TREATMENT OF MICHIGAN WINERY WASTEWATER WITH GRAVEL BED VERTICAL FLOW CONSTRUCTED WETLANDS

Katelyn Skornia[1], Younsuk Dong[1], Umesh Adhikari[1], Steven Safferman[1]

ABSTRACT

Intermittent production and high concentrations of biochemical oxygen demand (BOD), ammonia, and soluble phosphorus (SP) make winery wastewater challenging for on-site treatment. Currently, many Michigan wineries use land application for wastewater management, but new regulatory recommendations require more land so a compact alternative is desirable to prevent the loss of vineyard space to wastewater treatment area. To reduce treatment area, this study investigated the use of gravel bed vertical flow constructed wetlands (GBVFCWs) in removing high concentrations of BOD, nitrogen, and SP from winery wastewater. The investigated GBVFCWs consist of three subsurface gravel cells connected in series that utilize aerobic and anoxic conditions to promote biological degradation. The addition of SP adsorption media removes high SP concentrations from the effluent wastewater. In this study, a bench-scale GBVFCW exhibited rapid performance when inoculated with secondary effluent from a domestic wastewater treatment facility prior to winery wastewater flow. At 68°F and at various loading frequencies, the GBVFCW removed an average of 99% COD (used as a proxy for BOD), 62% nitrate, 94% total nitrogen, and ammonia to levels below detection limits. Nearly all treatment occurred within the first cell, indicating that aerobic and anoxic environments were present within the cell. The SP adsorption media, PO4Sponge by MetaMateria (Columbus, OH), removed 99.8% of total phosphorus from the effluent wastewater. Additionally, results indicate that performance of the system is not impacted at a reduced temperature of 50°F. A HYDRUS Constructed Wetland 2D model is being evaluated for its potential use in this application. Based on this research, GBVFCWs are a compact and effective option for winery wastewater treatment.

INTRODUCTION

In 2018, there were nearly 150 wineries that produced more than 2.7 million gallons of wine in Michigan, resulting in this industry being the fifth largest in the United States (Michigan, 2019). Further, Michigan wineries are popular tourist destinations with more than 1.7 million visitors each year (Michigan, 2019).

More than 7 gallons of wastewater is produced to make 1 gallon of wine (Turner, 2010). The characteristics of this wastewater can vary greatly, as shown by data from five Michigan wineries in Table 1. Because this wastewater is considered high strength and most Michigan wineries are on small plots of land, traditional onsite wastewater treatment may be difficult to fit on the site. Meeting the recently established Michigan Department of Environment, Great Lakes, and Energy (EGLE) maximum loading rate of 50 lb BOD/acre/day requires a significant amount of land that may reduce area available for vineyards and negatively impact winery profitability. Alternatives have been examined but the periodic nature of wine production and the likelihood of substantial

[1] Michigan State University, Department of Biosystems and Agricultural Engineering, East Lansing, MI 48824

flows in late autumn add to the challenge of finding effective and affordable wastewater treatment options.

Table 1. Winery Wastewater Composition

Parameter (mg/L)	Five Michigan wineries (Lakeshore, 2015)		
	Average	**Minimum**	**Maximum**
Chemical Oxygen Demand (COD)	3,236	493	5,722
Biochemical Oxygen Demand (BOD_5)	2,046	336	3,578
pH	6.2	5.5	6.8
Total Phosphorus (TP)	5.26	1.29	9.19
Total Nitrogen (TN)	7.60	2.63	18.5

The gravel bed vertical flow constructed wetland (GBVFCW) treats diverse sources of high strength waste. It has proven to be effective by over 60 installations of a similar design by Aqua Treatment Technologies, some of which have been operating for nearly 20 years (AQUA, Projects). GBVFCWs treat wastewater biologically in three sub-surface gravel cells. A layer of soil above the GBVFCW provides insulation for subsurface wastewater application, preventing freezing. All microbial processes occur within the lined cells preventing any chance of metal mobilization from anaerobic conditions as a result of the application of high BOD wastewater (Safferman, 2011). After treatment, wastewater is discharged into drain fields, filter strips, or used for irrigation. This type of wetland has previously been researched for its utility in treating high strength milking facility wastewater (Campbell, 2015) and is now the basis for a Natural Resource Conservation Service (NRCS) standard (Michigan Gravel Contactor for Treating Milking Center Wastewater).

Phosphorus is not biologically degraded or removed in a GBVFCW. Gravel within the cells will remove phosphorus via adsorption but will become exhausted over time due to the limited adsorption sites on the material (Campbell, 2015). Complete and continuous removal of this parameter must be achieved for adequate wastewater treatment and is especially important if subsurface discharge is into groundwater that rapidly progresses to surface water. MetaMateria Technologies (Columbus, OH) manufactures an engineered media, PO4Sponge, that uptakes phosphorus. This media can be regenerated and reused or directly applied as a fertilizer.

It was hypothesized that a GBVFCW combined with adsorption media to remove and recover phosphorus will effectively and efficiently treat winery wastewater so that it can be discharged without impact to the environment. Consequently, the objective of this project was to conduct a bench-scale evaluation of this integrated system and use the collected data to develop design criteria for the winery industry. Further, a mathematical model of the system was examined.

MATERIALS AND METHODS

Studies and Phases

This project consisted of two separate studies. A column study (Column Study) investigated the use of a GBVFCW to treat winery wastewater under various conditions. The use of PO4Sponge assessed removal of total phosphorus from treated effluent (Phosphorus Removal Study). These studies used process wastewater collected from a local winery. Samples from the experimental treatment systems were collected and tested two to three times per week. Experimental treatment systems and flow rates through the systems were maintained and monitored weekly. In the Column Study, each column was inoculated one week prior to operation with secondary effluent wastewater to establish a microbial community within the columns.

Different operating conditions, in distinct phases, were tested in the Column Study. During the first phase, wastewater was distributed into the GBVFCWs four times a day at 8 am, 11 am, 2 pm, and 5 pm. This schedule was chosen to simulate the frequency of wastewater production at a winery. Subsequent phases and the Phosphorus Removal Study were loaded with wastewater at 6-hour increments throughout the day, simulating the discharge at a winery with an equalization tank. Wastewater was loaded into the system at an organic loading rate of 1.06E-2 lb COD/ft^2/day. This loading rate, as well as a maximum hydraulic loading rate of 0.504 gal/ft^2/day, were previously determined to be optimum for a GBVFCW.[7] Hydraulic loading rates were not a limiting design factor, but were checked to ensure that biofilm was not sloughed off during loading. Assuming a conservative COD concentration of 6,000 mg/L, 7 gallons of wastewater produced per 1 gallon of wine, and 750 mL of wine in a bottle, this organic loading rate results in a GBVFCW with a surface area requirement of 6.5 ft^2 per bottle of wine produced per day. These operating conditions for the studies and phases are summarized in Table 2.

Table 2. Project Studies and Phases

Study	Description	Phase	Operating Conditions
Column Study	Evaluation of wetland performance on various loading conditions	Phase 1: Normal operating conditions	Room temperature (68˚ F), uneven loading frequencies, loading rate of 1.06E-2 lb COD/ft^2/d Columns inoculated with domestic secondary effluent wastewater prior to operation
		Phase 2: Even loading frequency	Room temperature (68˚ F), even loading frequencies, loading rate of 1.06E-2 lb COD/ft^2/d
		Phase 3: Reduced temperatures	Reduced temperatures (50˚ F), even loading frequencies, loading rate of 1.06E-2 lb COD/ft^2/d
Phosphorus Removal Study	Evaluation of PO4Sponge performance in phosphorus removal from winery wastewater	N/A	Room temperature, even loading frequencies, flow rate of 3 mL/min for 13.92 min 4 times per day

Experimental Design

In the Column Study, a wetland system of three columns simulated a GBVFCW with each column representing a cell. Only the surface area was scaled down, resulting in 4 foot tall columns with the subsurface inlet of wastewater 1.5 feet below the top of the column (representing ground level). The diameter of the columns was scaled to 4 inches; research has shown that columns should have a diameter that is ten times the diameter of the largest particle size used in the column in order to minimize wall effects (Radolinski, 2018). All columns were filled with gravel, 0.25 inch in diameter. A 1.5 inch thick layer of river rock, 0.75 inches in diameter, at the bottom of each column kept the gravel from blocking the bottom inlet/effluent hose barb. Both the gravel and river rock were cleaned prior to use. The columns were constructed of polyvinyl chloride (PVC) pipe, placed vertically, and sealed at the bottom with a PVC cap. A hose barb fixed into the PVC cap served as the outlet for effluent wastewater in Columns 1 and 3, and as the inlet for wastewater in Column 2.

A single wetland system is shown in Figure 1. Masterflex noeprene tubing conveyed the flow of wastewater through the system. Influent wastewater was introduced into the first column through an inlet barb, 1.5 feet below the top of the column, and flowed top-down to promote aerobic conditions. Effluent wastewater from the first column was then either pumped back into the top of the first column (recycled) or into the bottom of the second column. The recycling ratio was maintained at 3:1 with three times as much wastewater going into the first column as the second, based on previous unpublished research. Filling the second column from the bottom resulted in water saturation and an anoxic environment within the second column. Effluent wastewater from the second column was then pumped to the top of the third column, which served as a polishing column and had aerobic conditions. Treated effluent wastewater flowed out of the bottom of the third column into a collection bottle. The influent and effluent wastewater pumps, Pump 1 and Pump 4 (Figure 1), operated on timers set to run four times throughout the day. The recycle pumps operated on float switches to maintain the 3:1 recycle ratio of wastewater from Column 1.

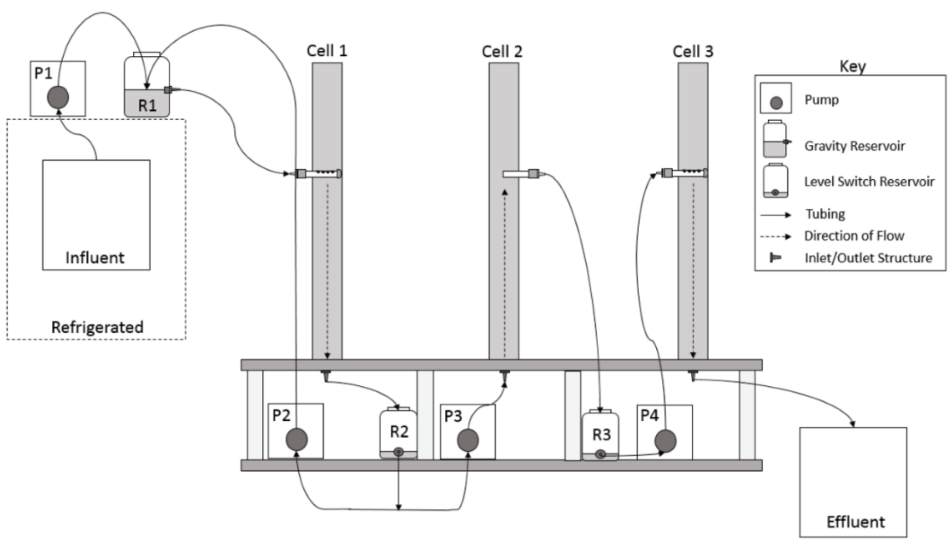

Figure 1. Bench-Scale Experimental Wetland (Campbell, 2015)

Plants were not investigated in this research as nutrients are not continuously taken up in cold climates during winter. Additionally, the subsurface application of the wastewater in a GBVFCW occurs below the root zone of most wetland plants. As such, it was determined that excluding plants would result in a more conservative design.

The Phosphorus Removal Study used 1.5 inch diameter PVC columns sealed at the bottom with a PVC cap to contain the PO4Sponge. A 1 inch layer of Vigoro pea gravel at the bottom of the columns prevented washout of the media through the effluent hose barb; the effect of the type of pea graval on phosphorus removal was not investigated in this study. One column served as the control and contained only the gravel layer (Control). Two columns contained the gravel layer and 90 mL of PO4Sponge (Test and Replicate). Treated effluent wastewater from the Column Study was supplemented with monopotassium phosphate to match the average phosphorus concentration of the untreated wastewater. This wastewater was pumped into the top of the columns directly onto the adsorption media (Figure 2).

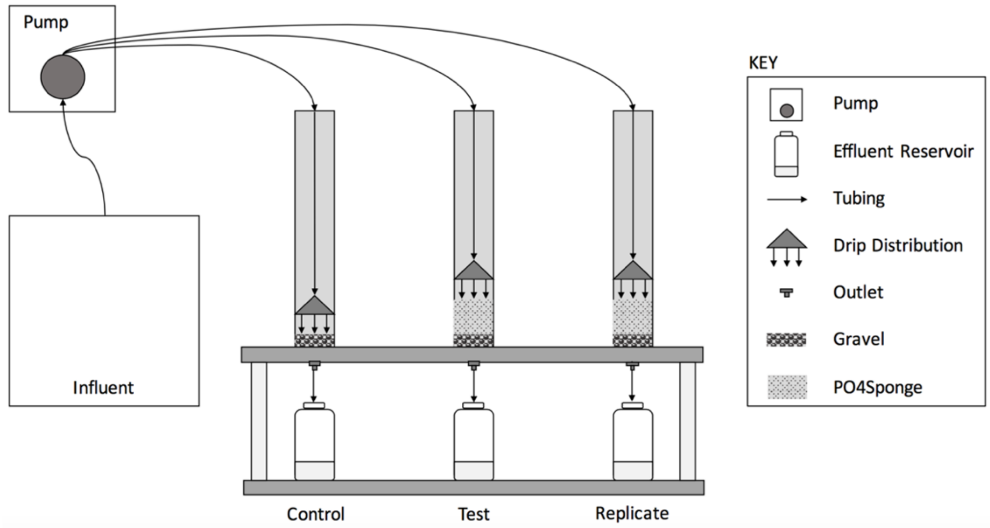

Figure 2. PO4Sponge Experimental Design

Previous research has demonstrated that PO4Sponge, a granular engineered adsorption media, can reduce total phosphorus from concentrations of 1 mg/L to less than 0.3 mg/L (Safferman, 2015). This study is unique as winery wastewater had not been previously tested. The quantity of PO4Sponge was determined following the manufacturer recommendation for empty bed contact time (a function of the amount of adsorption media and the flow rate of wastewater through the adsorption media) for the specific influent concentration of total phosphorus.

Testing

Grab samples for each study were collected immediately following wastewater loading into the treatment systems. Samples were then refrigerated or tested immediately. If a sample was not tested within 24 hours of collection, it was preserved with concentrated sulfuric acid and tested within 28 days of preservation. Preserved samples were neutralized with 5 N sodium hydroxide solution before testing. Testing followed standard procedures from HACH (Table 3).

Table 3. Testing Methods

Test	Method	Detection Range	HACH
Phosphorus, Total (HR)	Ascorbic Acid	0.5-5.0 mg/L PO_4-P	10209
Phosphorus, Total (ULR)	Ascorbic Acid	10-500 µg/L PO_4-P	10209
Chemical Oxygen Demand	Reactor Digestion	20-1,500 mg/L	8000
Nitrogen, Total	Persulfate Digestion	1-16 mg/L	10208
Nitrogen, Nitrate	Dimethylphenol	0.2-13.5 mg/L NO_3-N	10206
Nitrogen, Ammonia	Salicylate	1-12 mg/L NH_3-N	10205
pH	pH Probe	1-13	Calibrated Probe

RESULTS AND DISCUSSION

The total percent removal by the system for each parameter and phase in the Column Study is given in Table 4. Change in pH was also measured; the average influent values were 4.62, 5.12, and 5.30 and the average effluent values were 8.07, 8.01, and 8.14, in Phases 1, 2, and 3, respectively.

Table 4. Total Removal

	Phase 1	**Phase 2**	**Phase 3**
Total Phosphorus	99.9 %	99.2 %	99.7 %
Chemical Oxygen Demand	99.6 %	99.4 %	99.7 %
Nitrogen, Total	93.4 %	94.0 %	93.6 %
Nitrogen, Nitrate	61.7 %	63.7 %	59.4 %
Nitrogen, Ammonia	100 %	100 %	100 %

Column Study

Total Phosphorus

The average influent concentrations for Phases 1, 2, and 3 were 18.4 mg/L P, 22.2 mg/L P, and 26.7 mg/L P, respectively. These concentrations were reduced to below the detection limit of 0.5 mg/L in 96% of all final effluent samples. Of the total removal, an average of 83%, 76%, and 68% removal occurred in the first column during Phases 1, 2, and 3, respectively.

The main mechanism of removal of TP was adsorption to the gravel in the columns. Although the biofilm uptakes some phosphorus, it is only removed if the biofilm sloughs from the system and is therefore negligible in comparison to adsorption to the gravel. This removal mechanism is unlikely to be influenced by varying the application times or reducing the temperature, as shown in Table 4. Although this study did not find the breakthrough point of phosphorus adsorption to the gravel, previous research on GBVFCWs have shown that eventually the adsorption capacity of the gravel will be reached.[7] Due to the higher concentrations of total phosphorus found in winery wastewater, an alternative means of phosphorus removal, such as PO4Sponge, is critical.

COD

The average influent concentration of COD varied throughout the study but overall stayed between 5,000 to 6,000 mg/L. Phase 1 had an average influent concentration of 6,189 mg/L, Phase 2 was 4,997 mg/L, and Phase 3 was 5,851 mg/L. Despite the varying influent concentrations, all effluent samples were below 40 mg/L and 32% of measurements were below the detection limit of 20 mg/L. Of the total removal, an average of 89%, 96%, and 95% removal occurred in the first column during Phases 1, 2, and 3, respectively.

There is no clear influence of phase on the removal of COD from the entire system; all phases exhibited greater than 99% removal. The main mechanism of removal of COD is microbial activity, which can occur in both aerobic and anoxic conditions. Microbial activity in Column 1 reached an equilibrium within 11 days of the initial wastewater loading. Reduced treatment in Column 1 during this start-up period did not impact the final effluent concentrations from Column 3.

Nitrogen, Total

The influent concentration of TN varied substantially throughout the study. The average influent concentration of each phase was 33.8 mg/L N, 37.2 mg/L N and 27.7 mg/L N. This variation was likely a result of microbial degradation within the influent container and subsequent efforts were made to maintain the nitrogen levels by supplementing the wastewater with ammonium chloride. However, the varying influent concentrations did not have a large impact on the performance of the treatment system as the average effluent concentrations were 2.25 mg/L in Phase 1, 1.97 mg/L in Phase 2, and 1.55 mg/L in Phase 3. An average of 71% of total removal occurred within the first column of each system during Phase 1, 79% during Phase 2, and 86% during Phase 3.

Overall, there was no clear trend of the influence of the phase on system performance. TN decreased throughout the system as a result of microbial activity within the columns. In the aerobic conditions of Columns 1 and 3, TN nitrogen decreased due to nitrification. Some denitrification also occurred in the first column as a likely result of pockets of anoxic environments within the aerobic columns. TN decreased in the second column of each system as a result of the anoxic conditions that was caused by the saturated cell. Residual TN in the final effluent of each system is expected to be nitrate and organic nitrogen.

Nitrogen, Nitrate

Influent concentrations varied widely throughout the study resulting in varying effluent concentrations. Average influent concentrations were 3.09 mg/L N in Phase 1, 7.84 mg/L N in Phase 2, and 2.43 mg/L N in Phase 3. Overall, the second column behaved as expected as the concentrations of nitrate in the second column effluent of each system was reduced by an average of 96%, 96%, and 93% in Phases 1, 2, and 3, respectively. However, nitrate increased in the third column, resulting in low total percent removals, shown in Table 4. Despite this increase, effluent concentrations never exceeded 2.75 mg/L N.

Final effluent concentrations were consistent across the three phases. Increases in nitrate concentration in the final effluent were more influenced by higher influent concentrations than by phase. Unexpectedly, nitrate was also reduced in the first column which was aerobic. This removal indicated that anoxic conditions were present, likely due to the heterogeneity of gravel and the growth of biofilm within the columns. The saturated environment in the second column allowed for nearly complete removal of nitrate by denitrification. Nitrate increased through the third column due to nitrification of any residual ammonia in the wastewater.

Nitrogen, Ammonia

Ammonia was removed completely and immediately by the first column to a concentration below the detection limit of 1 mg/L N. This was true regardless of the influent concentration which averaged 14.6 mg/L N in Phase 1, 12.7 mg/L N in Phase 2, and 11.5 mg/L N in Phase 3, and spiked as high as 29 mg/L N. Effluent from Column 2 had detectable levels of ammonia in Phases 2 and 3, however, this was always completely removed in Column 3. All final effluent samples collected during the study were below the detection limit. Consequently, this resulted in an average of 100% removal in each system and phase, as shown by Table 4.

There was no apparent impact of the phase on the final concentrations of ammonia. Regardless of wastewater application frequency or temperature, the final effluent concentrations of ammonia were below 1 mg/L N. Ammonia was removed in the first and third column of each system by nitrification, which was a result of the aerobic conditions present in the columns. The increase in ammonia through the second column was hypothesized to be a result of moderate nitrogen fixation by free-living bacteria within the columns. This was not of concern as the final effluent concentrations were consistently below detection limits.

pH

The average influent pH for Phases 1, 2, and 3 was 4.62, 5.12, and 5.30, respectively. The pH of the wastewater increased throughout the system with the majority of the increase occurring in the first column (an average increase of 51%, 45%, and 32% during Phases 1, 2, and 3, respectively, compared to the total increases of 75%, 58%, and 54%). Although this represents an overall decrease between phases, there was an increase in the pH of the influent water. Together, these resulted in similar effluent values. The average effluent for each phase was 8.07, 8.01, and 8.14.

Typically, nitrification results in a decrease in pH levels in wastewater. In this study, however, the pH levels increased with both nitrification and denitrification of the wastewater, which is hypothesized to have occurred because the gravel in the columns acts as a pH buffer, helping to stabilize the wastewater at a neutral pH. Although the exact composition of the gravel in this study is unknown, limestone and other calcium carbonate rocks are commonly used as pH buffers and are often found in commercial gravel.

Phosphorus Removal Study

Results of the Phosphorus Removal Study were consistent with previous studies in removing TP concentrations down to low levels. As was expected, the control column, which only had a 1-inch layer of gravel, removed negligible amounts of phosphorus. The Test and Replicate columns of PO4Sponge performed better than expected and removed high levels of TP from an average influent concentration of 17.4 mg/L P to less than 0.12 mg/L P TP. This is significantly lower than the expected value of 0.3 mg/L P. In 84% of the Test and Replicate samples, the effluent concentrations were less than or equal to 0.06 mg/L P. These results show that components in winery wastewater do not impact the performance of PO4Sponge and that loading the wastewater from the top does not reduce performance of the adsorption media. Consistent performance, regardless of the direction of wastewater flow, allows for flexibility in the full-scale design and implementation of a GBVFCW.

CONCLUSIONS

Overall performance of the GBVFCW was satisfactory. The system continued to treat the wastewater to low effluent concentrations even when subjected to varying loading concentrations and frequencies, and at reduced temperatures. Throughout the study, all final effluent concentrations were sufficiently below EGLE groundwater discharge limits. Effluent concentrations were considerably better than the quality of septic effluent, allowing for versatility in the final discharge of the treated wastewater.

Additionally, it was found that GBVFCWs began to remove nitrogen immediately upon operation. Over 90% of COD was removed in the first column within 11 days of beginning wastewater flow through a GBVFCW that had not previously been operated. Further, the inclusion of the phosphorus adsorption media, PO4Sponge, was found to be an effective means of removing total phosphorus from winery wastewater to low effluent concentrations.

These findings indicate that a GBVFCW is a robust onsite wastewater treatment system that can treat high strength winery wastewater down to groundwater discharge limits using a small surface area. The same NRCS standard used for milking facility wastewater can be used for winery wastewater so long as the wetland is sized with the organic loading rate of 1.06E-2 lb COD/ft^2/day and does not exceed a hydraulic loading rate of 0.504 gal/ft^2/day.

Preliminary testing indicates potential for modeling the GBVFCW system using HYDRUS CW2D. HYDRUS CW2D is a finite element model to simulate two-dimensional water and solutes movement in soil. HYDRUS CW2D considers both aerobic and anoxic transformation and degradation processes for organic matter, nitrogen, and phosphorus. Utilization of the HYDRUS CW2D for a GBVFCW requires calibration and validation using intensive monitoring laboratory data. This will require specialized reactor operation and additional analytical measurements. The resulting model has potential in the development of design criteria and operational strategies to maximize treatment. This can save consider resources, when compared to experimentally testing all options.

Not all factors can be accounted for in a laboratory study and a smaller surface area may be feasible. A field demonstration is needed prior to wide-scale adoption of this technology to help determine additional design and installation considerations. A demonstration site will also allow for producers to observe system maintenance and operational procedures.

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