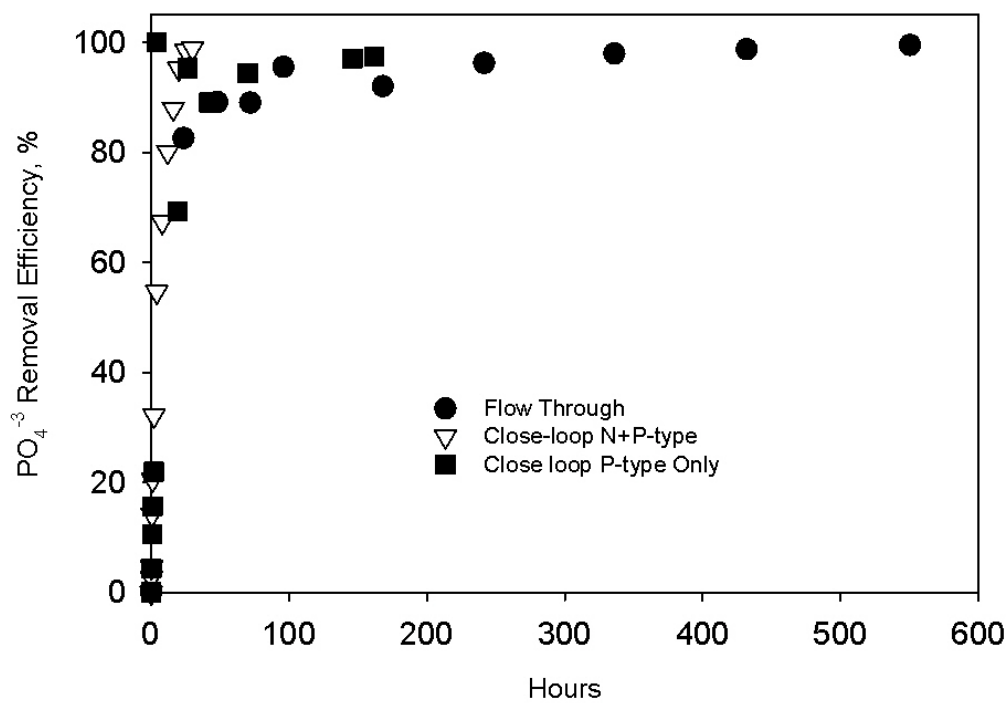Figure 7. Temporal Trend of nitrate and phosphate in the close-loop column system

(a)

(b)

Figure 8. Removal efficiency of (a) nitrate and (b) phosphate using the P-type pervious material with a flow through column

4. Finding Significance

Results obtained from this study suggest that the pervious filter material made from the coal combustion by-products, i.e., fly ash and stabilized FGD material, can effectively remove phosphate and potentially nitrate. The reduction of nitrate is unlikely through an adsorption mechanism. Other chemical processes, such as biological reaction, might have contributed to the observed nitrate reduction.

The addition of red mud to the pervious filter material made from coal combustion by-products did not have the expected adsorption effect on nitrate. Based on the results from batch adsorption experiments, the adsorption of nitrate on the filter material containing red mud is insignificant.

This study suggests an end-of-tail filtration approach using agricultural and industrial wastes can be developed as an alternative to current BMPs to reduce nutrient discharges from crop lands and produce value-added products containing concentrated phosphate. As demand for food increases, which results in more land to be used for agricultural purpose and a requirement for increased crop yields, the fertilizer demand have been projected to increase faster than world population[18]. Currently, the majority of phosphate rock from mining goes into artificial fertilizer production[19]. It estimates that sources of high-grade phosphate ore deposits could disappear within the next 100 years at current use rates[20]. With foreseeable increase in demand and depletion in reserve, use of recycled nutrients rather than a raw material is important step toward sustainable agricultural development.

5. References

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2) Publication citations (all journal articles, proceedings and presentations at conferences)

Not available.

3) Number of students supported by the project (MS/PhD/undergraduate/post docs) as well as their majors

Undergraduate Student

    Micah Sauder (January 2016-September 2016), Environmental Engineering.
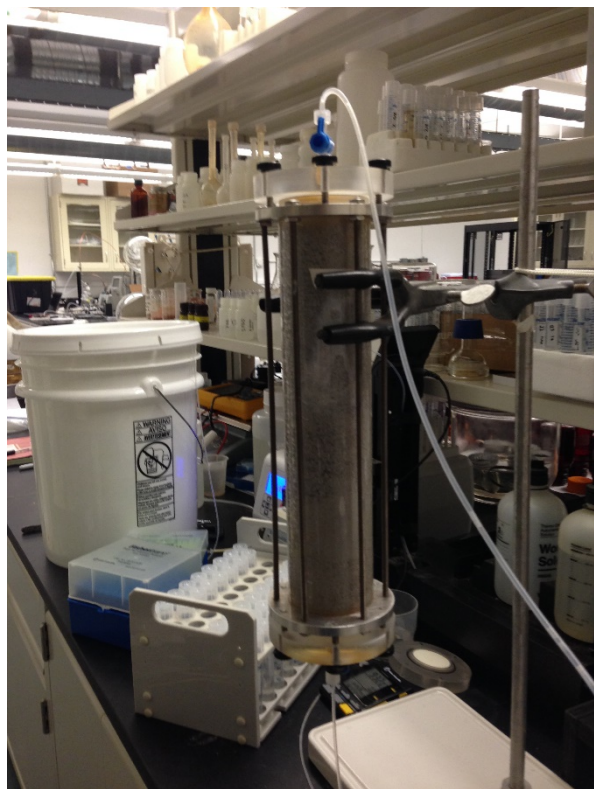
    Josh Miller (November 2017-May 2018), Environmental Engineering.
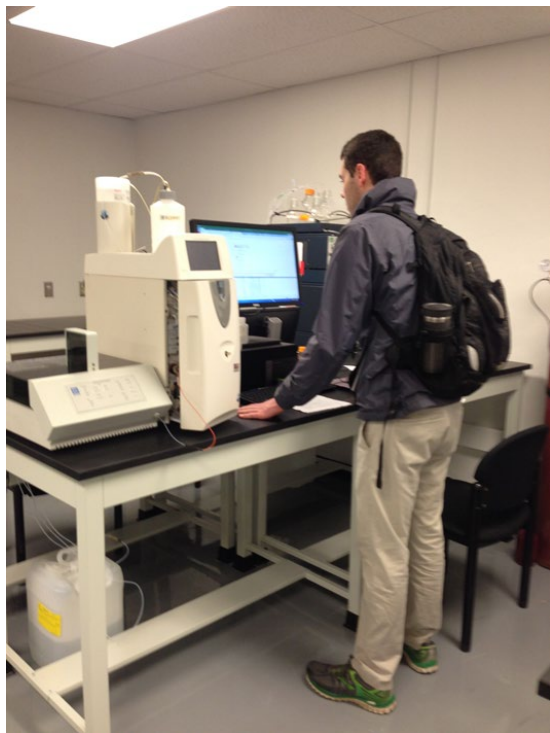
PhD student

    Vinny Anderson (June 2018-April 2019), Environmental Engineering.

4) Awards or Achievements (patents, copyrights), additional funding for this project

    $3,500 College of Engineering Undergraduate Summer Research Fund (2016)

Setup of the Column Tests

Josh Miller, an undergraduate research assistant, is operating ion chromatography.