Assessment of Recycled and Manufactured Adsorptive Materials for Phosphate Removal from Municipal Wastewater

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Abstract: Elevated concentrations of phosphorus (P) and other nutrients common in wastewater treatment plant (WWTP) effluent have been shown to contribute to the proliferation of harmful algal blooms, which may lead to fish kills related to aquatic hypoxia. Increased understanding of the negative effects associated with elevated P concentrations have prompted more strict regulation of WWTP effluent in recent years. The use of low-cost and potentially regenerative adsorptive phosphate filters has the potential to decrease P concentrations in WWTP effluent released to natural waters. This research focuses on assessing the capacities of recycled concrete aggregate (RCA), expanded slate, and expanded clay to remove phosphate from P-amended WWTP effluent. Results from a flow-through column study indicate that RCA consistently removed an average of 97% of phosphate over 20 weeks of continuous flow at an 8-hour hydraulic retention time (HRT). Expanded clay removed an average of 63% of introduced phosphate but decreased in removal capacity from 91 to 42% over the 20-week duration. Sorption data from batch studies were fitted to Langmuir models and RCA was shown to have the highest maximum sorption capacity (6.16 mg P/g), followed by expanded clay (3.65 mg P/g). RCA and expanded clay are promising options for use in passive filters for further reduction of phosphate from WWTP effluent.

Keywords: *Langmuir model passive filter, sorption, treatment*

Eutrophication is the process of accelerated plant and algae growth resulting from the introduction of excess nutrients into waters, potentially resulting in harmful algal blooms. Aerobic microorganisms then consume dissolved oxygen while breaking down the organic matter, resulting in lower levels of dissolved oxygen, known as water hypoxia (Paerl 2009). Increases in eutrophication levels can be caused by anthropogenic sources, such as domestic wastewater from wastewater treatment plants (WWTPs), or onsite wastewater systems, such as septic systems (Preisner et al. 2020). Inputs of phosphorus (P) from point and non-point source wastewater can negatively affect wildlife and human health. Limiting inputs of P to receiving waters can decrease the severity of eutrophication (Xie et al. 2013).

Hydrologic and climatic challenges, paired with aging infrastructure and increasingly stringent effluent regulatory limits, have incentivized WWTPs to seek alternative and supplemental treatment options. The use of low-cost and potentially regenerative adsorptive filters can remove P from wastewater and decrease concentrations released to natural bodies of water (White et al. 2021). These adsorptive materials typically contain compounds that can bind P, such as oxides of calcium (Ca), magnesium (Mg), iron (Fe), and aluminum (Al), which enable adsorption and precipitation processes to occur (Gubernat et al. 2020). Many natural, manufactured, and waste P adsorptive materials have been investigated; however, efficacy, cost, and availability of these materials vary widely (Cucarella and Renman 2009). Large volumes of concrete waste are

Research Implications
• Recycled concrete aggregate (RCA) and manufactured expanded clay may be used as passive adsorptive filters to further reduce phosphorus (P) loads from wastewater treatment plants (WWTPs).
• Potential negative environmental impacts from the use of adsorptive P filters, such as increased pH and leaching of ions, should be explored.
• Desorption characteristics of adsorptive P filters should be investigated to determine their potential as a substrate or soil amendment to grow crops, moving us toward a circular economy.

Materials and Methods

A locally produced RCA (Carolina Concrete Recycling, New Bern, NC, USA), a locally manufactured expanded slate (Stalite Lightweight Aggregate, Salisbury, NC, USA), a manufactured expanded clay (Filtralite®, Nordby, Norway), and a locally procured granite gravel (E.R. Lewis Construction Company, Greenville, NC, USA) as a control, were assessed to determine phosphate sorption efficacy. Two experiments were carried out: 1) a flow-through column study to characterize phosphate sorption efficacy under higher flow conditions than have generally been reported in the literature (Vohla et al. 2011), which could allow for treatment of higher volumes of water; and 2) a batch study to determine the phosphate sorption capacity of each material. All materials were sieved to the 1.00 to 3.35 mm particle size range using sieve pans. Effluent from a WWTP located in Greenville, NC, USA, was used in both studies and collected within one week prior to each experiment. Treated effluent was collected using a sump pump from the clear well, located after the UV disinfection process and just before release of treated effluent to a nearby river. Experimental conditions and sampling frequency are described below.

produced globally as older concrete buildings are demolished for new builds, and in many cases, this concrete is not reused (Deng and Wheatley 2018). Recycled concrete aggregate (RCA) has been investigated as a reactive material for P removal due to high lime content, with studies estimating up to 70 mg P/g maximum sorption capacity, according to the Langmuir model (Gubernat et al. 2020). Manufactured lightweight aggregates (e.g., slate and clay), which are natural minerals that have been crushed and heated to high temperatures, have also demonstrated high P sorption capacity – up to 12 mg P/kg (Zhu et al. 2003; Vohla et al. 2011; Baker et al. 2014; Gubernat et al. 2020).

While previous lab studies have proven many adsorbent materials to be effective at reducing P concentrations in various solutions, most are conducted at hydraulic retention times (HRTs) on the order of days (rather than hours) or exclusively as batch studies, and most do not assess actual effluent from a WWTP (Vohla et al. 2011; Wu et al. 2020). The objective of this study was to determine the phosphate sorption capacity of RCA, expanded slate, and expanded clay at the lab-scale under both batch and flow-through conditions using effluent from a local WWTP. Testing the materials at batch and flow-through conditions (i.e., at varying hydraulic conditions and scales) provides a more holistic understanding of how the materials would perform as adsorptive filters in a WWTP setting.

Flow-through Column Study

A column study was conducted to identify how much P was able to be removed in conditions similar to a WWTP. Physical properties of experimental materials are reported below (Table 1). Columns were filled with oven dried materials (tamping intermittently) and then saturated with water to fill the pore spaces. Porosity was calculated as the amount of added water divided by total column volume. Bulk density was calculated as the difference between empty and material-filled column weights divided by the column volume. As shown in Table 1, weight varied considerably amongst materials.

As shown in Figure 1, three replicate columns containing each material were made of clear polyvinyl chloride (PVC) and measured approximately 30 cm tall with a diameter of 10 cm (total volume approximately 2.36 L). Treated wastewater at the partnering WWTP typically

Table 1. Average porosity and bulk density values (n = 3) for materials used in batch and flow-through column study using recycled concrete aggregate (RCA), expanded clay and slate, and gravel.

Material	RCA	Expanded Clay	Expanded Slate	Gravel
Porosity (%)	48.8	31.6	33.0	83.7
Average Bulk Density (kg/m³)	1184	1445	850	464

Figure 1. Experimental set-ups of flow-through column study (left) and sorption capacity batch study (right) in Coastal Ecological Engineering Lab at East Carolina University.

contains less than 1 mg PO_4-P/L; however, during periods of high rainfall or due to unanticipated influent characteristics, effluent concentrations can reach 5 mg PO_4-P/L or higher at times. During the course of this experiment, the WWTP effluent maintained concentrations below 1 mg PO_4-P/L when effluent was collected. To represent a "worst-case" scenario and ensure P removal was not limited by low P concentrations typically observed in the effluent, the WWTP effluent was amended with approximately 5 mg PO_4-P/L and continuously pumped into the bottom of each column up to the top where it was connected to an effluent tube as shown in Figure 1. A calculated (i.e., theoretical) 8-hour HRT was maintained throughout the duration of the study from March to July 2021. Actual HRT, which can be determined using tracers, was not investigated, though could shed light on potential preferential flow or dead spaces. The flowrate of influent water for each material type was calculated by dividing the product of column volume and porosity by the calculated HRT. In order to determine representative long-term P removal efficacy, samples were collected from the influent and effluent of each column one to two times each week on average and analyzed for phosphate using the Hach (Loveland, CO, USA) DR6000 spectrophotometer and test-n-tube kits using Method 10209/10210. Only samples for which the detected concentration of the accompanying standard was within +/-10% of the standard concentration value were considered for statistical analyses. The pH of samples was measured using a multiparameter benchtop meter

(Orion™ Versa Star Pro™, Thermo Fisher Scientific Inc., Waltham, MA, USA). The temperature, conductivity, and oxidation reduction potential (ORP) were measured using a YSI ProDSS.

Percent PO$_4$-P removal was calculated as follows:

$$\% \ Removal = \frac{(C_i - C_e)}{C_i}(100) \qquad (1)$$

where C_i is the influent concentration and C_e is the column effluent concentration.

Average load removal was also calculated as follows for each material:

$$Load \ Removed = \frac{(\% \ Removal)}{100} * (C_{i \ avg}) * Q \qquad (2)$$

where $C_{i \ avg}$ is the average influent concentration and Q is the flowrate.

Statistical comparisons were made between treatments (material type) using JMP Pro 17 (SAS Institute 2022). Shapiro-Wilk tests indicated that data were not normally distributed ($p < 0.0001$); therefore, non-parametric Kruskal-Wallis tests were used to determine if phosphate reduction, pH, specific conductivity, and ORP differed significantly ($\alpha = 0.05$) by material type. When significant differences were found, the Steel-Dwass post-hoc test was used to separate treatment medians to determine which specific material types differed. Though transformation of data followed by a parametric comparison test could have been utilized, a non-parametric approach was chosen to avoid potential over-compensation and distributional assumptions that may not adequately address non-normality and unequal variance issues (Mahachie John et al. 2013).

Sorption Capacity Batch Study

The second portion of the study was a batch study to determine the maximum P sorption capacity of each material. The materials were dried in an oven for 24 hours before use. For the first experimental trial, wastewater effluent was amended with monopotassium phosphate (KH$_2$PO$_4$) to achieve the following initial concentrations of P (C_i) in solution, similar to those selected by White et al. (2021): 0, 5, 10, 20, 30, 50, 100, 200, 500, 1000, 1500, and 2000 mg/L. As KH$_2$PO$_4$ was added to the wastewater effluent, pH decreased as shown in Table 2. Therefore, a second experimental run was carried out in which initial pH of KH$_2$PO$_4$-

amended wastewater effluent was adjusted to the pH of the unamended wastewater (6.8) using potassium hydroxide to achieve the following initial concentrations of P (C_i) in solution: 0, 20, 50, 200, 500, and 1000 mg/L.

Each centrifuge tube contained 5 g of each material type along with 45 mL of amended wastewater effluent. There were three replicates of each material type for each different concentration. Once the weighed material and desired concentration of wastewater effluent were placed into the tube, the tubes were put into an orbital shaker for 24 hours at 150 rpm at room temperature (between 20 to 25°C), following methods by White et al. (2021). After 24 hours, samples were syringe-filtered (0.45 μm) and frozen until analysis (within 24 hours). All samples were analyzed for PO$_4$-P using a Smartchem 200 Discrete Analyzer (KPM analytics, Westborough, MA, USA) located in East Carolina University's Environmental Research Laboratory, using standard methods (APHA 2012).

Phosphorus sorption isotherms were created by plotting concentrations of sorbed PO$_4$-P (C_s, mg P/g substrate) for each replicate (n = 3) by concentration of PO$_4$-P remaining in solution (C_{aq}, mg P/L). Sorbed P was calculated as follows:

$$C_s = \frac{((C_i - C_{aq})(V))}{M_m} \qquad (3)$$

Table 2. Unadjusted initial pH of wastewater effluent amended with KH$_2$PO$_4$ at the start of the first trial of the batch study.

KH$_2$PO$_4$-amended Initial Wastewater Effluent Concentration (mg P/L)	Initial pH
0	6.8
5	7
30	6.6
50	6.4
100	6.6
200	6.2
500	5.7
1000	5.3

where C_i is the initial concentration of PO_4-P in solution, V is the volume of solution (0.045 L), and M_m is the mass of material (5 g). Following methods by White et al. (2021), experimental data were fit to non-linear Langmuir models using a spreadsheet developed by Bolster (2010), which uses non-linear least squares regression to predict sorption capacity.

Results and Discussion

Flow-through Column Study

In the column study, 97% of P was removed by RCA and 63% of P was removed by expanded clay as shown in Figure 2, representing a significant difference (p < 0.0001). The P load removed by RCA was 10.1 mg/d as compared to 10.0 mg/d removed by expanded clay. Percent P reduction did not vary significantly between the expanded slate (average 0.6% reduction) and gravel (average 4.4% addition). Expanded clay decreased in removal capacity over the 20-week duration (from 91 to 42% removal), while RCA maintained consistent removal. Jensen et al. (2022) achieved similar (up to 100%) P removal in columns containing a variety of calcareous materials, with P inputs ranging from 3 to 22 mg P/L. Ádám et al. (2007) demonstrated above 90% P removal from a 10 mg P/L-amended secondary wastewater solution within columns containing expanded clay, with average HRTs of around four

days. Potential contributing mechanisms of P removal include adsorption of phosphate ions due to high surface area and porosity (strong surface complex formation between Ca and Al compounds in RCA and with clay minerals in expanded clay), precipitation to form calcium phosphate minerals, ion exchange of hydroxide, chloride, or other ions, and surface complexation.

Columns were maintained at room temperature, with an average water temperature of approximately 21°C. The specific conductivity of effluent from columns containing RCA was higher than effluent from all other columns, as well as the influent (Table 3). This is likely due to leaching of ions, such as Ca, Mg, and bicarbonate, from the RCA, as has been documented in other studies (Engelsen et al. 2017). The ORP of effluent from columns containing RCA did not differ from columns containing expanded clay but was lower than the influent and effluent from columns containing expanded slate and gravel. The comparatively lower ORP values observed from effluent from columns containing RCA and expanded clay could be due to differences in surficial active sites which potentially allow for reduction of oxidizing agents in the water. Other studies have similarly observed increased P adsorption potential correlated with decreased ORP (Zhou et al. 2005; Andrés et al. 2018).

The pH did not vary between influent water and effluent coming from columns containing either gravel or expanded slate (Table 3; Figure 3), as
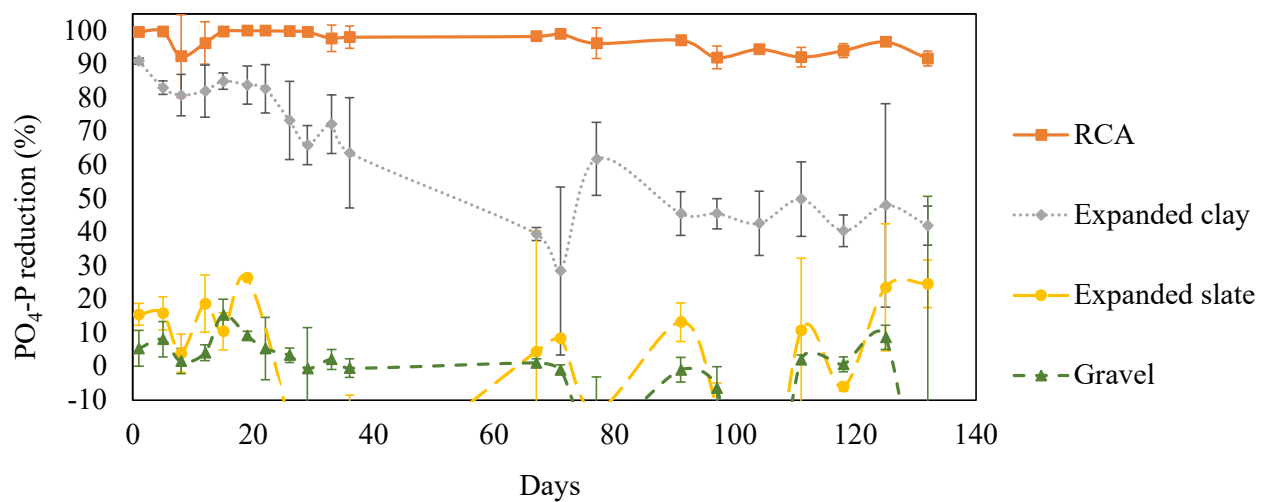
Figure 2. Average percent phosphate reduction for each material type over the duration of the flow-through study. Data points represented average percent reduction from three replicate columns as compared to the influent concentration with error bars representing standard deviation.

determined by statistical analyses. The effluent pH from columns containing RCA was greater than the effluent from columns containing expanded clay, which were both greater than the influent pH. The effluent pH from columns containing RCA decreased over the duration of the study (from 11.4 to 9.6), while effluent pH from columns containing expanded clay showed a decreasing trend over approximately the first month (from 9.4 to 7.8), followed by wider fluctuations of values over the remainder of the study. High pH values (up to 12.3) have been observed in other studies utilizing concrete, with pH generally increasing as particle size decreases (Gubernat et al. 2020). High pH values have also been observed from expanded clay within hybrid constructed wetlands, with effluent pH values between 8.1 to 8.8 observed in the first nine months of operation, then decreasing to 7.6 in the following three months (Põldvere et al. 2009).

Sorption Capacity Batch Study

The batch study revealed that the RCA and the expanded clay aggregate achieved the greatest modeled sorption capacities of the materials tested (Table 4; Figure 4). RCA achieved a 9.04 mg PO_4-P/g modeled maximum sorption capacity (R^2 =

Table 3. Average pH, specific conductivity, and oxidation reduction potential (ORP) values from influent and effluent water from flow-through column study, with standard deviation shown in parentheses. Treatments that share a letter are not significantly different ($\alpha = 0.05$).

Material or Sample Type	pH		Specific Conductivity (uS/cm)		ORP (mV)	
Influent	7.2 (0.35)	C	495.0 (131.4)	B	220.8 (42.7)	AB
RCA	10.6 (0.61)	A	777.8 (310.7)	A	109.9 (82.4)	C
Expanded clay	8.7 (0.64)	B	572.6 (193.1)	B	174.1 (66.7)	BC
Expanded slate	7.3 (0.21)	C	491.7 (160.4)	B	229.4 (79.2)	AB
Gravel	7.2 (0.22)	C	467.2 (158.5)	B	232.0 (70.0)	A
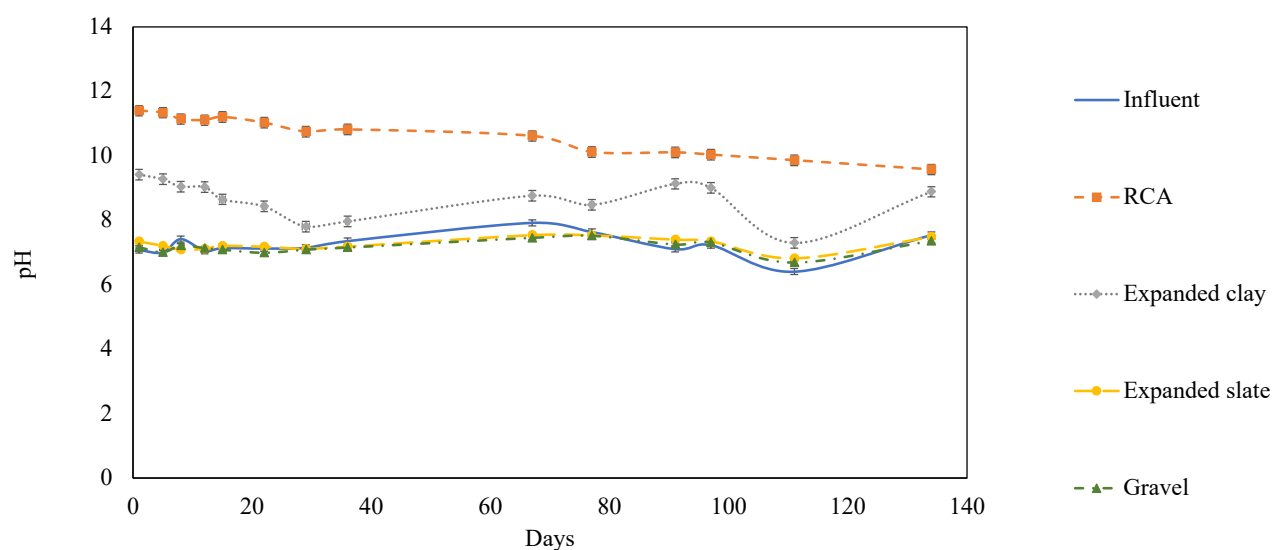
Figure 3. Average pH of influent and effluent (n = 3) water from columns over the duration of the flow-through study with error bars representing standard deviation.

0.99) and expanded clay achieved a 9.11 mg PO_4-P/g capacity ($R^2 = 0.98$) for the first experimental trial in which KH_2PO_4-amended wastewater pH was not adjusted. For the second experimental run of the batch study during which KH_2PO_4-amended wastewater pH was adjusted to the initial unamended wastewater pH, RCA achieved a 6.16 mg PO_4-P/g modeled maximum sorption capacity ($R^2 = 0.97$) and expanded clay achieved a 3.65 mg PO_4-P/g capacity ($R^2 = 0.93$). Both datasets for both experimental runs demonstrated good fit to the Langmuir model as indicated by the coefficients of determination. Coefficients of determination for expanded slate and gravel were negative, indicating that the Langmuir model is inappropriate for these data; therefore, modeled isotherms are not included in Figure 4. Differences in modeled maximum sorption capacities between the first and second experimental runs of the batch study highlight the impact that initial pH has on sorption capacity (as many studies have demonstrated), and the importance of adjusting pH for all tested P concentrations to ensure reliability of modeled sorption results (i.e., avoid confounding of experimental variables). Differences in modeled maximum sorption capacities between the first and second experimental runs of the batch study could also be attributed to differences in initial selected P concentrations in solution.

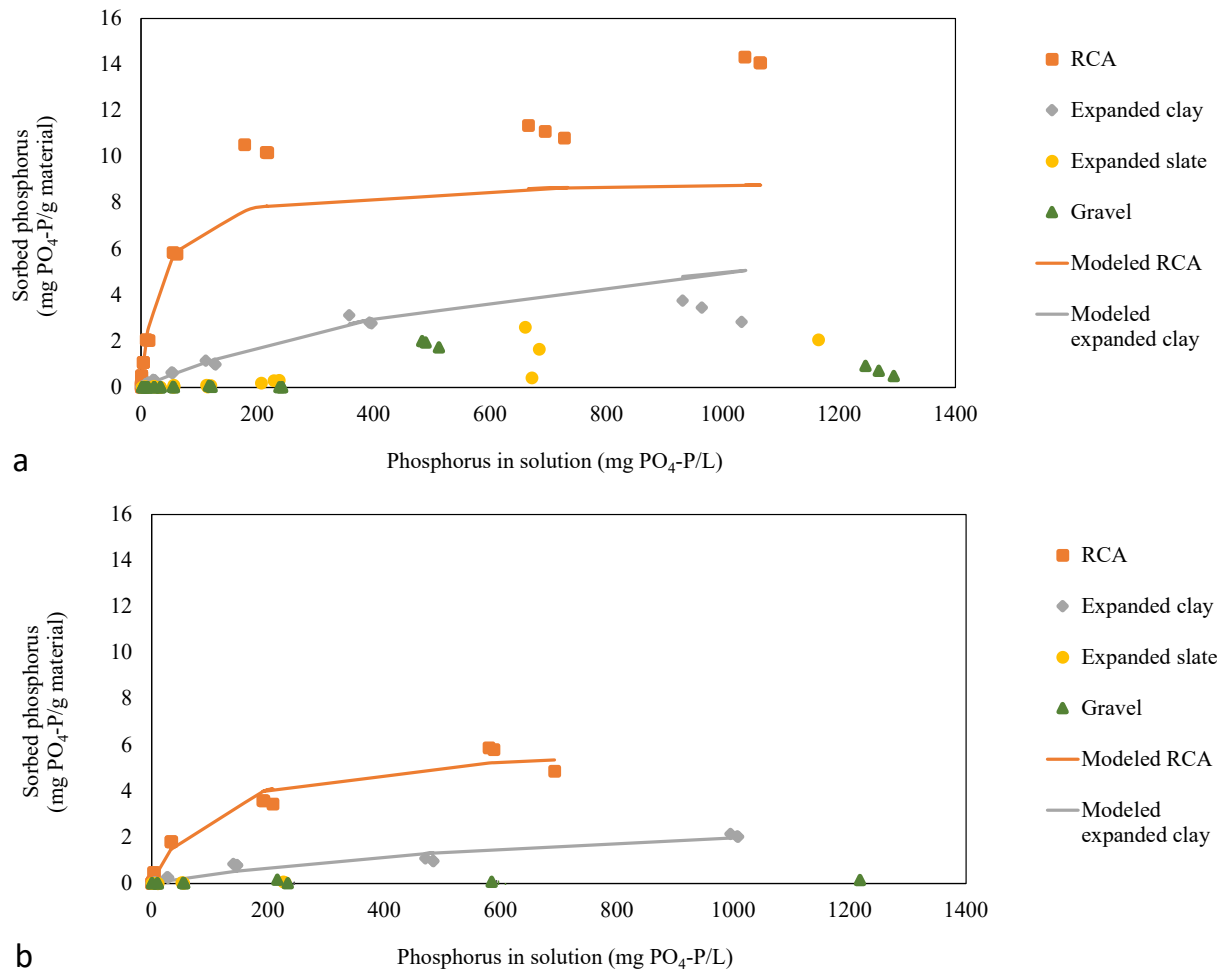The modeled maximum sorption capacities of the materials tested in this study are comparable to those reported in a review by Gubernat et al. (2020; Table 4). Of the seven low-cost materials that Boyer et al. (2011) evaluated in a series of jar tests and mini-column experiments, recycled concrete was among the best performing materials for phosphate removal; however, the increase of pH by greater than 2 units was noted as an undesirable secondary change that could negatively impact ecosystem health of receiving waters. In some instances, increased alkalinity in effluent filter water may actually be desirable. For example, the WWTP from which effluent was used in this study is interested in potentially using adsorptive filters to reduce P concentrations in sludge digestor decant water, a sidestream process during which this nutrient-dense water is recycled back to the head of the plant (J. Manning, personal communication, 2021). An increase in pH (and alkalinity) of P filter effluent that is routed to the head of the plant could contribute to the pH buffering capacity within the WWTP itself. In situations where effluent from P filters would be directly discharged to receiving waters, mitigation steps would potentially need to be put in place to decrease the effluent pH to within the allowable range for that specific receiving waterbody. Mitigation options could include dilution of the P filter effluent with WWTP effluent that was not treated through the P filter, or implementation of another treatment step.

Conclusion

Results from the flow-through column study indicate that the RCA and expanded clay materials effectively reduced P concentrations in P-amended WWTP effluent at a much lower HRT (higher flowrate) than has been reported in the literature (eight hours vs several days). The batch-scale study demonstrated that RCA and expanded clay also have the highest maximum sorption capacities of all materials tested in this study, according to the Langmuir model. RCA, the best performing material, was also associated with high effluent pH levels (10.6 on average). While such alkaline levels would not be suitable for release into the environment, this increased alkalinity may be useful in instances where P filter effluent could be recycled within a WWTP (i.e., to treat a sidestream process). These results highlight the potential of

Table 4. Phosphorus sorption capacities (C_{smax}, mg P/g) for recycled concrete aggregate (RCA) and expanded clay, according to the Langmuir model (current study denoted by asterisk), as compared to values reported in Gubernat et al. (2020) for similar materials.

Material Type	C_{smax} (mg P/g)
RCA	6.16*
Expanded clay	3.65*
Autoclaved concrete	0.28 – 70.90
Biochar (raw)	2.39
Lightweight aggregate	2.50 – 12.00
Sand	0.06 – 0.13
Zeolite	0.46 – 2.19

Figure 4. Phosphorus sorption isotherms of materials in phosphate-amended wastewater effluent for first experimental run of the batch study for which initial pH of KH_2PO_4-amended wastewater effluent was not adjusted (a) and second experimental batch study run for which initial pH of KH_2PO_4-amended wastewater was adjusted to unamended wastewater effluent pH using potassium hydroxide (b). Isotherm data were fit to a non-linear Langmuir model for RCA and expanded clay materials (solid lines).

recycled materials such as RCA to serve as a cost-effective add-on treatment technology to reduce P loads from WWTPs to receiving waters. These add-on technologies would not require substantial modification to existing infrastructure and would make beneficial reuse of a waste product.

The next steps for this study include investigation of the P desorption characteristics of adsorptive materials. Future work could also include exploration of P removal at various HRTs, temperatures, and starting P concentrations. Insight into mitigation measures to address increased pH of P filter effluent and other potential negative environmental impacts, as well as desorption

behavior of materials, could enable investigation of spent P filter materials as a potential substrate or soil amendment for plant growth. Reuse of recycled materials for incorporation into passive filters and then for plant growth would serve to close the gap from production to disposal, bringing us closer to a circular bioeconomy.

Acknowledgements

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Natasha Bell (corresponding author) is an assistant professor and professional engineer in the Department of Biological Systems Engineering at Virginia Tech. She was an assistant professor in the Department of Engineering at ECU when this study was carried out. Her research takes an ecological engineering approach to development of water remediation and reuse strategies that inform decision-making within the water-energy-food nexus. She aims to integrate research into the classroom through course-based undergraduate research experiences (CUREs) and community-based projects. She is a first-generation college graduate. She may be contacted at natashabell@vt.edu or by mail at 155 Ag Quad Lane, Blacksburg, VA 24061.

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