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Nitrogen management in nitrification-hydroponic systems by utilizing their pH characteristics

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ABSTRACT

In the past decade, a wave of articles was published on the liquid fertilizer by-product from the anaerobic-digestion-of-biomass process. This is not surprising given that the fertilizer (termed "digestate") is highly nutrient dense and is produced at low cost from waste biomass. Digestate has been proposed as an alternative hydroponic nutrient solution, provided that a fraction of the ammonium in the digestate be converted to nitrate. Nitrification bacteria are abundant and they readily accomplish this task upon aeration of the digestate. Although much attention has been paid to these nitrification-hydroponic systems, the field is still within its infancy and standard control methodologies have not yet been established. In traditional hydroponics, nutrient concentrations are controlled through electrical conductivity measurements, but the imbalance of nutrients and the high salt content of the digestate makes this method ill-suited for nitrification-hydroponic systems. These systems have pronounced pH responses, however, which provide information about the nutrient uptake characteristics of the plants and the nitrifying bacteria. In this study, these pH characteristics were used to control the total nitrogen concentration in solution. This was accomplished by controlling the pH via hydroxide and ammonium dosing (synthetic digestate) at a ratio of 1.5 molar. High plant growth rates were maintained at nitrogen concentrations 10 times lower than in conventional operation, thus improving the system's nitrogen use efficiency proportionally. Given the growth rate of this sector and its nutrient pollution potential, the results serve as steppingstones on the road to improved utilization of digestate as hydroponic feed solutions.

1. Introduction

Anaerobic digestion is likely to play an important role in the future of sustainable living on this planet. Biomass from sources such as food and agricultural waste (which are plentiful) still contain large amounts of nutrients and energy which go unutilized (Do et al., 2021; Gustavsson et al., 2011; Oliveira et al., 2021; Sheets et al., 2015). Anaerobic digestion is the microbial breakdown of this organic substrate into simpler, more-useable forms (Möller and Müller, 2012). Methane has been the target product from this process for some time with less attention being given to the digestate by-product. This liquid fertilizer has been applied in conventional agriculture since it is rich in all the essential nutrients required for plant growth (Koszel and Lorencowicz, 2015). Its liquid form, however, is suitable for the rapidly growing hydroponic industry (Bergstrand et al., 2020; Grand View Research, 2020; Stiles et al., 2018; Stoknes et al., 2016).

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Nomenclature

δ	Hydroxide-to-ammonium ratio in the digestate. Specifically, the mols of hydroxide required to raise the pH of the digestate (containing 1 mol of ammonium) from the operating pH of the hydroponic system to the current pH of the digestate. mol mol^{-1}
η_1	Effective proton uptake by plant the (total of all acidic and basic effects) per nitrate absorbed. mol mol^{-1}
η_2	Effective proton exudation by the plant (total of all acidic and basic effects) per ammonium absorbed. mol mol^{-1}
η_3	Effective proton exudation by the nitrifying bacteria (total of all acidic and basic effects) per ammonium converted. mol mol^{-1}

Hydroponic nutrients solutions are traditionally composed of inorganic salts because organic-based fertilizers can have phytotoxic effects when used in hydroponic systems (Mupambwa et al., 2019). The first successful use of an organic fertilizer in hydroponic systems was published by Shinohara et al. (2011) (according to Edgar et al., 2018). This was made possible by the addition of soil bacteria able to convert the organic fertilizer to a more useable form. Thereafter, interest in biogas digestate spiked and several publications followed (reviewed by Baştabak and Koçar, 2020). Several studies aimed to quantify the effects of digestate fertilizer on hydroponic performance. Some researchers reported reduced performance (Mupambwa et al., 2019; Neal and Wilkie, 2014), while others reported equal or improved performance, including benefits such as enhanced plant nutrition (Bergstrand et al., 2020; Pelayo Lind et al., 2021; Ronga et al., 2019; Stoknes et al., 2018). Pre-treatment of the digestate was carried out in most successful cases, which primarily involved the microbial conversion of ammonium to nitrate (nitrification). The nitrogen liberated from the biomass by the anaerobic bacteria is in the form of ammonium (or ammonia depending on the pH) and may reach concentrations of several grams per litre in the digestate (Svehla et al., 2017). Although plants can utilize ammonium, it can result in suboptimal plant growth and have fatal effects if supplied as the sole nitrogen source (Hachiya and Sakakibara, 2017; Neal and Wilkie, 2014; Pelayo Lind et al., 2021). Nitrification of digestate is therefore an important step in the valorization of digestate as a hydroponic fertilizer.

Hydroponic systems are known to produce large amounts of wastewater resulting in significant point-source nutrient pollution, particularly nitrogen pollution (Kanter et al., 2020; Prystay and Lo, 2001; Withers and Lord, 2002). This is due to the need to replace of the nutrient solution frequently in order to maintain high nutrient concentrations and prevent the build-up of salt and toxic species (Kumar and Cho, 2014; Silberbush and Ben-Asher, 2001). Nitrogen pollution from fertilizer spillage is known to have caused severe environmental impacts such as eutrophication, air pollution, biodiversity loss, climate change and stratospheric ozone depletion (Kanter et al., 2020). Many studies have been conducted to reduce nitrogen pollution from hydroponic systems (Castellar et al., 2019; Gagnon et al., 2010; Prystay and Lo, 2001; Rufí-Salís et al., 2020; Saxena and Bassi, 2013). Most commonly, the electrical conductivity (EC) of the nutrient solution is used as an inferential measurement of the total nutrient concentration (Christie, 2014; Domingues et al., 2012; Lenord Melvix and Sridevi, 2014). The total nutrient concentration can then be controlled by adding additional nutrients, thereby requiring less frequent replacement, and resulting in less pollution (or lighter load on wastewater treatment processes after the hydroponic unit). The EC method relies on knowledge of the nutrient uptake ratios of the plants so that nutrient can be added in the same proportions. Otherwise, some nutrients will deplete while others accumulate (Bugbee, 2004). Biogas digestate is unlikely to contain nutrients in the correct proportions. Also, the digestate contains high NaCl concentrations, especially those derived from food wastes (Bergstrand et al., 2020), which will rapidly accumulate in solution and render the EC signal less useful. Ion-selective-electrodes have also been used to control nutrient levels in hydroponic systems (Cho et al., 2018; Kim et al., 2013). These instruments, however, are expensive and exhibit practical limitations such as signal drift, reduced accuracy over time and interference from other ions in solution (Kim et al., 2013). The high NaCl concentrations, including the potentially toxic species found in the digestate will require even higher dilution rates (solution replacements) than in conventional systems. Proportionally high pollution rates will hence result, and thus alternative control strategies are required for efficient and environmentally friendly use of this fertilizer.

This study investigates the potential of online pH measurement as the sole input to control the nitrogen levels in nitrification-hydroponic systems. pH sensors and controllers are relatively cheap and routinely incorporated in hydroponic systems. A pH-based control scheme can therefore be implemented easily and will provide farmers with a convenient and affordable option to improve the nitrogen use efficiency in their systems. The development of the control scheme in this study was performed with synthetic digestate to reduce experimental noise and improve repeatability in the development process. It is therefore assumed that no organic carbon is present in the digestate that could result in pH effects (fermentation) interfering with the controller input signal.

2. Materials and methods

2.1. Equipment

Four independent, ebb-and-flow hydroponic systems, each designed to host a single plant, were used. Each system had a capacity of 1.8 L and was equipped with two peristaltic dosing pumps and a water addition pump. A photo of

Fig. 1. Annotated photo of the experimental setup showing four independent hydroponic systems. All four systems, each hosting a single Kale plant, were operated in parallel under the same conditions. Each system (or plant) is labelled "system 1" to "system 4" (from right to left) in the data reports below.

the four systems is shown in Fig. 1 (labelled "system 1" to "system 4" from right to left). For each run, all four systems were operated in parallel under the same conditions, which essentially represents a quadruplicate (four repeat runs). A total of 5 runs were conducted, hence 4 × 5 single plant runs. An Arduino Mega 2560™ was employed as the controller platform which controlled all four systems simultaneously. HAOSHI™ pH probes ("pH meter Pro") were used for online pH measurements. Kamoer © peristaltic pumps ("Precision Peristaltic Pump + Intelligent Stepper Controller") were used for dosing ammonium, acids, and bases. DFrobot™ peristaltic pumps ("digital peristaltic pump") were used for automated water addition. All chemicals/nutrients were purchased from Merck™ (BioXtra ©, ≥ 99.0%). For plant lighting, four Mars Hydro™ 400 W blue/red LED lights (Mars II 400 LED Grow Light ©) were used, producing 10 000 Lux at the canopy. Kale seeds (*Brassica oleracea* var. *Sabellica* or Vate's Blue Curled Kale) were purchased from Raw™. The main recirculation pumps (responsible for the ebb-and-flow mechanism) were purchased from Xylem™ ("Flojet Diaphragm Electric Operated Positive Displacement Pump, 3.8 L min^{-1}, 2.5 bar, 12 V DC"). For seedling propagation, aeroponic systems (Aeroponic Cloner) purchased from hydroponic.co.za™ were used. DoPhin® "14 in 1 nitrifying bacteria" was used as an inoculum. Four Gravity™ analog electrical conductivity sensor V2 were used for online EC measurements. Four Regent® 9500 air pumps were used for sparging. Evolution Aqua Kaldnes® K1 Media was used for microbial attachment media. Kale (*Brassica oleracea* var. *Sabellica*) was used as the model plant in all experiments.

2.2. Synthetic wastewater and operational conditions

Runs 1 and 2 involved plant growth and were conducted for a period of 10-days without solution replacement under 24-h light. Run 3 entailed the cultivation of nitrifying bacteria in the four systems which was conducted for 8 weeks. The established biofilter was used in all subsequent runs. Runs 4 and 5 employed larger plants to better observe the control action (explained later) and thus runtimes were shorter (6 days). This investigation concentrated on the pH dynamics involved in integrated nitrification-hydroponic systems where ammonium and nitrate are present in the nutrient solution. The effects of actual digestate on plant characteristics such as nutrition and toxicity are beyond the scope of this paper (these have been established and can be found in the literature). For this reason, inorganic (synthetic) nutrients were used in all runs with variations of Hoagland's solution (Hoagland and Arnon, 1938) representing both raw and pre-nitrified digestate. Variations between these solutions (synthetic digestate) are given in Table 1. Raw-digestate dosing was mimicked by dosing ammonium from a reservoir of 0.1 M $(NH_4)_2SO_4$ and hydroxide from a separate reservoir of 0.3 M KOH, as shown in Fig. 1.

In run 1, plants were cultivated under standard hydroponic conditions with nitrate as the sole nitrogen source. A single dosing reservoir containing 0.03 M HCl was used for pH control (at 6.1). The nutrient solution charged initially was composed of 4 mM KNO_3, 4 mM $Ca(NO_3)_2{\cdot}4H_2O$, 2 mM $MgSO_4{\cdot}7H_2O$, 1 mM KH_2PO_4, 6 mg L^{-1} NaOH, 7.5 mg L^{-1} Fe-EDTA, 0.05 mg L^{-1} Cu-EDTA, 2.9 mg L^{-1} H_3BO_3, 1.8 mg L^{-1} $MnCl_2{\cdot}4H_2O$, 0.2 mg L^{-1} $ZnSO_4{\cdot}7H_2O$ and 0.1 mg L^{-1} $Na_2MoO_4{\cdot}2H_2O$. Nutrient concentrations were kept high via relative addition (Raistrick, 1999). The nitrate concentration in solution was measured via analysis of liquid samples taken daily.

In run 2, plants were cultivated in the same fashion as in run 1 except for substituting nitrate with ammonium, and controlling the pH with 0.03 M NaOH (instead of HCl as in run 1). The nutrient solution was composed of 5 mM $(NH_4)_2SO_4$,

Table 1
Macronutrient concentrations in the solution (synthetic digestate) charged in each run initially. All values are in mM. The same micronutrient formulation was added to each run which consisted of 7.5 mg L^{-1} Fe-EDTA, 0.05 mg L^{-1} Cu-EDTA, 2.9 mg L^{-1} H_3BO_3, 1.8 mg L^{-1} $MnCl_2{\cdot}4H_2O$, 0.2 mg L^{-1} $ZnSO_4{\cdot}7H_2O$ and 0.1 mg L^{-1} $Na_2MoO_4{\cdot}2H_2O$.

	Run 1	Run 2	Run 3	Run 4	Run 5
NO_3^-	8	0	0	8	1
NH_4^+	0	10	15	0	0
$H_2PO_4^-$	1	1	1	1	1
K^+	5	5	1	1	1
Ca^{2+}	4	4	4	4	4
SO_4^{2-}	2	4	2	2	2
Mg^{2+}	2	2	2	2	2

4 mM $CaCl_2{\cdot}2H_2O$, 2 mM K_2SO_4, 2 mM $MgSO_4{\cdot}7H_2O$, 1 mM KH_2PO_4, 6 mg L^{-1} NaOH, 7.5 mg L^{-1} Fe-EDTA, 0.05 mg L^{-1} Cu-EDTA, 2.9 mg L^{-1} H_3BO_3, 1.8 mg L^{-1} $MnCl_2{\cdot}4H_2O$, 0.2 mg L^{-1} $ZnSO_4{\cdot}7H_2O$ and 0.1 mg L^{-1} $Na_2MoO_4{\cdot}2H_2O$. The experimental setup had never been charged with ammonium and no additional aeration was employed. Therefore, it is assumed that no nitrifying bacteria established during the run (given their significantly slow growth rates). This was confirmed by nitrate analysis of liquid samples in which no nitrate was detected.

Run 3 entailed the cultivation of nitrifying bacteria (without plants). No organic carbon was added, and therefore the conditions were exclusive to autotropic nitrifying bacteria. The experimental conditions were near-identical to those of run 2. The pH was controlled at 6.5 using 0.3 M KOH instead of 0.03 M NaOH, using a single dosing reservoir. The nutrient solution was composed of 7.5 mM $(NH_4)_2SO_4$, 4 mM $CaCl_2{\cdot}2H_2O$, 2 mM $MgSO_4{\cdot}7H_2O$, 1 mM KH_2PO_4, 6 mg L^{-1} NaOH, 7.5 mg L^{-1} Fe-EDTA, 0.05 mg L^{-1} Cu-EDTA, 2.9 mg L^{-1} H_3BO_3, 1.8 mg L^{-1} $MnCl_2{\cdot}4H_2O$, 0.2 mg L^{-1} $ZnSO_4{\cdot}7H_2O$ and 0.1 mg L^{-1} $Na_2MoO_4{\cdot}2H_2O$. Additional aeration was employed (sparging) and a large nitrification bacterial inoculum was supplied which had previously been activated. Daily sampling commenced 2 weeks after inoculation. The total cultivation period was 8 weeks. Subsequently, run 4 was performed which included plant cultivation. Finally, the integrated nitrification-hydroponic experiment was operated.

In run 4, a similar nutrient solution as in run 1 was charged (nitrate as the sole nitrogen source) which was composed of 4 M $Ca(NO_3)_2{\cdot}4H_2O$, 2 mM $MgSO_4{\cdot}7H_2O$, 1 mM KH_2PO_4, 6 mg L^{-1} NaOH, 7.5 mg L^{-1} Fe-EDTA, 0.05 mg L^{-1} Cu-EDTA, 2.9 mg L^{-1} H_3BO_3, 1.8 mg L^{-1} $MnCl_2{\cdot}4H_2O$, 0.2 mg L^{-1} $ZnSO_4{\cdot}7H_2O$ and 0.1 mg L^{-1} $Na_2MoO_4{\cdot}2H_2O$. The pH was controlled at 6.1 by dosing ammonium from a second dosing reservoir containing 0.1 M $(NH_4)_2SO_4$ and hydroxide from the first dosing reservoir (used in run 3) containing 0.3 M KOH, in a constant ratio of 1.5 molar OH/NH_4 (hence equal volumetric rates from each reservoir). This dosing strategy is explained in Section 3.2.

Run 5 was essentially a repeat of run 4 but at a lower initial nitrate concentration of 1 mM. The nutrient solution was composed of 0.5 mM $Ca(NO_3)_2{\cdot}4H_2O$, 4 mM $CaCl_2{\cdot}2H_2O$, 2 mM $MgSO_4{\cdot}7H_2O$, 1 mM KH_2PO_4, 6 mg L^{-1} NaOH, 7.5 mg L^{-1} Fe-EDTA, 0.05 mg L^{-1} Cu-EDTA, 2.9 mg L^{-1} H_3BO_3, 1.8 mg L^{-1} $MnCl_2{\cdot}4H_2O$, 0.2 mg L^{-1} $ZnSO_4{\cdot}7H_2O$ and 0.1 mg L^{-1} $Na_2MoO_4{\cdot}2H_2O$.

2.3. Analytical method

Liquid samples were taken daily during each run and analysed for nitrate, nitrite, and/or ammonium (depending on the run). The analysis was done using Merck™ photometric cell tests (nitrate test: DMP 0.10–25.0 mg/l NO_3-N, nitrite test: 0.002–1.00 mg/l NO_2-N, and ammonium test: 2.0–150 mg/l NH_4-N Spectroquant®). The absorbance was measured in a spectrophotometer (Agilent Technologies™, Cary 60 UV–Vis, G6860 A) at 340, 525, and 690 nm for nitrate, nitrite, and ammonium, respectively.

3. Results and discussion

3.1. Quantifying the pH dynamics in nitrification-hydroponic systems

pH changes in the liquid environment surrounding plant roots are predominantly the result of nitrogen absorption and assimilation by the plant (Lea-Cox et al., 1999). When nitrate is supplied as the sole nitrogen source, the pH rises. This is due to the release of hydroxide ions (including hydrogen carbonate ions) upon nitrate absorption (Dijkshoorn, 1962). Alternatively, when ammonium is supplied as the sole nitrogen source, the pH drops due to proton exudation from the plant roots (Hachiya and Sakakibara, 2017). Although the absorption of other nutrients also has pH effects, these effects are smaller than those induced by nitrogen absorption (Dijkshoorn, 1962). Also, they generally occur in proportion to nitrogen absorption and hence the overall pH effects are dominated by the ratio of ammonium to nitrate in the nutrient solution. This phenomenon has been studied extensively and many ammonium-to-nitrate feed ratios have been suggested to maintain pH levels (Imsande, 1986; Pitts and Stutte, 1999; Smith and Raven, 1979). These ratios

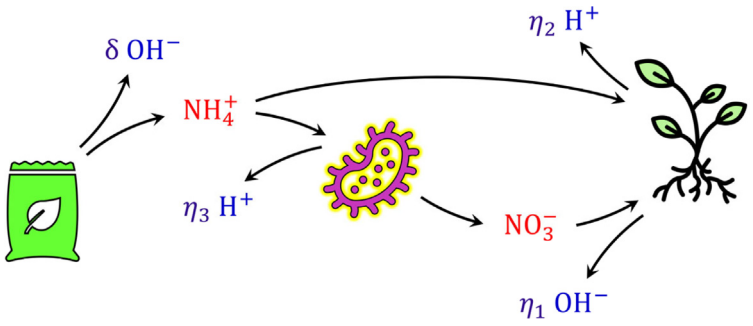

Fig. 2. Illustration of pH effects caused by ammonium oxidation (proton excretion), ammonium absorption by the plant (proton excretion) and nitrate absorption by the plant (hydroxide excretion). The digestate fertilizer is typically alkaline, hence it is depicted as releasing hydroxide ions upon addition to the solution. δ is the hydroxide-to-ammonium ratio in the digestate (mol mol^{-1}) and the η variables are the respective proton-to-nitrogen (or hydroxide-to-nitrogen) exudation characteristics of each organism in mol mol^{-1} (see Nomenclature). It is assumed that nitrite is rapidly oxidized to nitrate.

change significantly in the presence of ammonia oxidizing bacteria however, which induces a strong acidic effect upon conversion of ammonium to nitrite. These bacteria are abundant and will naturally colonize aerated environments in the presence of ammonia. Although they may take several months to become well established, these bacteria are to be expected in hydroponic systems in which ammonium is fed (Cytryn et al., 2012), yet their inevitable colonization is rarely accounted for. Once established, three effects dominate the solution's pH dynamics, namely, nitrate absorption by the plant, ammonium absorption by the plant and ammonium oxidation by the nitrifying bacteria. These effects are depicted in Fig. 2.

The theoretical amount of hydroxide released per nitrate assimilated by the plant is 2/3 mol mol^{-1}, and the number of protons released per ammonium absorbed by the plant is 4/3 mol mol^{-1} (Raven, 1985). For the nitrification bacteria, 2 mols of protons are released per mol of ammonium oxidized to nitrite, and the conversion of nitrite to nitrate is pH neutral (Madigan et al., 2003). These pH effects are defined solely per nitrogen molecule absorbed and may differ from the overall pH effects when each species is absorbed. For example, nitrogen absorption by plants is accompanied by the absorption of other nutrients which also have pH effects. The overall proton/hydroxide flux (sum of all pH effects) per unit nitrogen absorbed is a more practical variable to use because it relates directly to changes in the bulk-solution's pH. Therefore, the variables η_1, η_2 and η_3 shown in Fig. 2 are defined as the overall proton/hydroxide fluxes per nitrogen molecule converted (see Nomenclature).

These variables (η_1, η_2 and η_3) were determined in runs 1 to 3, respectively, as described in Section 2.2. Fig. 3 reports the results of runs 1 to 3, in columns 1 to 3 respectively. Subplots (a) and (d) give the hypothetical nitrate and ammonium concentrations of runs 1 and 2. The hypothetical concentrations are the measured (actual) concentrations minus the amounts of nitrate/ammonium added (manually with a pipette to prevent depletion). Subplot (g) gives the actual ammonium concentrations. No further ammonium was added and instead, the solution was replaced periodically (hence the jump in concentrations for systems 1 and 4 on day 18). The corresponding acid or base dosing rates of each run are given in subplots (b), (e) and (h). Subplots (c), (f) and (i) show the calculated η values (see Nomenclature) of runs 1 to 3. These are essentially the HCl or NaOH/KOH dosing rates divided by the nitrate or ammonium uptake rates.

From Fig. 3(c), (f) and (i), it can be seen than η remains constant with $\eta_1 \approx 0.5$, $\eta_2 \approx 1$ and $\eta_3 \approx 2$ (mol mol^{-1}). Therefore, the assumption made in Fig. 2 of constant nitrogen to proton uptake/exudation characteristics is valid and will hence form the basis of the control scheme developed in the following section. These values deviate somewhat from the theoretical values by Raven (1985). The slightly lower η_1 value of 0.5 (compared with the theoretical value of 2/3) is likely due to additional acidic effects produced by the uptake of the remaining nutrients (besides nitrogen) since the values defined by Raven (1985) considered nitrogen assimilation in isolation. The remaining nutrients (K^+, Ca^+, Mg^{2+}, SO_4^{2-}, $H_2PO_4^-$) constitute a net-positive charge when absorbed since K^+ and Ca^+ are absorbed in higher quantities than SO_4^{2-} and $H_2PO_4^-$. The absorption of a net positive-charge is typically associated with proton exudation to maintain electrical neutrality in the solution (Dijkshoorn, 1962), thus producing an additional acidic effect. However, the lower η_2 value of 1 (compared with the theoretical value of 4/3 (Raven, 1985)) indicates an additional basic effect rather than an acidic effect. This suggests that SO_4^{2-} and $H_2PO_4^-$ are absorbed in higher quantities than K^+, Ca^+ and Mg^{2+}. This is corroborated by Akl et al. (2003), Zou et al. (2005) and Na et al. (2014), who found significant decreases in cation uptake when ammonium was supplied as the sole nitrogen source.

3.2. Controlling the nitrogen concentration using pH

From Fig. 3, it can be seen that the ammonium uptake rate by the plants is similar to the ammonium oxidation rate by the bacteria. However, the ammonium oxidation rates continued to increase in the months after run 3 commenced. By

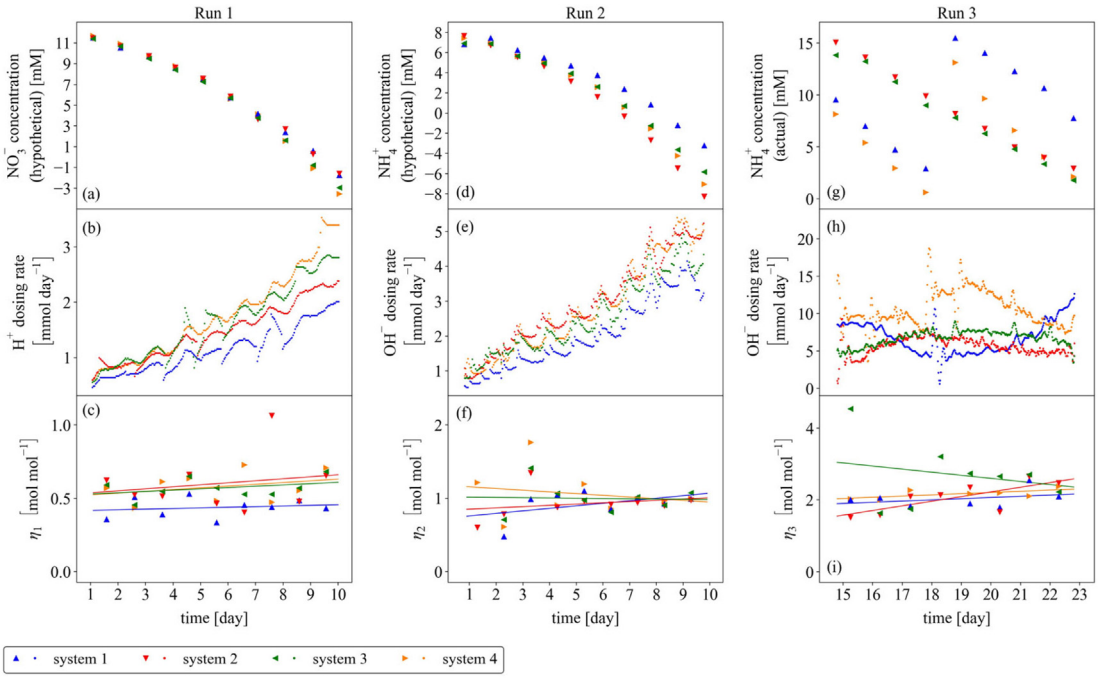

Fig. 3. Results from runs 1 to 3 reported in columns 1 to 3, respectively. For run 1, the hypothetical nitrate concentrations are given in (a). The hypothetical concentrations are the actual (measured) concentrations minus the amounts added (manually with a pipette to prevent depletion). The pH was controlled via automatic HCl dosing, the rates of which are given in (b). The ratio of the HCL dosing rates to the nitrate uptake rates (slopes between the hypothetical concentration values) are given in (c). Run 2 was near-identical to run 1 except for substituting nitrate for ammonium and using NaOH to control the pH. In run 3, nitrifying bacteria were cultivated without plants by supplying the same ammonium solution-formulation used in run 2 and controlling the pH with KOH. Negligible amounts of nitrite were measured in run 3 (around 0.001 mM) and nitrate concentrations (not shown) corresponded to the measured ammonium concentrations.

the time run 4 was performed, the ammonium oxidation rates were around ten times faster than the typical nitrogen absorption rates of the plants. Under these conditions, it was postulated that the pH could be controlled by dosing ammonium to the system containing a nitrate-based nutrient solution. For example, since ammonium oxidation occurs rapidly (relative to the plants), the effect of dosing ammonium will be acidic since most of the dosed ammonium is rapidly oxidized by the bacteria (releasing η_3 protons). Therefore, if nitrate is the sole nitrogen source in the nutrient solution (or there is a high nitrate-to-ammonium ratio), the pH rises continuously, and ammonium dosing can be used to control the pH. i.e., the same conditions as in run 1 are employed (standard hydroponic conditions) but substituting HCl for ammonium.

If the above dosing strategy is used, it can be deduced from Fig. 2 that the ammonium dosing rate will be 25% of the nitrate uptake rate by the plant (assuming $\eta_1 = 0.5$ and $\eta_3 = 2$ according to Fig. 3, and assuming that negligible amounts of ammonium are absorbed by the plant). The nitrate concentration in the solution will therefore decrease over time. Consider, however, whether instead of dosing ammonium only, a mixture of hydroxide and ammonium were to be dosed. If the ratio of hydroxide-to-ammonium (δ) does not exceed the value of η_3 (≈ 2), the effect of dosing will remain acidic but higher dosing rates will result since the effect is less acidic. Higher ammonium addition rates will be realized and thus the decrease in the nitrate concentration in solution will be slower with increasing δ values. It can hence be deduced from Fig. 2 that a δ value of 1.5 mol mol^{-1} will result in the ammonium addition rates being equal to the plant nitrate uptake rates. Since all ammonium is converted to nitrate, the nitrate concentration will remain constant, thereby achieving nitrogen concentration control.

Run 4 was performed to test this control strategy. The pH was controlled by dosing ammonium and hydroxide in a ratio of 1.5 mol mol^{-1}. Ammonium as $(NH_4)_2SO_4$ and hydroxide as KOH was dosed individually from separate dosing reservoirs (representing digestate) as shown in Fig. 1. Hoagland's solution was charged with nitrate as the sole nitrogen source (representing pre-nitrified digestate). A sequential function chart is given in Fig. 4 to convey the control algorithm. From trial runs, it was found that a constant feed of ammonium was required to maintain the vitality of the nitrifying bacteria. Therefore, the controller dosed 1 mmol day^{-1} ammonium and 1.5 mmol day^{-1} hydroxide at 30 min intervals (thus, 1/48 mmol ammonium per instance), regardless of the pH. This is shown as "Low" at the bottom of Fig. 4 in which D_{NH^+} is the ammonium dosing rate and D_{OH^-} is the hydroxide dosing rate. When the pH rose above the specified setpoint $SP = 6.1$, higher amounts of ammonium and hydroxide were dosed (still in a ratio of 1.5). These rates are abbreviated as "High", which were proportional to the setpoint error (proportional control action), specifically, $D_{NH_4^+} =$

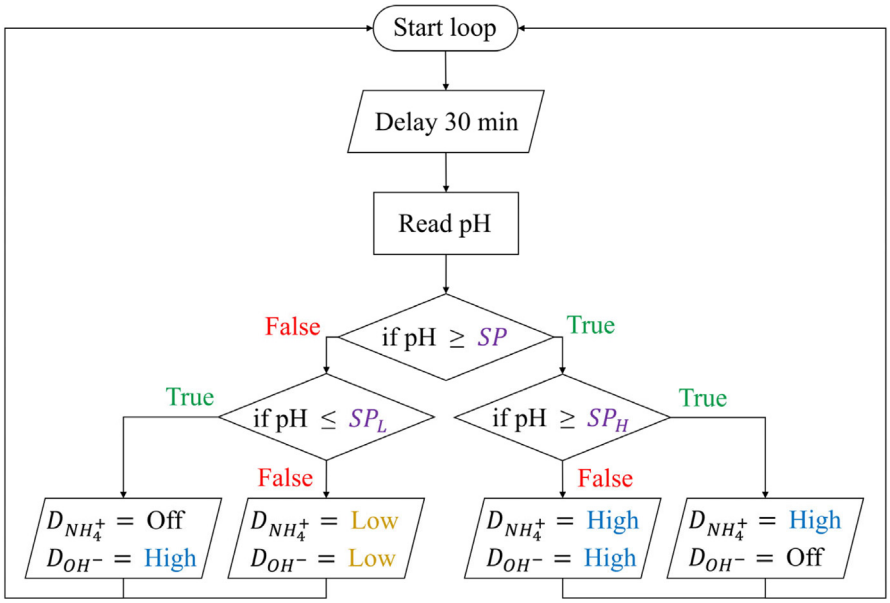

Fig. 4. Sequential function chart of the control algorithm. High dosing rates of both chemicals were actuated (in the constant ratio $\delta = 1.5$, as discussed above) when the pH was above a setpoint value $SP = 6.1$, but below a higher setpoint value $SP_H = 6.4$. If the pH rose above SP_H, hydroxide dosing was halted (safety feature). If the pH was below $SP = 6.1$ but not as low as $SP_L = 5.8$, low dosing rates were actuated (also in the constant ratio $\delta = 1.5$). This was necessary to maintain the vitality of the bacteria and prevent inactivation. If the pH was below $SP_L = 5.8$, ammonium dosing was halted, and high hydroxide rates were actuated.

"High" =30× (pH − 6.1) + 1 mmol day^{-1} and $D_{OH^-} = 1.5 \times D_{NH_4^+}$. The "Low" nitrogen dosing rate is equal to the nitrogen uptake rate of a small Kale plant of about 10 g. For this reason, larger plants (as compared with runs 1 and 2) were employed to better observe the control action. The two outermost branches at $SP_L = 5.8$ and $SP_H = 6.4$ were primarily added as safety features in case of disturbances in biofilter inactivity. These branches did not play a significant role in the subsequent runs and can thus be neglected, but they are nonetheless recommended.

The results of run 4 are reported in Fig. 5. Subplot (a) gives the nitrate concentrations of the four systems (triangle markers). No ammonium or nitrite was detected in the solution (measurements were below the calibration limits of 0.1 mM and 0.001 mM, respectively). A slow downward drift in the nitrate concentration profiles is observed. The nitrate depletion rate is around 19% (± 6%) of the rate in run 1, in which no additional nitrate was dosed (hence 81% of the plant's nitrogen was dosed as ammonium). Predictions of the nitrate concentrations, if no additional nitrogen was dosed, are also shown for comparison. These predictions are based on exponential fits of the nitrate concentrations in run 1 (standardized based on initial plant mass). A higher δ value may be employed to reduce the downward drift in the nitrate concentrations of run 4 (provided $\delta < \eta_3$). However, inevitable drift (either up or down) is expected because small errors in pump calibration and genetic variations in the plants and bacteria (regarding the proton/hydroxide to nitrogen uptake characteristics) are bound to exist (Van Rooyen and Nicol, 2021). These parameters are probably dependent on the system and plant species. Therefore, calibration would be required if the strategy is implemented. Subplot (b) shows the pH profiles for each system. Tight pH control is observed indicating the success of the control strategy. The ammonium and hydroxide dosing rates increased with plant size. The results shows that the nitrate concentration in nitrification-hydroponic systems can be maintained at relatively constant levels by controlling the pH with an alkaline digestate at a specific hydroxide-to-ammonium ratio. Similarly, for a digestate at the same pH as that of hydroponic system, 1.5 mol of hydroxide must be dosed together with 1 mol of digestate ammonium. As an added benefit, this reduces the amount of base required (hence cation build-up) compared to using pre-nitrified digestate only (nitrification occurring in an external unit, i.e., external from the plant growth unit), which requires 2 mol of hydroxide to oxidize one mol of ammonium. In addition, feeding this pre-nitrified digestate to the hydroponic system would require additional acid dosing (hence anion build-up) to control the pH.

3.3. Controlling the nitrogen concentration at lower levels

Since the nitrogen concentration can be maintained at a relatively constant value (given a slow downward drift in concentration when $\delta = 1.5$), lower operating concentrations may be employed. As the load of nitrogen spillage is proportional to the nitrogen concentration in the solution, this strategy will improve the nitrogen use efficiency of the system and reduce pollution (Van Rooyen and Nicol, 2021). Lower nitrogen concentrations can be employed without

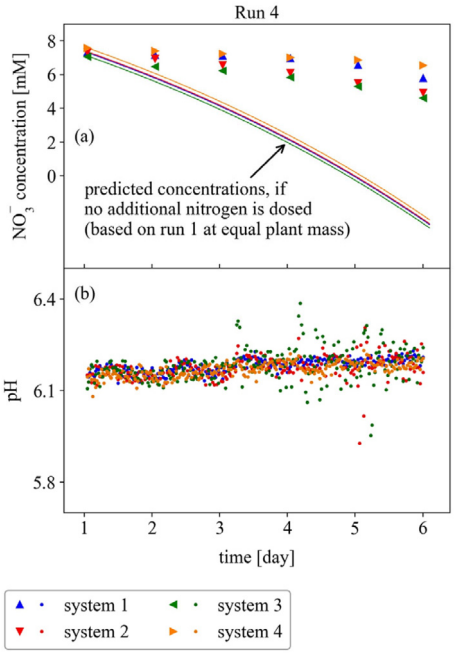

Fig. 5. Results from run 4 in which the nitrate concentration was controlled by controlling the pH with ammonium and hydroxide dosing in a ratio of 1.5 (mol hydroxide per mol ammonium). An overview of the control algorithm is given in Fig. 4. Subplot (a) shows the nitrate concentrations in solution (triangle markers) together with predicted concentrations (based on run 1, Fig. 3(a)) if no additional nitrogen was dosed. Subplot (b) shows the online pH measurements (raw) for each of the four systems. All ammonium and nitrite measurements were at the calibration limits of the analytical tests (0.1 mM and 0.001 mM, respectively); hence they are assumed to be negligible.

affecting plant growth or nutrition (Kuzyakov and Xu, 2013; Le Deunff et al., 2019), but lower nitrogen levels mean a higher risk of nitrogen extinction in solution. Therefore, good controller performance and robustness are required. Run 5 was performed to determine the feasibility of operating at lower nitrogen concentrations using the proposed control strategy. Run 5 was conducted under the same conditions as run 4 (also employing a δ value of 1.5), except for charging a lower initial nitrate concentration of 1 mM. The results are reported in Fig. 6 in the same format as in Fig. 5. Like run 4, no ammonium or nitrite was detected (measurements were below the calibration limits of 0.1 mM and 0.001 mM, respectively).

Similar to run 4, a slow depletion rate in the nitrate concentrations is observed (with a small initial increase in system 2). However, the depletion rates are 3% ($\pm$3%) of those in run 1 which are significantly slower than those of run 4 (which were 19% ($\pm$6%) of run 1). These rates are reported in Fig. 7 which is intended to compare the growth characteristics between the runs. The relative growth rates (*RGR*) and nitrogen content of the plants are also compared in Fig. 7. As can be seen from Fig. 7, all the *RGRs* are around 0.2 day^{-1}. A significant decrease in the plant nitrogen content is observed for run 5 (ANOVA, $p = 0.06$), which is believed to be due to the low nitrogen concentrations employed. When comparing the nitrate depletion rates of runs 4 and 5, the rates appear to decrease with the nitrate concentration in solution (ANOVA, $p = 0.006$). To explain this phenomenon, mathematical modelling of the flux model depicted in Fig. 2 was undertaken. The model and simulation results are given in the Supplementary Material, which agree with the observed phenomenon in which the rate of nitrate depletion decreases with the nitrate concentration. The simulation predicts a steady-state nitrate concentration which demonstrates controller robustness. This steady-state concentration was not achieved in the current system, however, which is believed to be due to imperfect mixing and system instabilities as observed in the pH and dosing profiles of the previous runs. However, it was shown that the control strategy effectively safeguards against nitrate extinction in solution.

From Fig. 7, it can be seen that plants in runs 1 and 2 had similar nitrogen contents. The only difference between the runs was the nitrogen source, with only nitrate being supplied in run 1 and only ammonium in run 2. This shows that the influx of total nitrogen is regulated by the plant, rather than strictly-separate uptake mechanism for nitrate and ammonium (Boudsocq et al., 2012). If the influx of total nitrogen was not regulated by the plant, double the amount of plant nitrogen content can be expected if both nitrate and ammonium is supplied in sufficiently high concentrations. Therefore, efforts to model plant nitrogen uptake should consider the total nitrogen concentration in solution rather than model nitrate and ammonium uptake separately. A suggested correlation is given in the Supplementary Material (Equation 5).

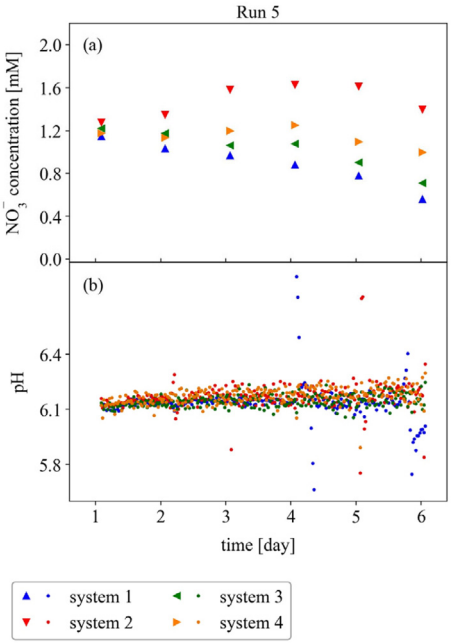

Fig. 6. Results from run 5 in which the nitrate concentration was controlled by controlling the pH with ammonium and hydroxide dosing in a ratio of 1.5 (mol hydroxide per mol ammonium). An overview of the control algorithm is given in Fig. 4. Subplot (a) shows the nitrate concentrations in solution (triangle markers). Subplot (b) shows the online pH measurements (raw). All ammonium and nitrite measurements were at the calibration limits of the analytical tests (0.1 mM and 0.001 mM, respectively); hence they are assumed to be negligible.

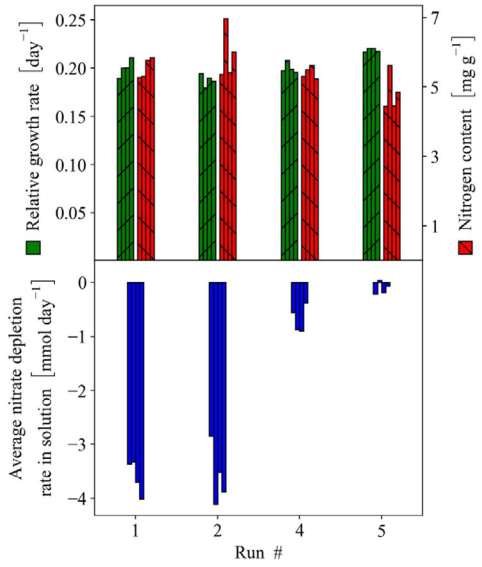

Fig. 7. Comparison of the relative growth rates of the plants, the nitrogen content of the plants, and the average nitrate depletion rates in solution between runs 1, 2, 4 and 5. The average nitrate depletion rates are standardized based on initial plant mass. The relative growth rates were calculated from the initial and final plant fresh masses. The nitrogen content was calculated from a nitrogen mass-balance over the solution and the change in plant fresh mass (thus units of mg elemental nitrogen per change in plant fresh mass).

4. Conclusions

It was shown that the nitrogen concentration can be controlled at various levels in nitrification-hydroponic systems using online pH measurement only. This was accomplished by controlling the pH of a nitrate rich medium by dosing hydroxide and ammonium in a constant ratio of 1.5 mol mol^{-1}. This demonstrates that when digestate is used to control

the pH of the nutrient solution, the nitrogen concentration can be controlled simultaneously if the pH of the digestate is adjusted such that the same ratio of hydroxide-to-ammonium (1.5 mol mol^{-1}) exists in the digestate. It was further shown that the control strategy inherently inhibits nitrogen extinction in the medium, allowing for low nitrogen concentrations to be employed at low risk of nitrogen extinction. The results demonstrate that good nitrogen use efficiency can be achieved in these systems and that nitrogen pollution can be reduced proportionally. Given the growth rate of this sector and the lack of readily available nutrient management strategies for these systems, the control methodology presented here may prove to be a much-needed alternative.

CRediT authorship contribution statement

Ignatius Leopoldus van Rooyen: Experimentation, Construction, Installation, Design, Conceptualization, Implementation, Technique creation, Methodology, Data analysis, Writing, Figure generation, Literature review. **Willie Nicol:** Supervision, Hypothesis, Data analysis, Validation, Reviewing and Editing, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary material related to this article can be found online at https://doi.org/10.1016/j.eti.2022.102360.

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