HIGH RATES OF AMMONIA REMOVAL IN CONSTRUCTED TREATMENT WETLAND MESOCOSMS USING OXYGENATION

By

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To the Faculty of Washington State University:

The members of the Committee appointed to examine the thesis of HUCKLEBERRY RICHARDSON PALMER find it satisfactory and recommend that it be accepted.

Chair

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Abstract

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Despite the potential for oxygenation to enhance ammonia removal in constructed wetlands, little investigation has been published on the topic. In this study, two oxygenated and two control surface flow wetland mesocosms (0.258 m^2) were constructed with *Typha* plants. All mesocosms received a synthetic wastewater with 20 mg/L COD and 10 mg/L NH_3-N, were operated at 5 and 2.5 day hydraulic retention times (HRT), and the oxygenated mesocosms were oxygenated to 14.0 mg/L DO for >6 HRT and subsequently to 5.4 mg/L DO. Percent ammonia removals and area-based removal rate constants were typical for treatment wetlands in the control mesocosms (5 day HRT, 11.6 ± 4.7% and 2.06 ± 0.84 m/year; 2.5 day HRT, -1.6 ± 10.7% and -0.32 ± 3.55 m/year), but were significantly higher in the oxygenated mesocosms (5 day HRT and 14.0 mg/L DO, 94.0 ± 2.0% and 48.87 ± 5.26 m/year; 2.5 day HRT and 14.0 mg/L DO, 83.3 ± 10.0% and 57.67 ± 19.99 m/year; 2.5 day HRT and 5.4 mg/L DO, 76.1 ± 1.9% and 45.47 ± 2.58 m/year). Generated nitrate in the oxygenated mesocosms accounted for 96.2 ± 7.3%% and 80.1 ± 16.0% of removed ammonia minus loss in total nitrogen for the 5 day HRT and 2.5 day HRT respectively, implying ammonia removal by nitrification.

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Dedication

This thesis is dedicated to my wife, Sarajoy Van Boven, for all of her support.

INTRODUCTION

Nitrogen surface water pollution from municipal WWTP effluent is a serious problem worldwide (ESA, 1998). The primary negative impacts of aquatic ammonia pollution include degradation of water bodies through eutrophication and depressed dissolved oxygen (DO) levels, particularly in nitrogen-limited coastal waters and ammonia toxicity to aquatic biota (Horne, 2001). Constructed treatment wetlands (CTWs) offer an attractive treatment method for nitrogen pollution. CTWs can bio-transform a range of contaminants, are comparatively low cost, and can provide wildlife habitat (Mitsch and Gosselink, 2000; Kadlec and Knight, 1996).

CTWs have been employed very successfully to treat nitrate pollution (Reilly et al., 2000), but CTWs have been shown to typically remove ammonia at roughly one tenth the rate at which they remove nitrate (Kadlec and Knight, 1996). This is because wetlands are predominantly reducing environments rich in organic carbon and low in dissolved oxygen (DO). As a result, anaerobic biological processes, such as denitrification of nitrate to N_2 gas, are greatly enhanced, while aerobic biological processes such as nitrification of ammonia to nitrate are inhibited (Vymazal, 2005; Edwards, 2006). This translates to expansive and sometimes prohibitive land requirements for ammonia removal by CTWs. This places a critical limitation on the use of CTWs to polish effluent from domestic wastewater treatment plants.

Oxygen limitation has been identified as the major impediment to ammonia removal in CTWs (Schlesinger, 1997; Wu et al., 2001). A number of studies have described the effectiveness of different approaches to overcome this limitation. Smith et al. (2000) found five-fold denitrification rate increases in response to increased nitrification achieved by reconfiguring an ammonia-rich treatment wetland to include more open water, higher DO (3-6 mg/L) zones. Similarly, Thullen et al. (2002) found that limiting the area in which wetland plants could grow was a key to improving ammonia removal efficiencies in a constructed wetland treating WWTP effluent. In a study of an aerated surface flow (SF) wetland mesocosm, Jamieson et al. (2003) observed a reduction in effluent ammonia levels from ~100 to < 5 mg-N/L. Lansing and Martin (2006) achieved 99% ammonia nitrogen removal with an Ecological Treatment System involving aerobic and anaerobic reactors, and multiple treatment wetlands. Cottingham et al. (1999) aerated a reed-dominated pilot-scale CTW treating primary domestic effluent and found that nitrification rates increased.

One method of enhancing oxygen availability in CTWs that has not received much examination is the use of pure oxygen gas. The addition of pure oxygen to surface waters has been shown to improve water quality in a number of aquatic systems including lakes (Moore et al., 1996), reservoirs (Speece, 1994), and rivers (Speece, 1996). Pure oxygen systems can reach much higher DO levels than the atmospheric saturation value of 9.2 mg/L. Rysgaard et al. (1994) found a linear relationship between surface water DO and oxygen penetration into aquatic sediments between 0 and 23.5 mg/L DO. With respect to profundal sediments in lakes and reservoirs, maintenance of a well-oxidized sediment-water interface has been shown to dramatically decrease release rates of ammonia (Beutel, 2006). Advantages of using oxygen gas rather than air include the relative simplicity of oxygenation systems, the high transfer efficiency and small size of oxygenation systems, the low cost of liquid oxygen, and the ease of storing enormous volumes of gaseous oxygen on site as liquid oxygen (Beutel and Horne, 1999). However, the effectiveness of using pure oxygen to enhance biological oxidation of ammonia in constructed treatment wetlands has not received scientific study.

In this study we made use of SF experimental wetland mesocosms and a synthetic wastewater of a strength similar to municipal wastewater treatment plant (WWTP) effluent, 20 mg/L COD and 10 mg/L NH_3-N, to evaluate the effectiveness of oxygenation with pure oxygen gas in enhancing the removal of ammonia in WWTP effluent polishing SF CTW. The oxygenation treatment was run in duplicate, and consisted of dissolving pure oxygen gas in a side stream at the front end of the wetland mesocosms. Duplicate control treatments without supplemental oxygen were run in parallel. Two hydraulic retention times were studied, 5 days and 10 days. Both super-saturated, 14 mg/L, and sub-saturated, 5 mg/L, DO conditions were maintained to test for enhanced ammonia removal at super-saturated DO conditions.

MATERIALS AND METHODS

Mesocosm Setup and Operation

Two experimental treatments, oxygen addition (mesocosms 1 and 2) and no oxygen addition (mesocosms 3 and 4), were carried out in duplicate, two-celled wetland mesocosms. The mesocosms were monitored for 31 days under steady-state conditions at a 10 day two-cell hydraulic retention time (HRT), and 19 days under steady-state conditions at a 5 day two-cell HRT. This was equivalent to a hydraulic loading rate (HLR) of 2.17 and 4.34 (4.34 and 8.68 per cell) cm/d, respectively. Each mesocosm consisted of two aquariums, 50.8 cm (l) by 25.4 cm (w) by 45.72 cm (h), in series (Fig. 1). Cell A preceded cell B in each mesocosm, and in mesocosms 1 and 2, cell A was oxygenated. Wetland sediment, water, and cattail (*Typha* spp.) were collected during the spring of 2007 from a mature CTW in Moscow, Idaho, used to polish secondary effluent from a domestic wastewater treatment plant. A sediment-rhizome bed 20.3 cm in depth was constructed in each aquarium. To facilitate plant establishment, water levels were maintained at 5 cm for two weeks, and then were increased by 2.5 cm every two days up to maximum levels of 23 cm. Total surface area for each

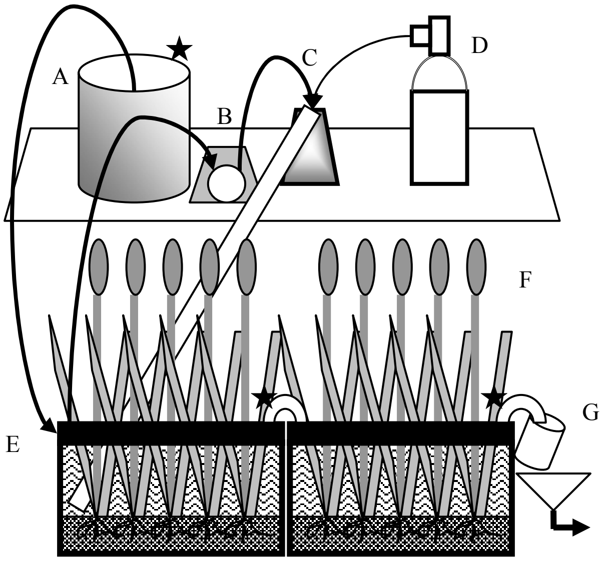

Fig. 1 - Oxygenated wetland mesocosm with (A) wastewater source, (B) peristaltic pump, circulating water from inlet area though (C) oxygenation cone, (D) oxygen tank, (E) first cell “A”, (F) second cell “B”, (G) outlet structure, and sample sites (★).

mesocosm was 0.258 m^2, water volume, assuming a porosity of 0.95 for cattail plantings, (Jamieson, 2003) was 56.0 L, and plant density was 133.7 ± 23.5 plants/m^2. Room temperature was maintained at 20.0 ± 1.0˚C and plants were exposed to 12 h/d of indoor plant lighting as well as natural light from nearby windows.

To avoid the complexity of a pumped system, mesocosms were operated under pseudo flow-through conditions. Mesocosms were each fed by gravity siphon from a reservoir filled daily with 5.6 L of synthetic wastewater during 10 d HRT phase and 11.2 L during the 5 d HRT phase. The reservoirs drained into the mesocosms over approximately 8 hours. Mesocosms were fed with synthetic wastewater simulating treated secondary effluent from a typical domestic wastewater treatment plant. Synthetic wastewater was composed of de-ionized water, dried whey, ammonium chloride, and sodium bicarbonate as an alkalinity source. Wastewater properties included a chemical oxygen demand (COD) of approximately 20 mg/L, total nitrogen of 9.6 ± 1.3 mg-N/L, and ammonia of 9.5 ± 1.3 mg-N/L.

For the oxygenated mesocosms, oxygenation was achieved by bubbling pure oxygen gas through a side stream taken from and returned to near the mesocosm inlet. The side stream was moved by a peristaltic pump, so that DO in the first cells of the oxygenated mesocosms could be controlled by the pump flowrate. Mesocosms were operated at supersaturated DO conditions, 14 ± 1.1 mg/L, for both HRTs. Sub-saturated conditions, 5.4 ± 1.0 mg/L DO, were examined at the 5 day two-cell HRT only.

Water Quality Monitoring

Water samples from influent, first-cell effluent, and second cell effluent were collected from each mesocosm, preserved, and analyzed for a range of compounds.

Dissolved inorganic nitrogen samples (ammonia and nitrate + nitrite) were filtered with pre-washed 0.45 µm Millipore filters and frozen. Total nitrogen (TN) samples were frozen unfiltered. Nitrate plus nitrite (subsequently termed nitrate) was analyzed colorimetrically on a Lachat 8500 QuickChem auto analyzer (Lachat Instruments, Milwaukee, WI). Ammonia was analyzed by the phenate method, Standard Methods 4500-NH_3 F. TN was analyzed using Hach Test'N Tube Low Range TN reagent sets measured on a Hach DR 2800 spectrophotometer. Analysis of samples was scaled-back to one oxygenated (mesocosm 2) and one control (mesocosm 4) for samples for samples collected during 5 day HRT for all analytes, except nitrate/nitrite. Temperature and DO in mesocosms were also measured at the three sampling locations (fig. 1) throughout the experiment with Hach standard luminescent DO (LDO) IntelliCAL probes attached to HQ40d digital meter/data loggers (Hach Company, Loveland, CO). Measurements of pH at sample sites were conducted with HACH PHC301-01 probes in conjunction with HQ40d meters.

Data Analysis

To facilitate comparison of results to other studies, an area-based, first order ammonia disappearance model was applied to the ammonia data. To be conservative in rate constant estimates, plug-flow conditions were assumed (Kadlec and Knight, 1996).

$$k_{AN} = -q \ln\left(\frac{C_{AN,out}}{C_{AN,in}}\right) \qquad (1)$$

where k_{AN} = first order, areal ammonia removal rate constant (m/year)

q = hydraulic loading rate (m/day)

$C_{AN,out}$ = outlet concentration of ammonia (g/m^3)

$C_{AN,in}$ = inlet concentration of ammonia (g/m^3)

Removal rates were calculated for each sampling event, and system removal rates were taken as arithmetic means of those values.

RESULTS AND DISCUSSION

Oxygen, pH and temperature

During the lower HLR, DO levels in the oxygenated cells (1A and 2A) were maintained around 14.0 ± 4.1 mg/L (Fig. 2). DO levels dropped to <1 mg/L in the controls, regardless of influent DO. During the higher HLR, DO levels in oxygenated cells were maintained around 13.8 ± 3.2 mg/L for 8 days and then reduced to 5.4 ± 2.0 mg/L for the remainder of the experiment, to provide a comparison between super- and sub-saturated conditions (Fig. 3).

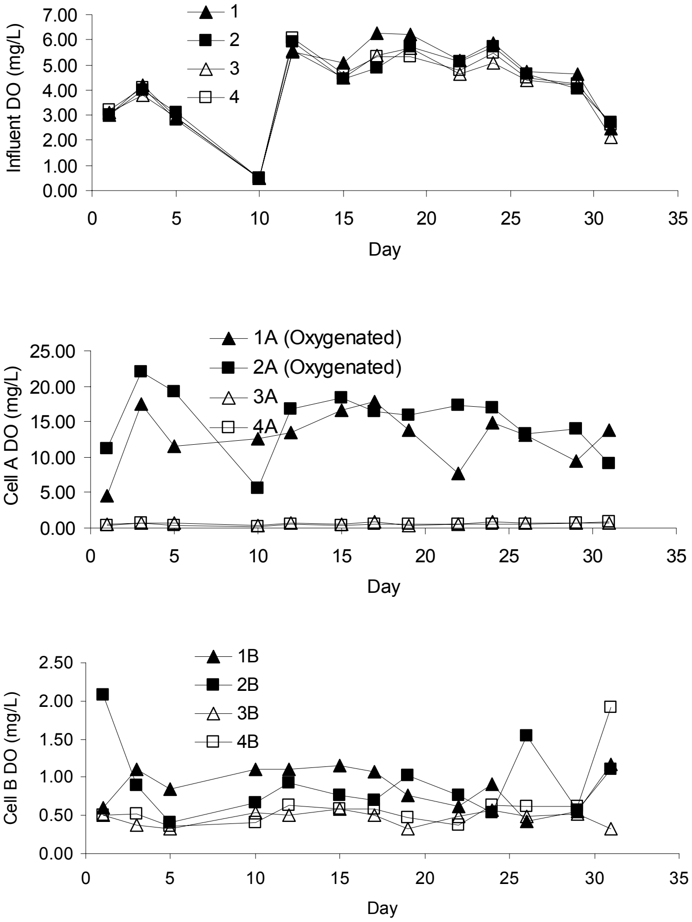

Fig. 2 – 10 day HRT DO profiles for influent, A cells (first cells), and B cells (second cells). Oxygenation occurred only in the A cells of mesocosms 1 and 2.

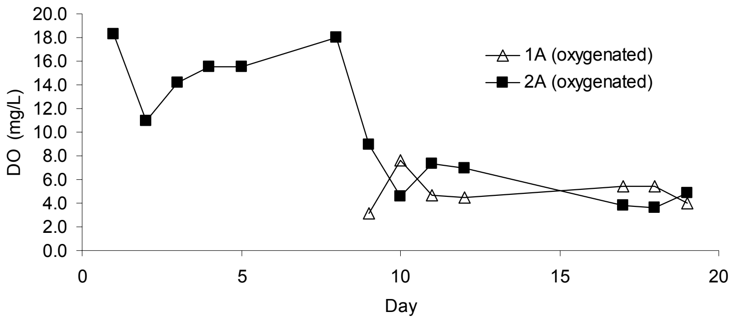

Fig. 3 – 5 day HRT first-cell oxygenated mesocosm DO.

Temperature and pH remained stable in the mesocosms throughout the experiment. Water temperatures in the influent wastewater and wetland cells were maintained at 20.0 ± 1.0 °C. Wetland pH was relatively stable, and comparable to similar full-scale treatment wetlands (Table 1).

Table 1 - Summary of pH conditions

Wetland Mesocosm	1	2	3	4
Influent	7.88 ± 1.24	7.97 ± 1.27	7.83 ± 1.30	7.86 ± 1.27
First Cells	6.70 ± 0.28	6.55 ± 0.29	6.93 ± 0.27	6.84 ± 0.25
Second Cells	6.67 ± 0.13	6.59 ± 0.13	6.91 ± 0.16	6.87 ± 0.19

Ammonia, Nitrate and Total Nitrogen

The greatest percent ammonia removal, 94.0 ± 2.0%, occurred in the first cells of the oxygenated mesocosms at the 10 day HRT. At the 5 day HRT, percent ammonia removal for supersaturated conditions, 83.3 ± 10.0%, and sub-saturated conditions, 76.1 ± 3.1%, were not statistically different, with P = 0.15 for a one-tailed t-test (Fig. 4) An increase in ammonia removal at supersaturated DO is not supported by this data. Percent ammonia removals in oxygenated mesocosms compare well to aerated wetland studies (Jamieson, 2003; Cottingham et al., 1999).

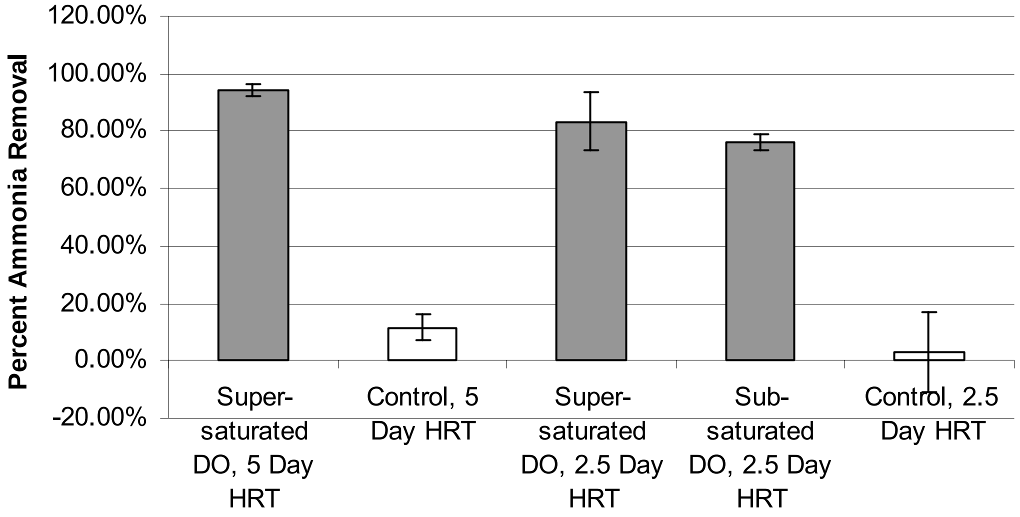

Fig. 4 – Percent ammonia removal in first cells of mesocosms. Error bars: 95% confidence interval for the mean.

Control mesocosms responded with roughly proportionally lower effluent ammonia concentrations to a decrease in ammonia loading (feed wastewater recipe error) beginning on day 18 and continuing through day 25 (Fig. 5). A corresponding decrease in effluent ammonia concentration was not observed in the oxygenated mesocosms. Second-cell effluents were higher in ammonia than first-cell effluents for the oxygenated mesocosms during 10 day HRT operation, possibly due to mineralization of ammonia from decay products or release of ammonia from sediments (Kadlec and Knight, 1996). This same phenomenon was not observed during 5 day HRT operation. (Fig. 6) Since the 5 day HRT experiment was observed 2 months after the 10 day HRT experiment, one explanation for this is that residual ammonia in the second cells could have “washed out’ in that time.

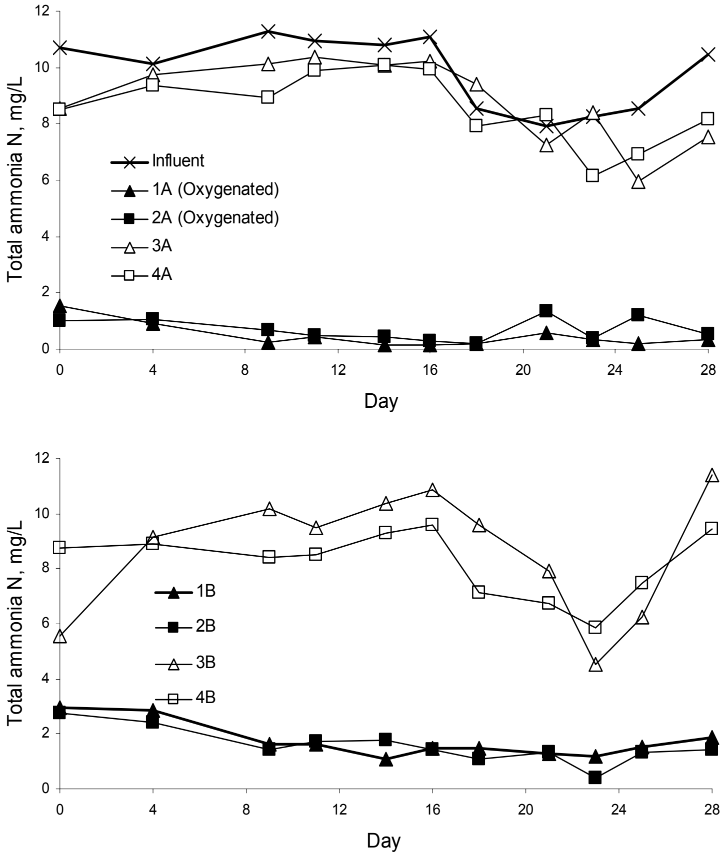

Fig. 5 - 10 day HRT ammonia concentrations for influent, first (A), and second (B) cells.

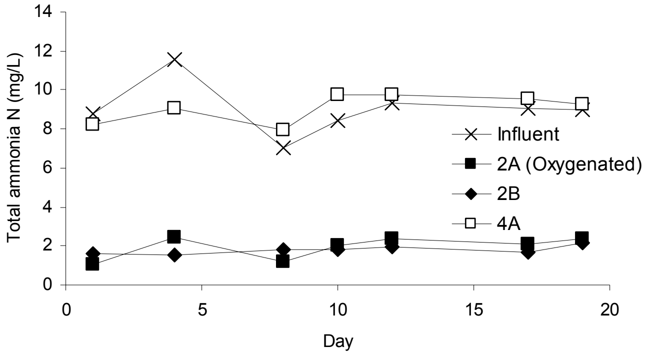

Fig. 6 – 5 day HRT ammonia concentrations for influent, oxygenated (2) and control (4) mesocosms.

In the oxygenated mesocosms, nitrate was generated in the first cells, and consumed in the second. Average wastewater influent nitrate was 0.1 mg/L. At the 10 day HRT, oxygenated mesocosm average nitrate levels were: 1A, 6.8 mg/L; 1B, 2.1 mg/L; 2A, 6.6 mg/L; 2B, 2.5 mg/L. Nitrification occurred rapidly enough to maintain similar first-cell oxygenated mesocosm average nitrate concentrations at the 5 day HRT: 1A, 7.0 mg/L; 2A, 4.0 mg/L. In the controls, nitrate was generated in much smaller amounts in the second cells, but not in the first cells (average concentrations: 3A, 0.1 mg/L; 3B, 0.4 mg/L; 4A, 0.1 mg/L; 4B, 1.0 mg/L). This is possibly due to competition from heterotrophic bacteria in the first cells, where available carbon was presumably less depleted. To confirm that ammonia removal was occurring through nitrification, nitrate generation was compared to ammonia loss, and expressed as a percentage (Fig. 7). Observed nitrate accounted for most of removed ammonia. Other possible sources of ammonia loss include migration into wetland sediments, incorporation into organisms, and nitrification followed by subsequent denitrification in deeper anoxic mesocosm sediments.

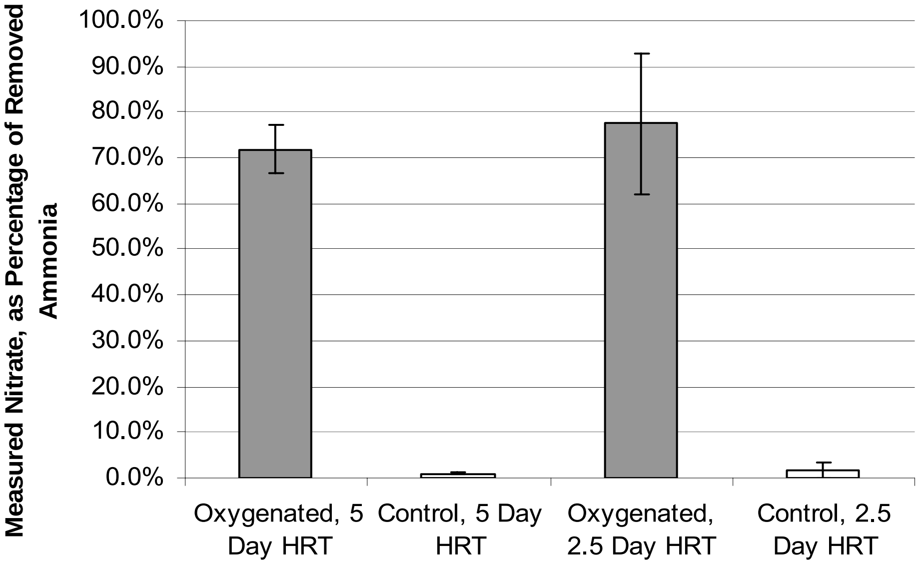

Fig. 7 – Nitrate generation demonstrates ammonia removal by nitrification--percentage of removed ammonia represented by measured nitrate generation. Error bars: 95% confidence interval for the mean.

TN concentrations are presented on Table 2. Total nitrogen removals in the two-cell oxygenated mesocosms were much lower than ammonia removals. This is because denitrification of nitrate in the second cell of the oxygenated mesocosms was incomplete (Fig. 8). These results bear upon the sizing of denitrification cells, but not upon the effectiveness of oxygen addition to CTW for ammonia oxidation.

Table 2 - Total Nitrogen (mg/L)

	10d HRT, supersaturated DO	5d HRT, supersaturated DO	5d HRT, sub-saturated DO
Influent	9.3 ± 1.9	9.2 ± 3.9	8.1 ± 2.3
Oxygenated			
First Cell	7.8 ± 0.8	7.3 ± 1.0	7.8 ± 1.2
Second Cell	4.9 ± 0.4	6.9 ± 3.0	7.4 ± 1.0
Control	9.3 ± 0.7	10.0 ± 2.9	9.8 ± 1.0

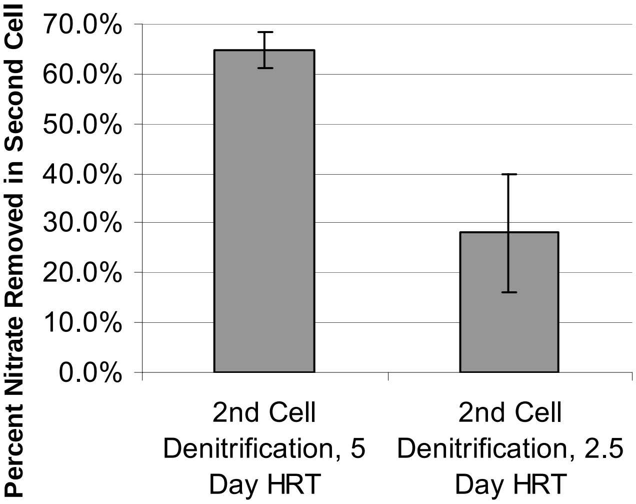

Fig. 8 – Removal of nitrate in oxygenated mesocosm second cells as a percentage of first cell nitrate effluent. Error bars: 95% confidence interval for the mean.

Nitrogen Removal Rates

For the 10 day and 5 day HRT, apparent first-order area-based ammonia removal rate constants based on the assumption of plug flow for the two oxygenated cells exceeded published rates of which the authors are aware (Fig. 9). Additionally these rate constants are much higher than typical reported values, including those for aerated SF wetlands (Table 3). It is noteworthy that removal rate constants were similar under sub-saturated DO conditions. Considering the similarity of concentrations near inlets and outlets, and the assumption of plug-flow conditions, these rates could be significantly underestimated.

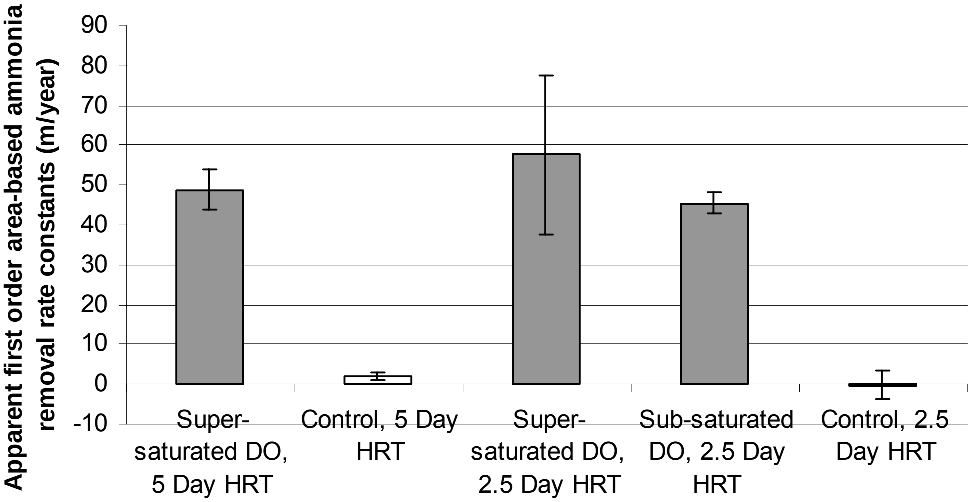

Fig. 9 – Area-based, first order apparent ammonia removal rate constants, plug-flow assumption. Error bars: 95% confidence interval for mean.

Table 3 - Comparison of first-order area-based ammonia removal constants

Wetland study	Type	Apparent k (m/yr)
Palmer and Beutel	oxygenated SF mesocosms	48.9
Palmer and Beutel	control SF mesocosms	2.1
Herskowitz	constructed marsh	2.4
Kadlec	natural marsh	22.1
Choate et al.	constructed marsh	-2.2
Walker and Walker	constructed marsh	-1.4
Bavor et al.	open water	15.4
Jamieson et al.	aerated SF mesocosm	13.1

Management Implications

Comparing an averaged k_{AN} for SF constructed wetlands (not including floating aquatic systems and adjusted to 20°C , assuming a $\theta = 1.05$) of 8 m/yr (Kadlec and Knight, 1996) to the ~50 m/yr observed in mesocosms 1 and 2, an estimate of size required for an oxygen enhanced treatment wetland relative to a standard SF treatment wetland can be made. If equal flows, equal discharge requirements, and plug-flow conditions are assumed, then:

$$\frac{A_{ox}}{A_{std}} = \frac{k_{AN}^{std}}{k_{AN}^{ox}} = \frac{8}{50} = 0.16 \cong 16\% \qquad (3)$$

where A_{ox} = surface area of oxygenated wetland

A_{std} = surface area of standard wetland

k^{std} = area-based ammonia-removal rate constant for standard wetland

k^{ox} = area-based ammonia-removal rate constant for oxygenated wetland

Reduced wetland area could result in substantially reduced capital costs, as well as open up constructed treatment wetlands as a wastewater polishing option to applications where much less land is available. Oxygenation could also allow for an upgrade of existing treatment wetlands to meet increased loading without expanding the area of the treatment wetland. Additionally, oxygenation could provide an easily controlled treatment variable in a natural treatment system to respond to changes in loading. Compressed oxygen can be purchased at low cost (~$200/1000 kg at time of writing), and oxygenation systems can be simple to install, visually unobtrusive, safe and compact, as well as require little maintenance.

Finally, it should be emphasized that ammonia-nitrogen oxidized to nitrate in an oxygenated wetland cell could be readily denitrified to nitrogen gas in a subsequent non-oxygenated wetland cell—effecting overall nitrogen removal.

CONCLUSION

The ability to remove ammonia nitrogen from municipal WWTP effluent with oxygenated CTW was investigated in a mesocosm study. Greater than 90% removal was achieved in a 5 day HRT. Apparent first order area-based ammonia removal rate constants were higher in the oxygenated mesocosm than in other ammonia removal CTW studies of which the authors are aware. Measured nitrate generation indicated that nitrification was the major route of ammonia removal in the oxygenated CTWs.

Many questions remain about the effectiveness of treating ammonia containing wastewaters in CTW with pure oxygen. Despite the expectation of increased available habitat for ammonia oxidizing bacteria due to increased penetration of DO into sediments under supersaturated conditions, no additional ammonia removal benefit was observed in this study. Understanding of ammonia removal processes can be enhanced by characterization of oxygen and ammonia penetration into CTW sediments, and assessment of ammonia oxidizing microorganism populations in the aerobic sediment surface layer. Other important unanswered questions regard the ability of oxygenated CTWs to treat higher strength wastewaters, and the effects of scale-up on system design and performance.

The observed ammonia removal efficiencies suggested that oxygenation of SF treatment wetlands can produce >90% ammonia removal in treatment wetlands of dramatically reduced size, and for high ammonia loading rates, without the potential clogging associated with sub-surface flow systems. The current state of oxygenation and oxygen storage technology allows for further investigation of oxygenated CTW at the pilot and full-scale.

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