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Potential Water Treatment Residual Used for Eutrophication Prevention: Varying Bake Time and Wet-Dry Release Experiments

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Potential Water Treatment Residual Used for Eutrophication Prevention:

Varying Bake Time and Wet-Dry Release Experiments

Honors Research Project

Noor Fahoum

May 2021

ABSTRACT

A challenge faced today in water ways is the excessive growth of algae due to eutrophication. These harmful algal blooms create problems for the environment and safety concerns for the use of water for drinking purposes. An increase in limiting nutrients such as nitrogen and phosphorus are one of the factors that contribute to eutrophication. This is a concern for the Akron Water Treatment plant where preventative measures are being taken to avoid the potential formation of harmful algal blooms and the cyanotoxins they may release in the water. Through various studies, water treatment residuals (WTR) have been found to be effective at reducing phosphorus levels through adsorption. WTR are byproducts of drinking water treatment plants that can be reused to prevent harmful algae blooms. The two types of WTR used in these experiments are Al-WTR and PAC-WTR. Both WTR's contain aluminum sulphate but PAC-WTR has powder activated carbon to assist with removing organics, taste, and odor compounds in the water during the treatment process.

One set of research was conducted to look at the effect different bake times had on the phosphorus adsorption capacity of the WTR's. The results from these experiments showed that Al-WTR baked for 8 or 16 hours at 175 ℃ and PAC-WTR baked for 4 hours at 150 ℃ had the maximum increase in capacity of phosphorus adsorption. The other set of research analyzed the wet-dry release of the WTR in the case of a rain event. It was determined that there is no concern in regard to the WTR desorbing the phosphorus back into the environment during a storm event. These experiments were run to test two different variables that could optimize the adsorption of phosphorus and see the effect of a storm event to ensure the safety of using these WTR's in the environment.

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INTRODUCTION

Freshwater only makes up roughly 4 percent of the water that exists on earth's surface, yet it is the primary source of drinking water around the world. Water quality is important to ensure safe drinking water. One of the primary threats to water quality and contributor to its degradation is eutrophication (EPA, 2015). Eutrophication is caused by excess nitrogen and phosphorus in the water, which increases algae growth that leads to the depletion of dissolved oxygen negatively impacting ecosystems (EPA, 2015). Excessive algal blooms can reduce the sunlight reaching the bottom of freshwater beds, making it challenging for photosynthesis to occur for plants to sustain life. If the bloom contains cyanobacteria, it has the potential to release cyanotoxins and is often classified as a harmful algal bloom (Cheung, 2012). Harmful algal blooms can also lead to water borne diseases. Some of the leading contributors to eutrophication are agricultural runoff and combined sewer overflows. These human activities increase limiting nutrients such as nitrogen and phosphorus.

Lake Rockwell is the main water source used and treated by the Akron Water Treatment plant to provide drinking water to the City of Akron. Algal blooms and the presence of cyanobacteria typically occur yearly between the spring and summer. Higher temperatures from these seasons are the optimal conditions for blooms to occur including the amount of rain events that contribute to an increase in agricultural runoff (EPA, 2019). The runoff increases phosphorus and nitrogen levels used as food sources by algae and cyanobacteria. The Akron Water Treatment plant wanted to prevent potential harmful algal blooms from occurring by reducing the phosphorus levels in waterways prior to entering Lake Rockwell. The two inputs to Lake Rockwell are the Cuyahoga River and Eckert's Ditch. Eckert's Ditch is shallow, slow moving, and high in nutrients whereas the Cuyahoga River is fast moving, and nutrients are less

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concentrated-more dispersed. Eckert's Ditch is the waterway where efforts are being taken to reduce the phosphorus levels.

One of the ways to prevent harmful algal blooms from occurring is by reducing the phosphorus levels in the water. Water treatment residuals have been found through various studies to have an affinity for phosphorus (Carleton, 2019; Spade, 2020). Both alum based WTR (Al-WTR) and Al-WTR containing powdered activated carbon (PAC-WTR) are byproducts of the water treatment plant. They are used in the coagulation process of water treatment plants to filter out tiny particles and reduce the turbidity of the water (Water Plant Division). Through research conducted at The University of Akron on WTRs, it was found that Al-WTR and PAC-WTR were more effective at adsorbing phosphorus as-is compared to baked; though it had been hypothesized that baked WTR would increase adsorption capacity. The most effective temperature for Al-WTR was 175 ℃ and 150 ℃ for PAC-WTR but even then, as-is WTR performed best (Spade, 2020). The laboratory evaluation was used to test the effect of WTRs reducing excess phosphorus from reaching Lake Rockwell. Successful results led to the development of a full-scale system consisting of a series of gates, each containing four cartridges designed to contain sleeves that held the WTR allowing water to flow through at Eckert's Ditch (Carleton, 2019). Multiple isotherms were run to replicate the conditions of the environment from the movement to the temperature of the water. Conductivity, pH, and aluminum concentration were tested to ensure that the WTR was not altering the content of the water besides reducing the phosphorus concentration. Although both Al-WTR and PAC-WTR have an affinity for phosphorus, as-is PAC-WTR was found to be the most efficient at adsorbing phosphorus (Carleton, 2019; Spade, 2020).

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There were two objectives for this project; to determine the optimal baking time and to evaluate potential desorption during a rain event. To achieve objective 1, an experiment was run to assess the impact of different bake times on the adsorption capacity. It was hypothesized that the longer the WTR was baked, the more phosphorus it would adsorb. Both Al-WTR and PAC-WTR were baked for 2, 4, 8, 16, and 24 hours at their respective optimal temperatures. Isotherms were then conducted to see the effect these bake times had on the adsorption and desorption capacity.

As mentioned above, the second objective was to evaluate the wet-dry release of the WTR. During the summer, the water level is low which would cause some of the WTR in the cartridge system to be dry. When a rain event occurs, the WTR is soaked at an "instant" with water. This experiment was conducted to see how much phosphorus might be released from the WTR back into Eckert's Ditch. It was hypothesized that the WTR would desorb a small amount of phosphorus that would not contribute to a dramatic increase of the nutrient in the water.

MATERIALS AND METHODS

The following materials were used to conduct both the bake time experiment and the rain study: Water treatment residuals (Al-WTR and PAC-WTR), distilled water, potassium phosphate monobasic (KH2PO4), HACH handheld colorimeter, vials, phosphorus reagent packets, scale, isotherm, graduated cylinder, test tubes, beaker, foil, and an oven.

The following steps were taken for the bake time experiment:

1. To prepare for the tests, samples of both WTRs were baked in the oven for 2, 4, 8, 16, and 24 hours. Al-WTR was baked at 175 ℃ and the PAC-WTR was baked at 150 ℃ based on the results of Spade (2020). After the specified bake time, samples were

removed from the oven and allowed to cool to room temperature. Samples were transferred to amber bottles until the initiation of the adsorption-desorption experiment.

- 2. A 2.25 mg/L phosphorus (PO₄) stock solution was made by mixing 13.5 mL of 100 mg- PO_4/L , created with KH₂PO₄ and distilled water, with 586.5 mL of distilled water to produce 600 mL of the solution. Two sets of this were made to be enough to conduct a triplicate for each bake time. The max reading of the HACH handheld colorimeter is 2.50 mg/L PO₄ so a 2.25 mg/L PO₄ stock solution was made to ensure it could be read by the HACH handheld colorimeter and is a high concentration situation to assess how the WTR will perform.
- 3. The Al-WTR isotherm was run first by adding 1.6 g of the WTR to three sets of test tubes for each bake time.
- 4. Forty mL of the PO⁴ stock solution was added to each test tube. They were covered with a piece of foil.
- 5. The test tubes were labeled 1 through 15 to keep track of them when placed into the isotherm, which was static and set at 20 degrees Celsius to replicate the conditions at Eckert's Ditch.
- 6. After 24 hours, the 2-hour bake time triplicates were pulled out. Thirty-five mL of solution was poured out of each test tube for determining the adsorbed amount. Five mL were left in the test tube to prevent any of the WTR from pouring out with the solution collected. Then 35 mL of distilled water measured by the graduated cylinder was added back to the test tubes and then placed back in the isotherm to initiate the desorption step.
- 7. Ten mL of the 35 mL collected solution was poured in the blank vial and 10 mL was added to another vial for analysis of the PO₄ concentration.

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- 8. The phosphorus reagent was added to the non-blank vial and shaken for 15 seconds before setting a 2-minute timer for the vial to sit.
- 9. After 2 minutes, the blank vial was inserted into the HACH handheld colorimeter and zeroed. Then the vial with the phosphorus reagent added to it was read and the $PO₄$ level was recorded.
- 10. Steps 5 through 9 were repeated for the remaining bake times.
- 11. After 24 hours, the 2-hour bake time triplicates were removed to determine the amount of PO4 that desorbed from the WTR. Ten mL of the solution was poured in the blank vial and 10 mL was added to another vial. Steps 7 and 8 were repeated to record the PO⁴ that may have desorbed from the WTR into the distilled water. This step was repeated for the rest of the bake time tri-sets to conclude the bake time experiment on the Al-WTR.
- 12. After test tubes were cleaned and dried, steps 2 through 10 were repeated for the PAC-WTR and all data was recorded.

There are two values calculated from the data collected from this experiment that are used for further analysis of the results, c_e and q_e . c_e is the amount of phosphorus left in solution that was not adsorbed in the units of mg PO4/ L of solution. To calculate the amount of phosphorus adsorbed, the following equation can be used:

$$
Phosphorus\ adsorbed\ (\frac{mg}{L})=2.25\frac{mg}{L}-c_e
$$

2.25 mg/L is the initial concentration of phosphorus in the stock solution. *q^e* represents the milligrams of phosphorus adsorbed per kilogram of WTR. This value can be calculated as follows:

$$
q_e \left(\frac{mg}{kg}\right) = \left(2.25 \frac{mg}{L} - c_e\right) \times 0.004L \times \frac{0.875}{0.0016 \ kg}
$$

0.004 L represents the background volume since 40 mL of stock solution was added to start the experiment and that volume is maintained throughout the experiment. 0.875 is the fraction replaced at each timestep, as determined in Spade (2020). The 0.0016 kg represents the 1.6 g of WTR added at the start of the experiment in each test tube.

The following steps were taken for the rain study experiment:

- 1. Firstly, representative subsamples of Al-WTR from 'spent cartridges' were obtained. Spent cartridges correspond to those that had been used in the full-scale gate system in Eckert's Ditch. The WTR within the cartridges were replaced as the material was no longer adsorbing PO4.
- 2. The mass of a pan was recorded then the scale was zeroed to measure 220 g of the wet Al-WTR. The WTR was left to dry for 48 hours and then the Pan+WTR was weighed again to calculate the percent moisture existing in the WTR.
- 3. Raw (i.e., surface) water was collected from Eckert's Ditch. The PO⁴ concentration was determined using a HACH handheld colorimeter as described in the bake time experiment.
- 4. Thirty-five g of Al-WTR was added to six 300 mL beakers. The mass of each beaker was recorded to be able to subtract later and calculate the percent moisture of the WTR for each experimental set.
- 5. Three beakers had 200 mL of distilled water added and three beakers had 200 mL of raw water from Eckert's Ditch. This was repeated for all three trials of this experiment. Each beaker was then covered with foil and placed on a stir machine under a hood to recreate the environmental conditions.
- 6. At 30 minutes, 1 hr, 2 hr, 4 hr, and 24 hr 20 mL samples were taken and the PO_4 content was measured using the HACH handheld colorimeter.
- 7. After the 24 hr sample was collected, the residual water was removed from the beakers while keeping the Al-WTR. The beakers and the Al-WTR were weighed, and the weight was recorded.
- 8. The beakers and Al-WTR were left to dry for 48 hours before steps 4 through 6 were repeated.
- 9. After the second 'wet event', steps 4 through 7 were repeated again to yield data collected from three 'wet-dry' events. The Al-WTR and Beaker was weighed to record the final dry mass.

Triplicates were used for both experiments to calculate and analyze the average and standard deviation. This is used to conduct environmental analysis of the performance of the WTR.

RESULTS AND DISCUSSION

Impact of Bake Time on Adsorption-Desorption

The results from the bake time experiment partially proved the hypothesis that the longer the WTR was baked, the more phosphorus it would adsorb. An increase in bake time did contribute to an increase of adsorption of phosphorus in both Al-WTR and PAC-WTR. Yet for both, the 24 hour bake time did not create the greatest adsorption capacity in the WTR's. The results for the Al-WTR are shown in *Figure 1* below where *q^e* represents the milligrams of phosphorus per kilogram of Al-WTR and *c^e* represents the concentration of phosphorus left in the solution.

Figure 1 Al-WTR baked at 175℃ 24 hr adsorption 24 hr desorption experiment where red represents the 2 hr bake time, yellow is 4 hr, pink is 8 hr, green is 16 hr, and blue is 24 hr. The bars represent standard deviation. Triplicates were made for each bake time where raw water *from Eckert's Ditch was used to test adsorption and distilled water was used to test desorption.*

The bake time that allowed the WTR to adsorb the most, without desorbing as much when the distilled water was added, is represented by having a greater *q^e* value and a smaller *c^e* value. Out of all the bake times, the 16-hour bake time, represented by green symbols, performed best. The 16- hour bake time had a *q^e* of 40.03 mg/kg for Al-WTR, which was the highest amount of phosphorus concentration per kg of Al-WTR. After the first 24-hrs when the phosphorus solution was replaced with distilled water, the *q^e* only decreased by 1.35 mg/kg showing that it did not desorb much considering the amount it took in. The original concentration of the phosphorus solution was 2.25 mg-PO4/L which was reduced to 0.42 mg/L, *ce*, after the 24- hour adsorption. The distilled water, which had a concentration of 0 mg-PO4/L, had a *c^e* value of 0.11 mg-PO4/L after the 24-hour desorption period. The least effective bake time for the Al-WTR was the 24 hour bake time which had a q_e of 23.84 mg/kg during the adsorption step which was the lowest compared to all bake times. After desorption, it resulted in a q_e of 24.97 mg/kg. This slight increase after adding the distilled water could have come from the 10 mL of raw water left in the

solution to prevent the WTR from pouring out when the solution was replaced between the 24 hour adsorption and desorption test. Refer to the appendix *Table 2* for the data collected for each bake time.

 The results for the PAC-WTR are shown in *Figure 2* below where the 4- hour bake time, represented by the yellow symbols, allowed the WTR to adsorb the most while desorbing the least.

Figure 2 PAC-WTR baked at 150℃ 24 hr adsorption 24 hr desorption experiment where red represents the 2 hr bake time, yellow is 4 hr, pink is 8 hr, green is 16 hr, and blue is 24 hr. The bars represent standard deviation. Triplicates were made for each bake time where raw water from Eckert's Ditch was used to test adsorption and distilled water was used to test desorption.

Unlike Al-WTR, the 4-hour bake time had the highest q_e value of 38.65 mg/kg, adsorbing the most phosphorus. The *q^e* value only decreased by 0.33 mg/kg and had a *c^e* value of 0.07 mg-PO4/L when the distilled water was added for the 24 hr desorption. That was the lowest amount of phosphorus desorbed. Although this *c^e* value was the same for the 24-hour baked PAC-WTR, the 24-hour WTR did not adsorb nearly as much, as shown by a q_e value of only 28.22 mg/kg. The similar c_e values between the 4-hour and 24-hour bake time could be due to the varying ability to hold on to phosphorus. The 4- hour bake time was able to adsorb more and hold on to

the phosphorus much stronger in the distilled water than the 24-hour bake time. This shows that the 4-hour baked PAC-WTR has a greater affinity for the phosphorus than the 24-hour baked PAC-WTR. Refer to the appendix *Table 3* for the data collected for each bake time.

 Between the two bake times that performed best for Al-WTR and the PAC-WTR, the 16 hour baked Al-WTR had the greater phosphorus adsorbed during the 24-hour adsorption experiment, with a *q^e* of 40.03 mg/kg-Al-WTR. The 4 -hour baked PAC-WTR had a *q^e* of 38.65 mg/kg-PAC-WTR which shows that it adsorbed less from the raw water compared to the Al-WTR. The PAC-WTR did desorb less with a c_e value of 0.07 mg/L whereas the Al-WTR had a slightly higher *c^e* value of 0.11 mg/L. Both WTRs performed well in the adsorption and desorption phase of the experiment with limited variability in their results.

Impact of Rain Events on Potential WTR Desorption

 For the Al-WTR rain study, the mass of the WTR was taken before and after the 48-hour drying periods of all three trials to calculate the moisture content as shown in *Table 3* through **Table 5** of the appendix. The average phosphorus concentration in the raw water from Eckert's Ditch used in all three experiments is shown in *Table 7*, *Table 9*, and *Table 11* of the appendix. The summary of the average and standard deviation of the results from all the consecutive wetdry release of the WTR can be seen in *Table 1* below.

Table 1 The averages and standard deviation values of phosphorus levels, in mg/L, for each wetdry experiment in distilled water (DI) and raw water (RW). The phosphorus levels in solution can be seen from 30 min to 24 hr of the WTR being in wet conditions. Between each experiment, the WTR was dried for 48 hours.

	30 min		1 ^{hr}		2 _{hr}		4 hr		24 _{hr}		
	Experiment Calculation	DI	RW	DI	RW	DI	RW	DI	RW	DI	RW
	Average	0.12	0.027	$\overline{0}$		$\overline{0}$	θ	0.027	0.0033	0.15	0.06
	SD	0.09	0.038				0	0.038	0.0047	0.025	0.045
	Average	0.21	0.15	0.053	0.05	0.017	0.03	0.043	0.077	0.073	0.05
↑	SD	0.10	0.11	0.038	0.064	0.012	0.022	0.033	0.0047	0.039	0.0082
	Average	0.073	0.26	0	0.15	0	0.097	0.06	0.077	0.11	0.1
$\mathbf{\Omega}$	SD	0.054	0.18	$\overline{0}$	0.15	0	0.071	0.078	0.056	0.034	0.051

 The first experiment looked at how dried WTR would respond to an initial rain event (i.e., 200 mL of water added to beaker). Data in row 1 of **Table 1** showed how in distilled water, the WTR desorbed 0.12 ± 0.09 mg/L phosphorus in the first 30 minutes but adsorbed phosphorus in the raw water that had an initial concentration of 0.30 mg/L PO_4 . This occurs because the raw water has an initial concentration of phosphorus, whereas the distilled water has no PO₄ to bind with so the WTR is willing to release some of what it has already bound. By the $24th$ hour, the WTR desorbed a greater amount of phosphorus in the distilled water, but adsorbed PO₄ from the raw water decreasing the original concentration from the starting concentration from the previous 4 hr PO⁴ concentration of 0.0033±0.0047 mg/L. As described in step 8 of the procedure, the WTR was allowed to dry before water was added to simulate a second rain event. For the second rain simulation, the WTR seems to release more $PO₄$ into the distilled water. For instance, after 30 minutes and 1 hr the PO₄ concentrations were 0.21 ± 0.10 mg/L and 0.053 ± 0.038 mg/L, respectively. However, by the $24th$ hour some of that PO₄ was readsorbed. In the raw water it adsorbs even more PO⁴ compared to the first experiment. The final experiment showed that the WTR was not releasing PO_4 back into the raw water in the first 30 minutes as the PO_4 concentration was 0.26 ± 0.18 mg/L, which was less than the starting concentration of 0.29 mg/L

as shown in **Table 11** of the appendix. Not as much PO⁴ was desorbed in the distilled water which had a concentration of 0.073 ± 0.054 mg/L after the first 30 minutes. By the 24th hour, the PO_4 level of 0.1 ± 0.051 mg/L in the raw water was still less than the original concentration, and PO₄ was still present in the distilled water with a concentration of 0.11 ± 0.034 mg/L. These three experiments of drying the Al-WTR for 48 hours and then wetting by adding either distilled or raw water show that the Al-WTR goes through a pattern of binding and releasing PO₄ into the solution. It seems that the WTR will desorb more when in distilled water compared to raw water, which is more realistic to the environmental conditions. The end result aligns with the hypothesis of being effective with adsorbing despite the wet-dry conditions. The desorption that occurred seems to be reversed over time, and the phosphorus levels after 24-hours in the raw water were always less than originally started with. This indicates that the low water conditions that cause the Al-WTR to dry up, and the rain events that instantly soak the WTR, are not a concern when it comes to releasing the PO_4 back into the environment. This demonstrates the impact of concentration gradient balance across media, in this case between the water and the WTR. Natural environments move towards some sort of balance which is what is seen here as the WTR adsorbed and released phosphorus during the different stages rather than continuously adsorbing phosphorus (Spade, et al., 2020). The data for each trial of this experiment can be found in *Table 8*, *Table 10*, and *Table 12* of the appendix.

CONCLUSION

 Eutrophication is a growing concern in water sources all over the world. Harmful algal blooms are becoming reoccurring events that affect water quality and create a health risk for communities. Various human activities have contributed over time to the increase of nutrients in water ways. An increase in phosphorus in these environments has been found to cause greater

amounts of algal blooms to occur. WTRs, which are already a byproduct from Water Treatment plants, can reduce excess phosphorus and have the benefit of reusing what would have been sent to a landfill as waste. This is a sustainable approach to ensuring safe water quality as seen implemented at Eckert's Ditch.

 It has already been proven that WTRs are effective at reducing phosphorus levels. The purpose of the bake time experiment was to show the potential benefit of baking the WTR for different periods of time on its capacity for phosphorus adsorption. The results showed that the 16-hour baked AL-WTR performed best by adsorbing the most phosphorus and desorbing the least. The 4-hour baked PAC-WTR had little variability in comparison to the AL-WTR. If time or the energy cost required to heat the WTR is a concern, the 4-hour PAC-WTR will still be effective at reducing the phosphorus levels in the water. The rain study showed that the WTR is capable of adsorbing phosphorus despite the wet-dry events that may occur. The experiments also showed that there is no great release of phosphorus back into the environment during an instant rain event.

 By knowing WTRs ability to bind phosphorus, they can be implemented in various ways to reduce eutrophication and the potential occurrence of harmful algal blooms. Since WTRs are typical byproducts from water treatment plants, they are easily accessible for areas that contribute to the factors that increase harmful algal blooms. WTRs can be implemented in water ways before the water reaches water treatment plants and can be used in systems that treat agricultural runoff before reaching any body of water. This can contribute to improving water quality not just in Akron, but all over the world, as eutrophication is a growing worldwide concern as human activities continue to impact water sources.

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RECOMMENDATIONS

There are various experiments that can be done to optimize the use of WTR's and determine what variables, when altered, allow them to adsorb more phosphorus. The bake times conducted for the first set of research were only between 2 hours and 24 hours. Other times between this range and beyond it should be tested to see if baked WTR may have a greater affinity for phosphorus than as-is WTR. There are also other WTR that have different iron compounds added to them that can be tested to determine what is ultimately best at reducing phosphorus and has the greatest capacity to do so. For the rain study, it is recommended that more trials of this type of experiment are run with extended periods of time beyond 24 hours. This can help determine the point at which the WTR is no longer able to adsorb effectively or desorbs a great amount of phosphorus back into the environment making it ineffective. Based on the results from the bake time experiments, it is recommended that the Akron Water Treatment plant uses as-Is WTR to have the most optimal phosphorus adsorption until further research is conducted to determine another potential bake time that may have a higher adsorption capacity in comparison.

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APPENDIX

Bake Time Experiment:

Table 2 The data corresponding to the Al-WTR bake time experiment and graphed on Figure 1.

Table 3 The data corresponding to the PAC-WTR bake time experiment and graphed on Figure 2.

Rain Study Experiment:

Table 4 The initial mass of the Al-WTR from the "spent cartridges" and the mass after 48 hr of drying.

Table 5 The initial amount Al-WTR added to each beaker and the mass of the beakers.

	Beaker	WTR added
DI#1	111.94	35
DI#2	104.25	35
DI#3	117.86	35.01
RW#1	117.91	35.01
RW#2	121.24	35
RW#3	110.71	35

Table 6 The mass of the Al-WTR + beaker after the residual solution was removed and after 48 hours of drying after each trial with the moisture content calculated.

Eckerts Ditch Upstream (Raw water)			
Sample	Phosphorus, mg/L		
	0.31		
	0.30		
	0.28		
Average	0.30		

Table 7 The phosphorus levels in the raw water collected from Eckert's Ditch for the first trial.

Eckerts Ditch Upstream (Raw water)			
Sample	Phosphorus, mg/L		
	0.39		
	0.24		
	0.25		
Average	0.29		

Table 11 The phosphorus levels in the raw water collected from Eckert's Ditch for the final trial.

