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Effect of pH on Cucumber Growth and Nutrient Availability in a Decoupled Aquaponic System with Minimal Solids Removal

Caroline Blanchard [1,*], Daniel E. Wells [1], Jeremy M. Pickens [1] and David M. Blersch [2]

[1] Department of Horticulture, Auburn University, 101 Funchess Hall, Auburn, AL 36849, USA; wellsda@auburn.edu (D.E.W.); jeremy.pickens@auburn.edu (J.M.P.)

[2] Biosystems Engineering Department, Auburn University, 214 Colley Building, Auburn, AL 36849, USA; dmb0040@auburn.edu

* Correspondence: caroline.e.blanchard@gmail.com

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Abstract: Decoupled aquaponic systems are gaining popularity as a way to manage water quality in aquaponic systems to suit plant and fish growth independently. Aquaponic systems are known to be deficient in several plant-essential elements, which can be affected by solution pH to either increase or decrease available nutrients. To determine the effect of pH in a decoupled aquaponic system, a study was conducted using aquaculture effluent from tilapia culture tanks at four pH treatments: 5.0, 5.8, 6.5, and 7.0, used to irrigate a cucumber crop. Growth and yield parameters, nutrient content of the irrigation water, and nutrients incorporated into the plant tissue were collected over two growing seasons. pH did not have a practical effect on growth rate, internode length or yield over the two growing seasons. Availability and uptake of several nutrients were affected by pH, but there was no overarching effect that would necessitate its use in commercial systems. Nutrient concentrations in the aquaculture effluent would be considered low compared to hydroponic solutions; however, elemental analysis of leaf tissues was within the recommended ranges. Research into other nutrient sources provided by the system (i.e., solid particles carried with the irrigation water) would provide further information into the nutrient dynamics of this system.

Keywords: pH; sustainability; tilapia

1. Introduction

Most soilless crop production systems allow the grower to provide plant-essential elements at predetermined levels and even be used to supplement certain nutrients beyond recommended levels, all to produce a higher quality crop [1]. Molybdenum fertilization in floating raft systems has been shown to increase the nutritional quality of leafy greens [2] and even the addition of nonessential, beneficial nutrients, such as selenium, has been shown produce a crop that has a higher nutritional value than when not supplied [3]. Aquaponics combines the nutrient delivery method of hydroponics with aquaculture by taking nutrient-rich wastewater from aquaculture production and using it as a nutrient solution for horticultural crops. Because nutrients are supplied by what is contained in fish waste, there are typically lower levels of certain nutrients in aquaponic systems, namely potassium, calcium, magnesium, and iron [4]. Aquaculture production maintains pH levels between 7.0 to 8.5 to favor nitrification in the biofilters [5], which allows more of the toxic NH_3 to be converted to NO_3. In regard to plant production, pH plays a vital role in the nutrition of soil-based systems [6] as well as hydroponic systems [7,8] with a recommended pH range for plant growth at 5.5–6.5. Different from conventional soil production, aquaponic plant production takes advantage of nutrients dissolved in solution rather than adsorbed to the surface of soil particles. Because aquaponics is linked to aquaculture production in coupled systems, the pH of the irrigation water is typically maintained at a level that favors fish production and nitrification rather than plant production, reconciling both systems at a pH of 7.0 [9]. Newer aquaponic system designs decouple the aquaculture and horticulture units. These decoupled systems allow each unit to be optimized separately.

Cucumbers are a major greenhouse crop primarily produced in hydroponic systems [10]. Cucumber production pH is managed at 5.5–6.5, levels commonly used for plant production. Several studies address the differences in pH between hydroponic and aquaponic production by using hydroponic solution at various pH levels to simulate the effects of nitrification in aquaponic solution [11,12]. Tyson [11] concluded that a pH of 7.0 would be a suitable compromising pH for aquaponic cucumber production even though early marketable fruit yields were limited. Tyson's and others' results may be best applied to clearwater systems in which organic solids are removed. Solids from aquaculture effluent contain mostly calcium (0.5%) as well as phosphorus (0.3%), a nutrient commonly limited in aquaponics, along with other plant-essential nutrients [13]. Monsees [13] reported aerobic treatment of sludge increased phosphorus (330%) and potassium (31%). Aerobic conditions, such as those available in the perlite media could provide opportunities for nutrients to be made available to plants. Therefore, a study was designed to determine the effects of pH on cucumber growth and yield in a decoupled, media-based biofloc-type aquaponic system with minimal solids removal.

2. Materials and Methods

2.1. Plant Culture

Cucumber (*Cucumis sativus* L. 'Delta Star') seeds were sown in 72-count round-cell (58 mL) seeding trays (Hydrofarm, Pentaluma, CA, USA). Seedlings were transplanted upon emergence of true leaves into 11 L rectangular Dutch buckets (Crop King Inc., Lodi, OH, USA) containing 100% perlite media.

2.2. Fish Culture and Irrigation Source

Fish production was conducted in a 9.1 × 29.3 m double polyethelene-covered greenhouse which contained two 102,000 L rectangular tanks, each holding an average of 6000 tilapia (*Oreochromis niloticus*). The aquaculture system was operated as an autotrophic, biofloc system in which an external nutrient source was not applied to promote biofloc production. Approximately 5% of the tank volume (5100 L) was used for irrigation of plants and replaced with fresh water daily, which was sourced from a series of ponds and fed via gravity to the aquaculture system. Tilapia were fed twice daily until satiation with a commercial aquaculture feed containing 36% crude protein, 6% crude fat, 3.5% crude fiber, and 0.9% phosphorus (Cargill, Franklinton, LA, USA). Water quality in the tanks was monitored daily for pH, ammonia, and dissolved oxygen levels. Water pH was maintained at 7.0 for fish production by adding a hydrated lime slurry several times a week as needed to raise pH to the appropriate level. Ammonia and dissolved oxygen levels remained within acceptable levels for fish production for the duration of the experiment. Suspended solids, including uneaten feed, feces, and microbial flocs, were settled and removed from aquaculture effluent (AE) using two passive clarifiers connected in a series. The clarifiers were 1500 L cone-bottom tanks located adjacent to the aquaculture system outside the greenhouse. Aquaculture effluent was continuously pumped into the first clarifier using an air lift and was forced to pass under a solid baffle separating the tank into two halves before being moved to the second clarifier which was used as the irrigation reservoir for the plant greenhouse. The clarifiers removed an average of 50% of suspended solids from AE before it was used to irrigate plants.

2.3. Experimental Design

Four pH treatments with target levels at 7.0, 6.5, 5.8, and 5.0 were randomly assigned to 16 plots in a randomized complete block experimental design to account for a temperature gradient in the greenhouse. The four blocks were arranged in the greenhouse with each block containing one experimental unit per treatment. Each experimental unit consisted of four Dutch buckets containing one cucumber plant each. Two additional Dutch buckets, each containing one cucumber plant, were placed on each end of the experimental unit to account for shading effects on the edge plants of the plot and to serve as plants for destructive tissue analysis throughout the season, for a total of 96 cucumber plants.

2.4. Treatment Application

Three Chemilizer chemical injectors (Hydro Systems Company, Cincinnati, OH, USA) were installed in the research greenhouse to inject acid (1M citric acid in spring 2019 and 33% sulfuric acid in summer 2019) to lower the irrigation water to the target pH. Citric acid was used to adjust pH in the spring trial; however, target levels were not reached. Sulfuric acid was used in the summer trial, and while treatment pH was lower compared to the spring trial, target pH levels were still not reached. pH is managed in the fish tank by using hydrated lime to increase pH, which creates a highly buffered system. Trials were analyzed separately due to acid type and seasonal changes. Acid was mixed into irrigation water post injection using an inline static mixer (Johnson Screens®, St. Paul, MN, USA) to ensure proper mixing of the acid with irrigation water. Acid was not added to one treatment level, target pH of 7.0, to observe the effects of unadjusted aquaculture effluent on plant growth. While pH was not adjusted in the horticulture unit for the 7.0 treatment, pH of the fish tank was monitored and adjusted daily by the addition of hydrated lime to the tank.

2.5. Cultural Practices

Plants received between 6 and 8 L of AE each day during daylight hours using a Sterling 30 irrigation controller (Superior Controls, Torrance, CA, USA) set to water at specific time intervals set by the grower. Irrigation was set higher in the summer to prevent water stress due to increased light intensity and temperatures. Water quality was monitored throughout the duration of the experiment for pH, electrical conductance (EC), and NO_3–N using a HI9813-6 Portable pH/EC/TDS/Temperature Meter (Hanna Instruments, Smithfield, RI, USA) and L-AQUA twin handheld meters (Horiba, Kyoto, Japan).

Vines were trained up Bato bobbins strung with twine (Crop King, Lodi, OH, USA) 2.1 m to the main trellis cable. Once vines reached the main trellis cable, they were leaned and lowered to continue growing, similar to tomato production practices. Lateral stems were removed as they appeared along the main stem according to Hochmuth [7].

2.6. Data Collection

Initial height, final height, and number of nodes were collected on each plant and averaged across the four plants in each experimental unit to obtain information on growth rate throughout production. Yield was calculated as a sum of marketable fruit at the end of a 60-day cycle length. Marketable fruit were defined as fruit that measured between 15 and 25 cm and were free of mechanical or insect damage. Tissue samples were collected as a composite sample of the two end plants on each plot for each experimental unit (n = 16) from the most recently matured leaves on 30 days after transplant (DAT) and 60 DAT and subjected to elemental analysis by Inductively Coupled Plasma Emission Spectroscopy (ICPES) using the Association of Official Agricultural Chemists (AOAC) official method 985.01 [14] at Waters Agricultural Laboratory in Camilla, GA, USA. Water samples from the emitter of each plot (n = 16) were taken at 30 DAT and 60 DAT and analyzed for plant-essential-nutrient levels at Auburn University's Soil Lab in Auburn, AL, USA, using Inductively Coupled Atomic Plasma (ICAP) Analysis. AE for solids collection was captured from emitters for each treatment in one block. AE was centrifuged at 4000 rpm for 15 min until 2 g of wet solids were collected. Solids were dried at 105 °C for 24 h and analyzed using ICPES using AOAC official method 985.01 [14] for total nutrient content (Waters Agricultural Laboratory, Camilla, GA, USA).

2.7. Data Analysis

Data were analyzed using SAS software (SAS Institute, Cary, NC, USA) as an analysis of variance (ANOVA) via PROC GLIMMIX and LSMEANS (SAS 9.4, Cary, NC, USA). Block was treated as a random variable. Nutrient concentrations in the water samples were compared to standard hydroponic solution standards [8] for cucumber production for each treatment using Dunnett's Test via PROC TTEST in SAS. Element concentrations in plant tissue were compared against standard plant-tissue levels [15] for cucumbers using PROC TTEST in SAS.

3. Results

A target pH of 7.0 resulted in a 9.5% increase in growth rate compared to the 5.0 treatment in spring 2019 when citric acid was used as the acidifying agent (Table 1), but a similar trend was not observed in summer 2019 when sulfuric acid was used as the acidifying agent. No other measured growth and yield factors, including internode length and total yield, were influenced by target pH treatment in either season (Table 1).

Table 1. Growth and yield of aquaponic cucumber at four pH treatments.

Target pH [z]	Actual pH (±S.E.)	Growth Rate (cm•day^{-1})	Internode Length (cm)	Yield (kg•plant^{-1})
		Spring		
7.0	6.9 ± 0.16	4.95 a [y]	8.66 ns	6.59 ns
6.5	6.7 ± 0.22	4.70 ab	8.63	6.01
5.8	6.4 ± 0.30	4.67 ab	8.58	6.63
5.0	6.3 ± 0.46	4.75 b	8.43	7.01
		Summer		
7.0	6.7 ± 0.22	9.80 ns	9.50 ns	8.39 ns
6.5	6.6 ± 0.22	9.73	9.70	8.24
5.8	6.3 ± 0.45	9.65	9.65	8.84
5.0	6.1 ± 0.70	9.98	9.70	8.99

[z] Target pH based on conventional recirculating aquaculture systems (RAS) (7.0), conventional agriculture (6.5), conventional hydroponics (5.8) and below plant- and fish recommendation (5.0). [y] Means with the same letters in a column and season are not significantly different at $p < 0.05$ as determined by analysis of variance and LSMEANS using the GLIMMIX procedure and type III sum of squares in SAS software. ns = not significant.

Midseason soluble macronutrient ion concentrations in aquaculture effluent (AE) were generally not affected by pH treatment, with the exception of potassium (K) in summer 2019, which decreased only 1.68%, from 175 to 172 mg·L^{-1} K, as target pH decreased from 7.0 to 5.0 (Table 2). When compared against standard hydroponic solution for cucumber production [8], all measured levels of plant-essential elements were lower than the recommended amount for hydroponic production according to *t*-test results using the TTEST procedure in SAS. Macronutrient uptake was not generally influenced by target pH treatment, with the exception of foliar P, which decreased quadratically by 16% in spring 2019 and linearly by almost 18% in summer 2019 (Table 3). When compared against standard percentages in cucumber dry matter [15], all nutrients were above the lower range of recommended percentage of dry matter for cucumber growth according to *t*-test results using the TTEST procedure in SAS.

By the end of each trial, soluble NO_3–N concentrations decreased as target pH decreased by 19% and 6% in spring and summer 2019, respectively (Table 4). Soluble calcium (Ca) and magnesium (Mg) in AE increased by 7% as target pH decreased from 7.0 to 5.0 each by the end of summer 2019 (Table 4).

When compared against standard hydroponic solution for cucumber production [8], all measured levels of plant-essential elements were lower than the recommended amount for hydroponic production according to *t*-test results using the TTEST procedure in SAS. Similar to midseason results, macronutrient uptake was generally not affected at the end-of-season by target pH treatments, with the exception of Ca, which decreased linearly by nearly 7% as target pH decreased from 7.0 to 5.0 (Table 5). This decrease in Ca uptake correlated seemingly well to the observed decrease in soluble Ca in AE at the same timeframe. However, a similar trend for Mg uptake was not observed. When compared against standard percentages in cucumber dry matter [15], all nutrients were above the lower range of recommended percentage of dry matter for cucumber growth according to *t*-test results using the TTEST procedure in SAS.

Table 2. Midseason macronutrient analysis ($mg{\cdot}L^{-1}$) of aquaponic irrigation water at four pH treatments.

Target pH	Actual pH (±S.E.)	NO_3–N	P	K	Ca	Mg
		Spring				
7.0	6.9 ± 0.16	187 ns ^{z}	8 ns	104 ns	187 ns	20 ns
6.5	6.7 ± 0.22	183	8	104	186	20
5.8	6.4 ± 0.30	184	8	104	188	20
5.0	6.3 ± 0.46	171	8	104	187	20
Significance	N/A	N/A	N/A	N/A	N/A	N/A
		Summer				
7.0	6.7 ± 0.22	93 a	11 ns	175 a	108 ns	20 ns
6.5	6.6 ± 0.22	86 bc	11	173 b	106	19
5.8	6.3 ± 0.45	88 b	12	173 b	106	19
5.0	6.1 ± 0.70	85 c	12	172 b	105	19
Significance	N/A	N/A	N/A	L ^{*y}	N/A	N/A
Recommended level ^{x}	N/A	216 ^{w}	58	286	185	185

^{z} Means with the same letters in a column and season are not significantly different at $p < 0.05$ as determined by analysis of variance and LSMEANS using the GLIMMIX procedure and type III sum of squares in SAS. ns = not significant. ^{y} Significance established using trend analyses in PROC GLIMMIX. L = linear trend. * = significance at $\alpha = 0.05$. N/A = no trend test conducted because of nonsignificant ANOVA. ^{x} Recommended levels obtained from Jones [16]. ^{w} ppm total nitrogen.

Table 3. Midseason foliar analysis of macronutrient content as percentage of dry matter for aquaponic cucumbers at four pH treatments.

Target pH	Actual pH (±S.E.)	N	P	K	Ca	Mg	S
7.0	6.9 ± 0.16	4.98 ns ^{z}	0.68 a	3.95 ns	8.36 ns	0.58 ns	0.76 ns
6.5	6.7 ± 0.22	4.96	0.52 b	3.65	8.16	0.56	0.74
5.8	6.4 ± 0.30	4.88	0.54 b	3.59	7.65	0.54	0.74
5.0	6.3 ± 0.46	4.90	0.57 b	3.31	8.28	0.58	0.74
Significance ^{y}	N/A	N/A	Q*	N/A	N/A	N/A	N/A
			Summer				
7.0	6.7 ± 0.22	5.72 ab	0.62 a	3.19 ns	4.56 ns	0.46 ns	0.98 ns
6.5	6.6 ± 0.22	5.57 c	0.51 b	3.38	4.71	0.51	0.90
5.8	6.3± 0.45	5.85 a	0.54 b	3.18	4.10	0.51	1.09
5.0	6.1 ± 0.70	5.68 bc	0.51 b	3.31	4.47	0.46	1.03
Significance	N/A	N/A	L *	N/A	N/A	N/A	N/A
Sufficiency levels ^{x}	N/A	4.30	0.30	3.10	2.40	0.35	0.32

^{z} Means with the same letters in a column and season are not significantly different at $p < 0.05$ as determined by analysis of variance and LSMEANS using the GLIMMIX procedure and type III sum of squares in SAS. ns = not significant. ^{y} Significance established using trend analyses in PROC GLIMMIX. L = linear trend, Q = quadratic trend. * = significance at $\alpha = 0.05$. N/A = no trend test conducted because of nonsignificant ANOVA. ^{x} Sufficiency levels obtained from Mills [11].

Table 4. End-of-season macronutrient analysis (mg·L^{-1}) of aquaponic irrigation water at four target pH treatments.

Target pH	Actual pH (±S.E.)	NO_3–N	P	K	Ca	Mg
			Spring			
7.0	6.9 ± 0.16	110 a ^{z}	20 ns	171 ns	139 ns	23 ns
6.5	6.7 ± 0.22	103 ab	15	167	137	23
5.8	6.4 ± 0.30	98 bc	19	154	126	22
5.0	6.3 ± 0.46	89 c	13	168	131	23
Significance ^{y}	N/A	L **	N/A	N/A	N/A	N/A
			Summer			
7.0	6.7 ± 0.22	96 a	12 ns	98 ns	110 b	28 c
6.5	6.6 ± 0.22	93 ab	13	98	111 b	29 bc
5.8	6.3 ± 0.45	93 ab	14	98	115 a	29 b
5.0	6.1 ± 0.70	90 b	13	98	118 a	30 a
Significance	N/A	N/A	N/A	N/A	L ***	L ***
Recommended levels ^{x}	N/A	216 ^{w}	58	286	185	185

^{z} Means with the same letters in a column and season are not significantly different at $p < 0.05$ as determined by analysis of variance and LSMEANS using the GLIMMIX procedure and type III sum of squares in SAS. ns = not significant. ^{y} Significance established using trend analyses in PROC GLIMMIX. L = linear trend. **, *** = significance at $\alpha = 0.01$ and 0.0001 respectively. N/A = no trend test conducted because of nonsignificant ANOVA. ^{x} Recommended levels obtained from Jones [16]. ^{w} ppm total nitrogen.

Table 5. End-of-season foliar analysis of macronutrients as percentage of dry matter for aquaponic cucumbers at four target pH treatments.

Target pH	Actual pH (±S.E.)	N	P	K	Ca	Mg	S
			Spring				
7.0	6.9 ± 0.16	5.26 ns^z	0.54 ns	4.11 ns	5.46 ns	0.41 ns	1.23 ns
6.5	6.7 ± 0.22	5.16	0.44	4.06	5.61	0.42	1.19
5.8	6.4 ± 0.30	4.84	0.44	3.82	4.92	0.40	1.07
5.0	6.3 ± 0.46	5.23	0.55	4.08	5.52	0.43	1.20
Significance ^{y}	N/A	N/A	N/A	N/A	N/A	N/A	N/A
			Summer				
7.0	6.7 ± 0.22	3.94 ns	0.38 a	2.70 ns	8.85 ab	0.59 ns	1.45 ns
6.5	6.6 ± 0.22	4.48	0.32 b	2.50	8.99 a	0.63	1.43
5.8	6.3 ± 0.45	5.10	0.33 b	2.52	8.54 bc	0.62	1.51
5.0	6.1 ± 0.70	5.16	0.34 b	2.66	8.24 c	0.62	1.54
Significance	N/A	N/A	N/A	N/A	L*	N/A	N/A
Sufficiency levels ^{x}	N/A	4.30	0.30	3.10	2.40	0.35	0.32

^{z} Means with the same letters in a column and season are not significantly different at ($p < 0.05$) as determined by analysis of variance and LSMEANS using the GLIMMIX procedure and type III sum of squares in SAS. ns = not significant. ^{y} Significance established using trend analyses in PROC GLIMMIX. L = linear trend. * = significance at $\alpha = 0.05$. N/A = no trend test conducted because of nonsignificant ANOVA. ^{x} Sufficiency levels obtained from Mills [15].

Soluble micronutrient ions were generally nondetectable or extremely low in AE at each sampling date in both spring and summer 2019 (Tables 6 and 7). At midseason, foliar Mn uptake followed a quadratic trend in relationship with target pH, decreasing by 36% as target pH lowered from 7.0 to 6.5, then increasing by 41% and 29% as target pH decreased to 5.8 and 5.0, respectively (Table 8). However, micronutrient uptake was generally not affected by target pH treatments at either sampling dates in spring and summer 2019 (Tables 8 and 9). When compared against standard percentages in cucumber dry matter [15], all micronutrient levels for both trials were above the lower range of recommended percentage of dry matter for cucumber growth according to *t*-test results using the TTEST procedure in SAS.

Table 6. Midseason micronutrient analysis (mg·L^{-1}) of aquaponic irrigation water at four target pH treatments.

Target pH	Actual pH (±S.E.)	Boron	Zinc	Manganese	Iron	Copper
		Spring				
7.0	6.9 ± 0.16	0.08	0.08	0.03	0.03	0.03
6.5	6.7 ± 0.22	0.09	0.13	0.04	0.09	0.03
5.8	6.4 ± 0.30	0.09	0.10	0.04	0.03	0.02
5.0	6.3 ± 0.46	0.08	0.08	0.05	0.05	0.02
		Summer				
7.0	6.7 ± 0.22	<0.10	<0.10	0.21	<0.10	<0.10
6.5	6.6 ± 0.22	<0.10	<0.10	0.21	<0.10	<0.10
5.8	6.3 ± 0.45	<0.10	<0.10	0.21	<0.10	<0.10
5.0	6.1 ± 0.70	<0.10	<0.10	0.22	<0.10	<0.10
Recommended levels [z]	N/A	0.70	N/A	1.97	6.85	0.07

[z] Recommended levels obtained from Jones [16].

Table 7. End-of-season micronutrient analysis (mg·L^{-1}) of aquaponic irrigation water at four target pH treatments.

Target pH	Actual pH (± S.E.)	Boron	Zinc	Manganese	Iron	Copper
		Spring				
7.0	6.9 ± 0.16	<0.10	<0.10	<0.10	<0.10	0.11
6.5	6.7 ± 0.22	<0.10	<0.10	<0.10	<0.10	0.15
5.8	6.4 ± 0.30	<0.10	<0.10	<0.10	<0.10	0.10
5.0	6.3 ± 0.46	<0.10	<0.10	<0.10	<0.10	0.17
		Summer				
7.0	6.7 ± 0.22	<0.10	<0.10	0.22	<0.10	<0.10
6.5	6.6 ± 0.22	<0.10	<0.10	0.22	0.45	<0.10
5.8	6.3 ± 0.45	<0.10	0.12	0.25	0.22	<0.10
5.0	6.1 ± 0.70	<0.10	0.13	0.27	1.13	<0.10
Recommended levels [z]	N/A	0.70	N/A	1.97	6.85	0.07

[z] Recommended levels obtained from Jones [16].

Table 8. Midseason foliar analysis of micronutrients as percent mg·kg^{-1} percent dry matter for aquaponic cucumbers at four pH treatments.

Target pH	Actual pH (±S.E.)	B	Zn	Mn	Fe	Cu
		Spring				
7.0	6.9 ± 0.16	60 ns [z]	95 ns	46 ns	106 ns	12 ns
6.5	6.7 ± 0.22	57	84	47	116	12
5.8	6.4 ± 0.30	55	82	46	120	11
5.0	6.3 ± 0.46	59	92	47	119	12
Significance	N/A	N/A	N/A	N/A	N/A	N/A
		Summer				
7.0	6.7± 0.22	43 ns	80 ns	184 ab	144 ns	11 ns
6.5	6.6 ± 0.22	38	61	118 c	78	9
5.8	6.3± 0.45	40	62	166 b	89	10
5.0	6.1 ± 0.70	38	79	214 a	86	10
Significance	N/A	N/A	N/A	Q [*y]	N/A	N/A
Sufficiency levels [x]	N/A	30	25	50	50	8

[z] Means with the same letters in a column and season are not significantly different at $p < 0.05$ as determined by analysis of variance and LSMEANS using the GLIMMIX procedure and type III sum of squares in SAS. ns = not significant. [y] Significance established using trend analyses in PROC GLIMMIX. Q = quadratic trend. * = significance at $\alpha = 0.05$. N/A = no trend test conducted because of nonsignificant ANOVA. [x] Sufficiency levels obtained from Mills [15].

Table 9. End-of-season foliar analysis of micronutrients as percent mg·kg^{-1} dry matter for aquaponic cucumbers at four pH treatments.

Target pH	Actual pH (±S.E.)	B	Zn	Mn	Fe	Cu
			Spring			
7.0	6.9 ± 0.16	58 ns [z]	95 ns	60 ns	104 ns	12 ns
6.5	6.7 ± 0.22	50	95	55	96	11
5.8	6.4 ± 0.30	48	86	54	93	11
5.0	6.3 ± 0.46	53	98	57	94.5	12
			Summer			
7.0	6.7 ± 0.22	59 ns	69 ns	391 ns	107 ns	11 ns
6.5	6.6 ± 0.22	53	59	342	100	10
5.8	6.3 ± 0.45	53	67	285	101	10
5.0	6.1 ± 0.70	54	77	367	109	10
Sufficiency levels [y]	N/A	30	25	50	50	8

[z] Means were not significantly different (ns) within column and season at $p < 0.05$ as determined by analysis of variance and LSMEANS using the GLIMMIX procedure and type III sum of squares in SAS. [y] Sufficiency levels obtained from Mills [15].

Macronutrients in the solids deposited from the emitter contained phosphorus and calcium in the highest amounts (Table 10). There were comparatively low amounts of micronutrients, boron and copper, in particular (Table 11). Of the micronutrients, manganese and iron were present in the highest amounts.

Table 10. Macronutrients (mg·kg dried solids^{-1}) contained in suspended solids of aquaculture effluent [z].

	P		K		S		Ca		Mg	
pH	**Pre-Plant**	**Post-Plant**	**Pre-Plant**	**Post-Plant**	**Pre-Plant**	**Post-Plant**	**Pre-Plant**	**Post-Plant**	**Pre-Plant**	**Post-Plant**
7.0	2075	1672	757	519	1050	850	1713	2825	300	300
6.5	2628	2392	591	498	1100	875	2625	4188	325	300
5.8	1892	1580	716	540	1150	925	5100	5013	350	400
5.0	1693	2037	747	581	1213	963	3550	3238	350	325

[z] Solids were dried at 105 °C for 24 h before analysis.

Table 11. Micronutrients (mg·kg dried solids^{-1}) contained in suspended solids of aquaculture effluent [z].

	B		Zn		Mn		Fe		Cu	
pH	**Pre-Plant**	**Post-Plant**	**Pre-Plant**	**Post-Plant**	**Pre-Plant**	**Post-Plant**	**Pre-Plant**	**Post-Plant**	**Pre-Plant**	**Post-Plant**
7.0	5	13	175	150	625	1250	850	938	25	25
6.5	5	1	188	38	338	600	963	488	25	13
5.8	5	1	125	150	450	2238	1038	813	25	13
5.0	1	1	13	150	13	1963	1113	1125	25	13

[z] Solids were dried at 105 °C for 24 h before analysis.

4. Discussion

4.1. Nutrients in Aquaculture Effluent

Nitrates in the summer 2019 trial showed a general trend of increasing concentration with increasing pH (Tables 2 and 4), which supports the trend observed by Zou [17]. Soluble K increased 1.68% from pH 5.0 to 7.0 (Table 2), which is not practical to consider when using pH for the sole purpose of increasing its availability. pH has been shown to have an effect on phosphorus availability in solution [18]; however, this trend was not observed in this study, potentially due to the inability of the acid injection to reach the target pH. Calcium was added to the fish culture tank in the form of hydrated lime to maintain the pH at 7.0. It is possible that the added calcium in solution reacted with the sulfuric acid used to adjust irrigation pH for the plant greenhouse to form calcium sulfate.

Shukla [19] found that the solubility of calcium sulfate increased with decreasing pH, which could explain the resulting increased calcium observed at lower pH in the summer trial. Magnesium is applied to the system through hydrated lime and magnesium oxide (MgO) in the fish feed. Uneaten feed can become available to the plants as small particles carried by the irrigation water. Solubility of MgO in water occurs more readily under acidic conditions [20], which could account for the observed increase in Mg under the low-pH treatments in the summer trial.

When compared against nutrient concentrations in standard hydroponic solution for cucumber production, all nutrients, except for Zn and Cu, in both mid- and late-season measurements were observed to be statistically lower than concentrations recommended for hydroponic growth [8].

4.2. Nutrients Assimilated into Plant Tissue

In this study, pH was observed to have an effect on phosphorus assimilation into plant tissues, indicating that pH could have a two-fold effect on phosphorus availability in solution and uptake through the roots. As previously mentioned, pH affects the availability of phosphorus in solution [18,21]. In regards to phosphorus uptake, it has been shown that there is a direct relationship between pH and phosphorus uptake with increased phosphorus uptake at high pH [22]. It has also been shown that the pH of the apoplast, the space between the cell wall and the cell membrane, influences phosphorus uptake where high pH (7.0) reduced uptake [23]; however, neither of these trends were observed in this study. It is important to note that interactions apart from phosphorus availability in solution could impact phosphorus uptake, including the presence of other elements in solution, which can lead to precipitation with phosphorus and thus its uptake and assimilation into plant tissues, especially when the availability of those elements is also affected by pH. The most prominent example is the formation of calcium phosphate precipitate, which occurs at high pH [18].

Several other macronutrients showed inconsistent changes with pH from mid- to late-season measurements and across trials. While light and temperature changes occurred due to changing seasons, yield and growth were not statistically different between seasons. Nitrate assimilation into leaf tissue varied significantly with pH during the midseason measurements of the summer 2019 trial showing highest assimilation at pH 5.8 and the lowest at pH 6.5, a 5% increase between the two treatments (Table 3). This was not observed in the late-season measurements nor at all in the spring trial. Calcium uptake showed the highest levels at pH 6.5 in the late-season measurements of the summer 2019 trial, a 9.1% increase from 5.0 to 6.5 (Table 5). Differences in mid- to late-season measurements could be due to nutrients being allocated to different plant organs at different rates according to seasonal changes or changes physiological requirements once fruit-set and development occurs [24,25]. Clark and Richardson [26] have recorded higher amounts of nutrients required once the reproductive phase of the plant's life cycle has been reached.

Manganese was the only micronutrient whose assimilation into plant tissues was affected by the AE pH. In soil-based systems, manganese uptake occurs by facilitated diffusion in soil systems and decreases with the addition of lime to the soil. The decreased uptake at higher pH observed could be due to the addition of lime to the irrigation source to manage pH in the fish culture tank.

When compared against standard tissue concentrations for healthy cucumber tissue [15] all nutrients—both mid- and late-season for the spring and summer trials—were observed to be statistically higher than the lower range of recommended tissue concentrations for cucumber leaf tissue. Cucumber plants were not deficient in any essential plant nutrients despite the irrigation water undersupplying nutrients, which indicates that nutrients are coming from another source and being made available for plant uptake. This could be from the accumulation of solids in the container over time, allowing plant roots to mine for nutrients in the container, or even from aerobic processes increasing available nutrients in the solid particles, which Monsees et al. [13] observed in aquacultural sludge.

4.3. Nutrients in Aquaculture Effluent Solids

Preliminary analysis of solids carried in the irrigation water was consistent with observations by Monsees et al. [13]. Phosphorus and calcium constituted the largest proportion of the solids. Sulfur, magnesium, iron, and manganese contributed to solid composition at 0.06%, 0.03%, 0.03%, and 0.002%, respectively. These nutrients could be made available to plants, providing adequate nutrition in aquaponic production. Additionally, Monsees et al. found that mobilization of these nutrients through aerobic digestion further increases the amount of phosphorus and potassium available. High levels of manganese and iron were observed in the captured solids, compared to low levels of boron and copper. Boron and copper were also observed to be low in solution. Despite low levels of nutrients, both in solution and contained in solids, compared to levels available in the same amount of hydroponic solution (Table 12), cucumber plants were not deficient in plant-essential nutrients. Examination of raw numbers of nutrient levels in both the solid and liquid fraction of aquaculture effluent may not be enough to capture the effects of nutrient flow in the system. It is possible that there are interactions occurring in the media over time that allow for adequate plant nutrition.

Table 12. Comparison of daily nutrient supply (mg) from aquaculture effluent ^{z} (AE) and hydroponic solution ^{y} based on irrigation rate of 7 L daily.

Nutrient	AE			Hydroponic Solution
	Solid	Liquid	Total	
N	N/A	651	651	1512
P	2.06	98	100	406
K	0.779	686	687	2002
S	1.25	N/A	N/A	N/A
Ca	5.55	805	811	1295
Mg	0.381	203	203	1295
B	0.005	0.7	0.705	4.9
Zn	0.136	0.84	0.976	N/A
Mn	0.490	1.75	2.24	13.8
Fe	1.13	1.54	2.67	478.0
Cu	0.027	0.7	0.727	0.49

^{z} Data for aquaculture effluent (AE) at target pH of 5.8. ^{y} Values extrapolated from Jones [16].

5. Conclusions

Certain nutrients are affected by the pH of nutrient solution either by influencing the ionic form of the nutrient in solution or by influencing the nutrient's uptake and assimilation into plant tissue. However, in the current study we observed no consistent increase in nutrient availability or uptake that would necessitate the use of pH adjustment in a decoupled aquaponic system. Furthermore, the observed effects of pH did not translate to an increase in yield at the pH range observed in this study.

Acids used to manage pH should be taken into further consideration. We used citric and sulfuric acids in the current experiment. Citric acid was chosen because it did not contribute nutrients to the system and represented a weak, organic acid. Sulfuric acid was chosen as a strong, mineral acid which not only increased plant-available sulfur but also potentially decreased calcium in solution due to precipitation of calcium sulfate. In either case, target pH was reached at the injector but increased again as AE moved through the irrigation system. The high buffering capacity in the system may have been due to multiple factors, including large additions of hydrated lime to the aquaculture unit, high amounts of solid and soluble organic matter containing pH-buffering functional groups, and/or possible denitrification reactions in irrigation lines. In order to overcome the observed pH drift in this type of system, pH sensors to inform the acid injector would need to be installed at the drip emitter position instead of at the injector, which is not a typical system design.

In either case, pH treatment or choice of acid did not affect cucumber yield, which can be explained by the fact that nutrient uptake was not limited by treatment. Levels of plant-essential elements incorporated into the plant tissues was sufficient despite the seemingly poor nutrition of the irrigation water observed in relation to a hydroponic solution standard. The cucumber plants were not deficient in any essential plant nutrients even with the irrigation water undersupplying nutrients. This indicates that there are nutrients coming from another source and being made available for plant uptake. The additional nutrients could come from the accumulation of solids in the container over time, allowing plant roots to mine for nutrients in the container, or even from aerobic processes increasing available nutrients in the solid particles, which Monsees et al. [13] observed in aquacultural sludge. Further research that investigates the nutritional and microbial composition of suspended solids carried in the irrigation water and dissolution of nutrients over time would provide additional information on the nutrient dynamics in this system.

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