Local-scale recovery of wastewater nitrogen for edible plant growth
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ABSTRACT
An anaerobic/ion exchange (AN-IX) system was developed for recovery and reuse of wastewater nitrogen at point-of-origin. AN-IX combines upflow solids blanket anaerobic treatment with ammonium ion adsorption onto granular natural zeolite. AN-IX operates passively and without energy input. A 57 L empty-bed prototype was operated for 355 days on wastewater primary effluent. Total nitrogen removal exceeded 95% over the first 214 days of operation and ammonia reduction exceeded 99%; accumulation of oxidized nitrogen species (NO$_3$ + NO$_2$) was not observed. The wastewater flow rate was increased during the last 35 days of operation to deliberately exhaust the ion exchange media. Spent granular media was removed from the AN-IX prototype and deployed in plant chamber experiments for cultivation of Solanum lycopersicum (cherry tomato). Wastewater nitrogen captured on zeolite was capable of supplying the total growth requirement for nitrogen. Canopy volume and plant flowering and fruiting were higher for wastewater nitrogen than for artificial fertilizer. The AN-IX process is a passive, mechanically simple and reliable system for local-scale nitrogen recovery. AN-IX is modular, scalable, adaptable and can be applied in diverse treatment contexts and recycling scenarios. AN-IX benefits include appropriate technology for local-scale nitrogen recovery, low capital and energy costs, and protection of health and the environment.

INTRODUCTION
Recovery of resources is emerging as an animating priority for wastewater management in developing countries and worldwide. The impacts of wastewater resource recovery extend through the nexus of food, water and energy and encompass public health protection and environmental quality. Recovery technologies are needed across the spectrum from large centralized infrastructure to systems that are appropriate at local-scale. Much of the world’s future population in rural, peri-urban and urban areas will rely on distributed and small-scale systems (Winblad & Simpson-Hebert 2004). One significant wastewater resource to be recovered is nitrogen, retention of which can provide nutrient for plant growth while reducing loadings to local environments and the global nitrogen cycle (Vitousek et al. 1997).

This paper presents the anaerobic/ion exchange (AN-IX) process for point-of-origin nitrogen capture from sanitation wastewater and use of captured nitrogen for propagation of a food plant. The nitrogen recovery process couples upflow solids blanket anaerobic treatment with ion exchange of ammonium using granular natural zeolite. Anaerobic treatment is recognized as a core technology for sustainable wastewater resource recovery (Zeeman et al. 2008) and enables maximum use of wastewater resources (Verstraete et al. 2009). Anaerobic treatment of wastewater with upflow solids blanket reactors is effective with low-strength household wastewater (Krishna et al. 2008; Lew et al. 2011; Chong et al. 2012) and is well suited for pre-treatment prior to ion exchange. Natural zeolites are cost-effective minerals with appreciable cation exchange properties and worldwide distribution (Mumpton 1999; Wang & Peng 2010). Zeolites can be used in an ion exchange/ammonia oxidation system to intensify ammonia removal by oxidation (Miladinovic & Weatherley 2008; Wu et al. 2008; Almutairi & Weatherley 2015). The AN-IX process contrasts with aerobic nitrogen removal systems by limiting oxygenation to preserve ammonium for sorptive recovery (Smith & Smith 2015). Anaerobic biological
treatment can also be enhanced by incorporating zeolites to sequester ammonium and reduce toxicity (Montalvo et al. 2014; Wang et al. 2015). The AN-IX process exploits the effectiveness of zeolite in retaining ammonium under anoxic conditions (Smith 2011), and might also benefit within its predominantly oxygen-free internal environments from zeolite treatment enhancements reported for other anaerobic systems.

The reuse of sanitation water nitrogen captured on zeolite has been previously reported to stimulate growth of Arthrospira platensis, a cyanobacteria (Markou et al. 2014); Ficus elastica, a landscape plant (Beler-Baykal et al. 2011), and maize (Malekian et al. 2011). The present study demonstrates growth of Solanum lycopersicum from nitrogen captured from wastewater with an AN-IX process. The AN-IX process reported in this paper provides a modular and adaptable platform for local-scale closed-loop nitrogen recycling from household wastewater.

MATERIALS AND METHODS

The AN-IX prototype had four upflow chambers each connected by a downflow channel (Figure 1). Wastewater primary effluent was pumped into the first chamber once per hour by peristaltic pump; flow through the prototype was otherwise by gravity. Wastewater first entered an anaerobic upflow solids blanket chamber and then flowed through three ion exchange chambers in series. Anaerobic treatment provides three critical functions that assist the nitrogen capture goal: (1) ammonification of organic nitrogen to NH₄⁺ ion, the nitrogen form which is amenable to cation exchange, (2) reduction in suspended and colloidal solids and organic oxygen demand, all of which decrease the propensity for clogging of the granular ion exchange media, and (3) maintenance of an anoxic headspace through production of biogas (methane/carbon dioxide). Anoxic headspace limits oxygen ingress into the ion exchange media and reduces the potential of ammonium oxidation to anionic forms (NO₃⁻, NO₂⁻), which would lower the nitrogen capture efficiency and release nitrogen into the effluent. AN-IX influent was influent to the Mayo Water Reclamation Plant, Anne Arundel County, Maryland, USA. The Mayo plant receives wastewater primary effluent that is collected from several thousand individual home systems and routed to the plant through a collection system. The AN-IX prototype received wastewater that was pumped from the wet well at the head end of the Mayo plant. Wastewater was supplied once per hour (24 doses/day). Empty bed residence times through the first 319 days of operation were 37 hours in the anaerobic chamber and 97 hours in the ion exchange chambers. To examine system resilience and accelerate exhaustion of the ion exchange media, the flow-rate was increased by factors of three and seven on Days 320 and 344, respectively. The ion exchange media in Chambers 2, 3 and 4 was Cabsorb ZS403H clinoptilolite, a natural hydrous sodium aluminosilicate zeolite (GSA Resources, Inc.). ZS403H has a manufacturer-reported clean water cation exchange capacity of 1.85 milliequivalents per gram.

A portion of AN-IX effluent was pumped to an external vertical unsaturated flow column (not shown in Figure 1) to assess post-treatment of AN-IX effluent in an unsaturated porous medium such as a receiving soil. The column had an inner diameter of 7.6 cm (3 in.) and a 61 cm (24 in.) depth of mixed media composed by volume of 70% expanded clay (2–4 mm), 25% limestone (1–2 mm), and 5% gypsum (0.5–2 mm). Effluent was dosed to the surface of the column media once per hour by peristaltic pump, at a mean daily rate of 41.5 cm/day (10.2 gal/ft²-day).
Monitoring was conducted by collecting influent samples from the Mayo wet well, AN-IX samples from the upper water column layer of each AN-IX chamber, and bottom percolate from the external column. Water quality analyses employed field probes and standard analytical methods (APHA 2002).

At the conclusion of operation, zeolite was removed from the AN-IX prototype to evaluate the ability of spent clinoptilolite to provide nitrogen for edible-plant growth. Propagation experiments were conducted in flood-and-drain, media-based hydroponic culture. In the flood-and-drain regime, reservoir water was pumped three times per day for 35 minutes to fully saturate the clinoptilolite beds, after which the water drained back to the reservoir to restore unsaturated conditions. Each media bed was connected to a dedicated liquid reservoir, which also served as the source of external growth nutrients. The three parallel treatments were (A) spent clinoptilolite with a multi-component nutrient suite that did not contain nitrogen, (B) fresh clinoptilolite with a nutrient suite that was identical to Treatment A except that it did contain nitrogen, and (C) spent clinoptilolite without external nutrients. To initiate the experiments, seeds of Solanum lycopersicum (cherry tomato) were placed approximately 1 cm below the surface of the clinoptilolite and operation was commenced. Experiments were conducted in a controlled growth chamber. Light was supplied uniformly to the growth chamber by a fluorescent 6,400 K grow light fixture (Hydrofarm T-5), with a daily cycle of 12 hours on and 12 hours off. Photosynthetic photon flux was ~250 mol/m$^2$-sec at 30.5 cm (12 in.) above the clinoptilolite surface, as measured using a quantum meter (Apogee MQ-200, Logan, Utah, USA). Cultivation temperature varied between 13.8 and 17.7 °C.

### RESULTS AND DISCUSSION

AN-IX performance on Day 56 is summarized in Table 1, which illustrates the general characteristics of AN-IX treatment. Mayo wastewater was representative of typical single residence wastewater, with low dissolved oxygen (DO) and oxidation reduction potential (ORP), and nitrogen in the reduced forms of organic nitrogen and ammonium. Total nitrogen removal by AN-IX was 95%, ammonia reduction was virtually complete, and oxidized nitrogen (NO$_3$ + NO$_2$) was not observed in AN-IX effluent. Water quality profiles through the AN-IX and external column are shown in Figures 2–4. Nitrogen speciation is shown in Figure 2. Organic and ammonium are dominant species in influent wastewater and anaerobic Chamber 1, as expected, but their higher levels in Chamber 1 effluent are attributed to typical daily variations in Mayo wastewater composition and to the 1.5 day residence time in Chamber 1. The concentration of all nitrogen species declined dramatically in porous media Chamber 2, and organic nitrogen comprised the only nitrogen form in the effluents of Chambers 3 and 4.

Profiles of ORP and DO are shown in Figure 3. ORP was negative in the AN-IX influent, which is typical of household wastewater, and increased somewhat through the chambers (Figure 3). However, a state of low oxygenation was passively sustained by AN-IX, sufficient to maintain a negative ORP through all chambers. DO was <0.1 mg/L.
in Chamber 1 and its levels increased and reached 5 mg/L in Chamber 4. The cause for the presence of DO in chamber effluents is unknown, but could be due to atmospheric gas exchange through fugitive leaks in the AN-IX cover and seals. Passage of AN-IX through the external column resulted in substantial increase in ORP and DO. AN-IX effluent was readily oxygenated by simple passage through 61 cm of unsaturated porous media, which is a typical unsaturated depth of a soil that receives wastewater effluent in onsite systems. The pH profile is shown in Figure 4. Wastewater pH remained circumneutral through AN-IX, which would limit the formation of un-ionized ammonia (NH₃) and possible volatilization losses. The increase in pH in the external column may be due release of carbon dioxide through air contact in the unsaturated column media.

The nitrogen timecourse through 355 days of operation is shown in Figure 5. Ammonia was not detected in AN-IX effluent for the first 200 days, for which total nitrogen was the predominant nitrogen form. Ammonium breakthrough was observed after 300 days of operation but was highly limited (Figure 5). To accelerate breakthrough and test the resiliency of AN-IX to higher hydraulic loadings, the flow-rate was increased on Day 320 by a factor of three and on Day 344 by a factor of seven. Ammonium breakthrough was rapid at the higher flowrates and complete breakthrough was approached at the termination of operation on Day 355 (Figure 5). The effective ammonia capacity of ZS403H for treatment of Mayo wastewater was estimated as 11.3 mg N/gram dry weight.

Solanum lycopersicum propagation was compared for different treatments based on vegetative growth, as measured through the increase in plant canopy volume. Significant differences were observed between treatments. Treatments A and B each received a suite of nutrients that was identical, with the exception that the Treatment A suite did not contain nitrogen. The greatest increase in canopy volume was observed for Treatment A, for which the only nitrogen source was the spent clinoptilolite in which it was cultivated (Figure 6). Treatment A also had
IX is a low maintenance process with small footprint and complements the advancement of AN-IX technology. AN-IX systems for plant propagation using spent clinoptilolite growth than synthetic fertilizer. Developing optimized systems for Solanum lycopersicum and provided higher vegetative wastewater nitrogen readily supported the propagation of those supplied from spent clinoptilolite. The zeolite-sorbed optimal plant propagation requires additional nutrients in growth response between Treatments A and C indicates additional added nutrients (Treatment C). The great contrast third treatment was growth on spent zeolite without synthetic fertilizer, providing higher productivity as well. A supplied wastewater nitrogen and supplanted the need for organic nitrogen fertilizer. Spent clinoptilolite readily B, which grew in a fresh clinoptilolite and received inorganic nitrogen fertilizer. Spent clinoptilolite readily supplied wastewater nitrogen and supplanted the need for synthetic fertilizer, providing higher productivity as well. A first treatment was growth on spent zeolite without additional added nutrients (Treatment C). The great contrast in growth response between Treatments A and C indicates that optimal plant propagation requires additional nutrients to those supplied from spent clinoptilolite.

CONCLUSIONS

The AN-IX process is effective for local-scale recovery of wastewater nitrogen in the form of a sorbed ion that can support propagation of edible plants. The AN-IX prototype provided resilient nitrogen capture for over 355 days with minimal operator attention. AN-IX continued to effectively convey wastewater in the last part of the study when the flow rate was increased by a factor of seven, with no observable adverse effect on physical operation. This suggests that the AN-IX system can well handle the flow variations that are typical of small wastewater systems. The zeolite-sorbed wastewater nitrogen readily supported the propagation of Solanum lycopersicum and provided higher vegetative growth than synthetic fertilizer. Developing optimized systems for plant propagation using spent clinoptilolite complements the advancement of AN-IX technology. AN-IX is a low maintenance process with small footprint and simple design, making it highly appropriate for local-scale nitrogen removal and recovery. Pilot studies can increase AN-IX process confidence by delineating uncertainties of susceptibility to clogging, low temperature operation, and the nitrogen retention capacity for a variety of wastewaters. Additional research is needed to combine extractive clinoptilolite regeneration with edible-plant propagation to foster recycling of both the sorptive media and the nitrogen resource.

REFERENCES


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