Ammonium in Nutrient Solutions Decreases Free Chlorine Concentration from Sodium Hypochlorite

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Abstract. The objective was to quantify the effect of water-soluble fertilizers on concentration of free chlorine level in a sodium hypochlorite solution. Research on the disinfection strength and phytotoxicity risk of chlorine compounds is needed, because control of waterborne pathogens has been based on response to free chlorine, whereas dual injection of fertilizer and chlorine is a common horticultural practice. Free chlorine from sodium hypochlorite was applied at 2.6 mg·L⁻¹ chlorine (Cl) to deionized water only (control) or deionized water with 11 nutrient solutions at 200 mg·L⁻¹ nitrogen (N). Nutrient solutions included reagent-grade ammonium sulfate (NH₄)₂SO₄, ammonium nitrate (NH₄NO₃), potassium nitrate (KNO₃), and urea salts and seven commercial blended N–P–K water-soluble fertilizers that contained both macro- and micronutrients. Commercial fertilizers contained ammonium-N at 0% to 50% of total-N, urea-N at 0% to 14% of total-N, and nitrate-N at 50% to 93% of total-N. Free Cl (mg·L⁻¹), total Cl (mg·L⁻¹), and oxidation-reduction potential (ORP, in mV) were measured 2 min and 60 min after Cl was applied. Combined Cl was calculated as the difference between the total and free Cl measurements. All solutions were maintained at pH 6 and 25 °C. In the control solution, free Cl was 2.6 mg·L⁻¹ after 2 minutes and decreased to 2.2 mg·L⁻¹ after 60 minutes. The ammonium-containing solutions (NH₄)₂SO₄ and NH₄NO₃ resulted in free Cl below 0.1 mg·L⁻¹ after 2 minutes. Urea reacted more slowly than ammonium salts, whereby free Cl decreased to 2.3 mg·L⁻¹ after 2 minutes and 0.4 mg·L⁻¹ after 60 minutes. In contrast, KNO₃ had less impact on free Cl with 2.4 mg·L⁻¹ free Cl available at both 2 minutes and 60 minutes. With all commercial fertilizers tested, free Cl decreased after 2 minutes to below 0.1 mg·L⁻¹. Total Cl remained above 2 mg·L⁻¹ after 60 minutes in all treatments, indicating that the majority of Cl was in a combined form for ammonium and urea salts and commercial fertilizers. The ORP of commercial fertilizer blends and ammonium-containing salts was lower than 600 mV, whereas deionized water, KNO₃, and urea treatments had ORP levels above 650 mV. Nutrient solutions containing ammonium or urea required 20 mg·L⁻¹ or more of applied Cl to provide residual free Cl above 2 mg·L⁻¹ at 2 minutes.

Chlorine from calcium hypochlorite, sodium hypochlorite, or Cl gas sources has complex chemistry in irrigation water. Once added to water, Cl is converted to hypochlorite (OCl⁻) and hypochlorous acid (HOCl), which along with dissolved Cl gas are collectively termed free Cl. The balance between hypochlorous acid and hypochlorite is pH-dependent, whereby hypochlorous acid predominates at solution pH below 7.5 or hypochlorite ions above pH 7.5 (Morris, 1966). Water pH also influences the sanitizing strength

of a Cl solution, because HOCl is estimated at up to 80× more effective as a biocide than OCl⁻ (White, 1992). The effect of pH on control of waterborne pathogens in a Cl solution was illustrated by Lang et al. (2008), whereby control of *Pythium aphanidermatum* and *P. dissotocum* zoospores was achieved at either 2.0 mg·L⁻¹ Cl at pH 8.1 or 0.5 mg·L⁻¹ Cl at a pH of 6.3.

There is extensive research on effects of free Cl on waterborne plant pathogens. Control of *Pythium* and *Phytophthora* zoospores has been reported with 2 mg·L⁻¹ of free Cl (Cayanan et al., 2009; Hong and Richardson, 2004; Hong et al., 2003; Lang et al., 2008). Chlorine oxidizes and chlorinates living tissue and organic compounds, resulting in damage to membranes, enzymes, and nucleic acids of microorganisms (Stewart and Olson, 1996). Increasing concentration of Cl, lowering solution pH, increasing ORP, and increasing contact time are factors that improve pathogen control (Lang et al., 2008).

Oxidative strength of a Cl solution can be measured in millivolts (mV) using an ORP meter. A positive correlation was found between

ORP and control of coliform bacteria by chlorination of wastewater (Yu et al., 2008). Coliforms and pathogenic bacteria were rapidly controlled in post-harvest wash water if ORP was maintained between 650 and 700 mV (Suslow, 2004). Lang et al. (2008) found that *Pythium aphanidermatum* and *P. dissotocum* zoospores were killed within 0.25 to 0.5 min when ORP was above 780 mV in chlorinated water.

In the presence of organic and inorganic N including ammonia, a range of equilibria reactions occur to form “complexed” chlorinated molecules such as chloramines in equations [1 to 3] adapted from the U.S. Environmental Protection Agency (EPA, 1999). The concentration of both free Cl and complexed Cl together make up the concentration of “total” Cl.

In municipal water treatment facilities, a dose of one part of inorganic N to every three parts of free Cl reportedly results in 99% conversion of free Cl to chloramines after 0.2 s at pH 7 or 147 s at pH 4 (White, 1992). In horticulture irrigation, water-soluble fertilizer containing ammonium, nitrate, and/or urea N is often supplied in irrigation between 100 and 200 mg·L⁻¹ total N and then treated with free Cl concentrations between 1 and 2 mg·L⁻¹ Cl. With such a high N to Cl ratio, the majority of free Cl would therefore be expected to rapidly convert to chlorinated N forms. Chlorine also reacts with urea, although the chain of reactions is more complex and slow-acting than chlorination of ammonia (Blatchley and Cheng, 2010).

Chloramines are considered weaker sanitizers than free Cl from hypochlorous acid (White, 1992) because of the longer contact time that chloramines require to control human pathogens compared with an equal concentration of free Cl. Control of 99% of *Escherichia coli* with hypochlorous acid at 1 mg·L⁻¹ Cl required a 1-min contact time, whereas with combined Cl forms including NH₂Cl and NHCl₂ at 1 mg·L⁻¹ Cl required more than 100 min (Akin et al., 1982). However, the greater stability of chloramines in the presence of organic compounds compared with hypochlorous acid may increase penetration of chloramines into biofilm, resulting in greater inactivation of biofilm bacteria (LeChevallier et al., 1988). The American Water Works Association (AWWA, 1991) provided guidelines for residual concentration of between 0.5 to 1 mg·L⁻¹ chloramine for disinfection of groundwater (White, 1992).

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Table 1. Nutrient concentrations used in the study (reported by manufacturer) on a percentage by mass for individual nutrients.

Fertilizer (N-P-K)	Source ^z	CaCO ₃ equiv. ^y	Acid base (A/B) ^x	NH ₄ -N	NO ₃ -N	Urea-N	P	K	Ca	Mg	S	B	Cu	Fe	Mn	Mo	Zn
N13-0.9P-10.1K	E	167	B	0.3	11.9	0.8	0.9	10.8	6.0	3.0	0.0	0.02	0.02	0.06	0.03	0.01	0.03
N15-2.2P-12.5K	E	65	B	1.1	11.8	2.1	2.2	12.5	5.0	2.0	0.0	0.02	0.02	0.07	0.04	0.01	0.04
N14-1.8P-11.6K	G	100	B	2.0	12.0	0.0	1.8	11.6	5.0	2.0	0.0	0.02	0.02	0.07	0.03	0.01	0.03
N16-2.1P-13.7K	G	7	B	4.1	12.4	0.0	2.1	13.7	3.3	1.6	0.0	0.02	0.02	0.08	0.04	0.01	0.04
N15-2.2P-16.6K	E	24	A	4.3	10.7	0.0	2.2	20.8	0.0	2.5	1.0	0.01	0.02	0.07	0.04	0.07	0.04
N20-0.9P-16.6K	E	145	A	7.2	12.8	0.0	0.9	16.6	0.0	1.1	0.7	0.02	0.02	0.10	0.05	0.01	0.05
N19-5.6P-19.4K	G	340	A	9.7	9.7	0.0	2.5	16.1	0.0	0.0	0.0	0.02	0.02	0.10	0.05	0.01	0.05
Urea	Reagent			0.0	0.0	45.7	0.0	0.0	0.0	0.0	0.0						
Potassium nitrate	Reagent			0.0	13.6	0.0	0.0	37.9	0.0	0.0	0.0						
Ammonium sulfate	Reagent			20.8	0.0	0.0	0.0	0.0	0.0	0.0	23.8						
Ammonium nitrate	Reagent			17.2	17.2	0.0	0.0	0.0	0.0	0.0	0.0						

^zG = Greencare Fertilizers; E = Everris Co.^yReported kg/metric ton.^xPotential acidity or basicity.

such as from wells or irrigation catchment areas. Based on experience with control of human pathogens in water supply, irrigation of edible crops with complexed Cl is likely to require longer contact times and/or higher total Cl application concentrations compared with free Cl for control of human pathogens. However, data are not available for plant pathogens.

There are limited research data on the residual level of free or total Cl in the presence of nutrient solutions despite the common practice of dual injection of Cl and water-soluble fertilizer and the potential impact on pathogen control. The research objective of this study was to determine the free and total Cl and ORP responses over time when nutrient solutions were blended with sodium hypochlorite.

Materials and Methods

Three experiments were run: Expt. 1, multiple fertilizers, 200 mg·L⁻¹ N from seven commercial blended fertilizers containing macro- and micronutrients and reagent-grade nutrient solutions were mixed with 2.6 mg·L⁻¹ Cl from sodium hypochlorite. In Expt. 2, multiple N concentrations, five concentrations of N from 0 to 600 mg·L⁻¹ from four reagent-grade nutrient solutions were mixed with 2.6 mg·L⁻¹ Cl. Expt. 3 high applied Cl quantified whether 10 or 20 mg·L⁻¹ applied Cl was sufficient to provide a residual of 2 mg·L⁻¹ Cl after a 2-min contact time in the presence of 200 mg·L⁻¹ N from commercial fertilizers and reagent-grade N salts.

The 11 nutrient solutions for Expt. 1 multiple fertilizers are shown in Table 1. Nutrient solutions ranged in the contribution of ammonium, nitrate, and urea to total N as well as presence of other macro- and micronutrients (Fig. 1). Both commercial blended fertilizers and reagent-grade salts were tested along with deionized water as the control. Fertilizers were sourced from two different manufacturers, Greencare Fertilizers (Kankakee, IL) and Everris NA, Inc. (Dublin, OH). All commercial fertilizer blends tested contained some ammonium or urea, including high nitrate fertilizer such as 13N-0.9P-10.8K (Fig. 1).

In Expt. 2 multiple N concentrations, the four reagent-grade salts from Table 1 were

Fig. 1. Nitrogen (N) source and its effect on free and total chlorine and oxidation-reduction potential (ORP) in Expt. 1 multiple fertilizers. **A** shows the percent contribution of ammonium, nitrate, or urea to total nitrogen in each nutrient source. **B–D** show the effect of 200 mg·L⁻¹ N from commercial fertilizer blends and reagent-grade nutrient solutions on (**B**) free chlorine (Cl), (**C**) total Cl, and (**D**) ORP after injecting sodium hypochlorite at 2.6 mg·L⁻¹ Cl and measured after 2 min and 60 min contact times. Letters represent mean comparisons using Tukey's honestly significant difference at the $P = 0.05$ level for the interaction of nutrient source and measurement time, $n = 3$ replicates.

applied at five N concentrations (0, 75, 150, 300, and 600 mg·L⁻¹ N) in combination with 2.6 mg·L⁻¹ Cl. In Expt. 3 high applied Cl, Cl was applied at 10 or 20 mg·L⁻¹ Cl from sodium hypochlorite to the fertilizer treatments in Table 1 at 200 mg·L⁻¹ N. Expt. 3 was analyzed separately from Expt. 1 despite a similar experimental design, because the procedure was run at a different time, dilution of the samples was necessary when analyzing higher levels of free and total Cl levels in Expt. 3, to provide in-range concentrations for measurement, and measurements were made at 2 min only. No sample dilution was required for Expt. 1.

Nutrient solutions were prepared in a closed system in 4-L plastic containers in all experiments. Nutrient solutions were adjusted to pH 6.0 using KOH or H₂SO₄ with constant mixing and were maintained at 25 °C. Chlorine solution was prepared by adding Clorox Regular-Bleach (6.15% sodium hypochlorite) (Clorox Company, Oakland, CA) to deionized water and then injecting sufficient solution into the closed nutrient solution container through a septum to provide a final concentration of 2.6 mg·L⁻¹ Cl for Expts. 1 and 2 and 2.6, 10, or 20 mg·L⁻¹ Cl in Expt. 3.

Free and total Cl, pH, temperature, and ORP were measured in the nutrient solution before addition of Cl and 2 and 60 min after addition of Cl. Free and total Cl concentrations were measured as described by methods

2350 B and 4500 Cl. F. 1d, respectively (American Public Health Association, 1995). Colorimetric determination of mg·L⁻¹ Cl was performed using a Thermo Scientific Orion AQUAfast IV® AQ4000 colorimeter, AC4P71 Chlorine Free and Chlorine Total reagent-packs (Thermo-Fisher, Barrington, IL). Solution pH was measured using an Orion Model 61-65 (Thermo-Fisher), solid-state probe and ORP measured using an Orion 91-79 Triode electrode and both pH and ORP were displayed simultaneously on a Four-Star meter (Thermo-Fisher).

In each experiment, the design was a randomized complete block design. On each day (block), a single replicate of each combined nutrient and chlorine treatment was run in random order. There were three blocks (days) for Expts. 1 and 2 and two blocks (days) for Expt. 3. For all experiments, data were analyzed using analysis of variance with SAS PROC GLM (SAS Version 9.1; SAS Institute, Cary, NC). Means were separated using Tukey's honestly significant difference test at the $P \leq 0.05$ level.

Results and Discussion

In Expt. 1 multiple fertilizers, there was a significant ($P \leq 0.001$) main effect of N source on the concentration of free Cl, ORP, and total Cl (Table 2), and time (2 or 60 min) affected total Cl ($P \leq 0.05$). With total Cl measurements there was a significant ($P \leq 0.001$) block effect of the measurement day. There was a two-way interaction ($P \leq 0.001$) of N source and time for measured ORP.

Control solutions of deionized water did not differ in measured variables between 2 min and 60 min (Fig. 1). At 2 min, free Cl (2.6 mg·L⁻¹ Cl) in the control was higher than

the free Cl concentration measured in all nutrient solutions containing commercial blended fertilizer, which was below the minimum reportable limit (0.15 mg·L⁻¹ Cl) of the colorimeter. Commercial blended fertilizers had less effect on total Cl than on free Cl. The ORP was lower in most commercial blended fertilizers compared with the control at both 2 and 60 min, regardless of the proportion of each N form.

Potassium nitrate and urea had free Cl concentration equal to the control at 2 min and 60 min, whereas with ammonium nitrate and ammonium sulfate, the free Cl concentrations were below the 0.15-mg·L⁻¹ Cl detectable limit of the meter. The ORP was higher in potassium nitrate than ammonium nitrate and ammonium sulfate and was equal to the ORP of the control. The free Cl concentration measured in the urea solution was higher than the total Cl at 60 min. Therefore, estimation of total and/or free Cl in solutions containing urea was not accurate because free Cl forms (hypochlorous acid, hypochlorite, and dissolved Cl) are components of total Cl, and free Cl should therefore be equal to or lower than total Cl. Urea had a higher ORP than any other solution at 60 min, emphasizing the complexity of reactions between Cl and urea (Blatchley and Cheng, 2010).

In Expt. 2 multiple N concentrations, there were significant interactions between reagent-grade N sources, concentration, and time (Table 3). There was no difference in the free Cl concentration for the control between 2 and 60 min (Fig. 2). Among the reagents, the higher level of free Cl was measured in nutrient solutions with potassium nitrate than urea. The lowest free Cl levels occurred in ammonium nitrate or ammonium sulfate at 75 to 600 mg·L⁻¹ N. Free Cl level did not

Table 2. Analysis of variance summary for Expt. 1 multiple nitrogen (N) sources, where free chlorine (Cl) was applied to 11 nutrient solutions at 200 mg·L⁻¹ N and a deionized water control (N source) at 2.6 mg·L⁻¹ Cl with 2 min and 60 min contact times (Time).²

	Free Cl (mg·L ⁻¹)	ORP (mV)	Total Cl (mg·L ⁻¹)
N source	***	***	***
Time	NS	NS	*
N source × time	NS	**	NS
Block	NS	NS	***

²The block was the measurement day (n = 3). NS = not significant, * $P < 0.05$, ** $P < 0.01$, *** $P < 0.001$. ORP = oxidation-reduction potential.

Table 3. Analysis of variance summary for Expt. 2 multiple nitrogen (N) concentrations where free chlorine (Cl) was applied at 2.6 mg·L⁻¹ Cl to four reagent nutrient solutions (N source) at six concentrations from 0 to 600 mg·L⁻¹ N ([N]) with 2- or 60-min contact time (Time).²

	Free Cl (mg·L ⁻¹)	ORP (mV)	Total Cl (mg·L ⁻¹)
N source	***	***	***
[N]	***	***	***
Time	NS	***	***
N source × time	*	***	***
N source × [N]	***	***	***
Time × [N]	NS	NS	***
N source × time × [N]	NS	***	***
Block	NS	NS	NS

²The block was the measurement day (n = 3). NS = not significant, * $P < 0.05$, ** $P < 0.01$, *** $P < 0.001$.

Fig. 2. Effect of nitrogen (N) concentration from four reagent-grade N sources on free chlorine (Cl) after injecting sodium hypochlorite at 2.6 mg·L⁻¹ Cl in Expt. 2 multiple N concentrations, measured after (A) 2 min and (B) 60 min. Letters represent mean comparisons using Tukey's honestly significant difference at the $P = 0.05$ level within each measurement time, n = 3 replicates.

decline significantly compared with the control for potassium nitrate or urea at 75 to 600 mg·L⁻¹ N.

In Expt. 3 high applied Cl, there were significant main and interaction effects of N source and applied Cl concentration ($P \leq 0.001$; Table 4). Free Cl concentrations for the control at 10 or 20 mg·L⁻¹ applied Cl were significantly different (9.9 and 19.9 mg·L⁻¹ Cl, respectively; Fig. 3). At both applied Cl concentrations, ammonium sulfate and ammonium nitrate, and all commercial fertilizer blends resulted in the same free Cl level, ranging from 0.20 to 1.26 mg·L⁻¹ Cl. For urea and potassium nitrate, the free Cl concentration was not significantly different from the control at both applied Cl concentrations.

The decrease in free Cl levels observed with reagent-grade salts and commercial fertilizer blends in these experiments has practical implications for combining fertilization and water sanitation in horticulture. Where ammonium was present in the solution, the majority of Cl is likely to be complexed, even at low concentrations (for example, 37.5 mg·L⁻¹ NH₄-N from ammonium nitrate applied at 75 mg·L⁻¹ N; Fig. 2). Most commercial fertilizers contain some ammonium, including ammonium sulfate, ammonium phosphate, ammonium nitrate, or as a component in calcium nitrate fertilizer. Nitrate did not decrease free Cl concentration, and urea reacted slowly, indicating these N forms would be more compatible with free Cl than

ammonium. Another management strategy would be to use holding tanks to inject Cl into unfertilized water to provide adequate contact time before injecting fertilizer. A further option to reduce Cl demand of fertilizer would be to apply granular or controlled-release fertilizer directly to the growing substrate rather than water-soluble fertilizer.

In the presence of ammonium, high applied concentration of Cl would be required to achieve residual free Cl for control of plant pathogens. Even at 20 mg·L⁻¹ Cl applied free Cl in Expt. 3, in the presence of ammonium N, the level of free Cl rapidly decreased to 1.26 or less mg·L⁻¹ Cl. Given that as high as 2 mg·L⁻¹ of free Cl may be required for control of *Pythium* and *Phytophthora* zoospores (Cayanan et al., 2009; Hong and Richardson, 2004; Hong et al., 2003; Lang et al., 2008), it may be difficult to achieve that level of free Cl without more than 10 times the applied Cl concentration. We have observed that some growers are indeed applying several times higher chlorine concentrations than 2 mg·L⁻¹ Cl when trying to control dosage based on a free Cl target while also injecting water-soluble fertilizer that contains ammonium. The implications in terms of phytotoxicity risk and pathogen control of that practice are unknown, and chlorination cost would increase.

An alternative sanitizing technology other than Cl could be used that is more compatible with water-soluble fertilizer, for example Cl dioxide. Copes et al. (2004) found the efficacy of Cl dioxide in nutrient solutions for control of *Fusarium oxysporum* was more affected by pH and micronutrients than N, and Cl dioxide at 2 mg·L⁻¹ was able to provide 50% decrease of *F. oxysporum* in nutrient solutions that contained 100 mg·L⁻¹ N and 3 mg·L⁻¹ of micronutrients (copper, iron, manganese, and zinc).

Our results emphasize the need for monitoring of chlorine levels and ORP in horticulture applications, where N and other contaminants create a Cl demand that is likely to vary over time, thereby affecting residual free Cl concentration. Research conducted by Lang et al. (2008) demonstrated that free Cl concentration and ORP were important for

disinfestation of *Pythium aphanidermatum*. Measurements of pH, free Cl concentration, and ORP are standard operating procedures to monitor to ensure treatment efficacy in water and wastewater treatment facilities (EPA, 1999), and these procedures should be adapted to horticulture operations.

Conclusions

The low level of free Cl found in these experiments when combined with fertilizers that contained ammonium indicate that hypochlorous acid is likely to be rapidly converted to complexed Cl forms when water-soluble fertilizers containing ammonium are used in combination with Cl. Urea also reacted with hypochlorous acid, although our results showed inconsistency in colorimetric measurement of free vs. total Cl in urea solution. Urea undergoes multiple N-chlorination steps that occur over a period of hours at 20 to 30 °C (Blatchley and Cheng, 2010). In contrast, free Cl was not decreased by nitrate N.

Several alternative strategies may avoid N reactions with Cl, including use of solid fertilizers, injecting Cl in a holding tank before fertilizer injection, using nitrate-based water-soluble fertilizer, or possibly using injecting urea and Cl with a short contact time. Monitoring is essential with Cl and any other water treatment technology with important parameters including solution pH, active ingredient levels of the sanitizing agent, ORP (if oxidizers are used), the density of colony-forming units of indicator organisms such as aerobic bacteria plate counts, and the presence or absence of particular pathogenic organisms of concern.

Research is needed on the efficacy of chloramines for controlling plant pathogens in horticulture. Chloramines have greater stability than hypochlorous acid, which results in increased residual activity for applications such as biofilm control in irrigation lines (LeChevallier et al., 1988) but requires a longer contact time to control bacteria (Degreimont, 1979). Unlike hypochlorous acid, chloramines do not form carcinogenic trihalomethanes (Degreimont, 1979), although trichloramine (NCl₃, a byproduct of urea and hypochlorous acid) is a respiratory and skin irritant (Blatchley and Cheng, 2010). Chloramines caused root necrosis in hydroponic lettuce when a low hypochlorous acid concentration (0.3 mg·L⁻¹ Cl) was combined with 9.4 mg·L⁻¹ of ammonium N (Date et al., 2004). More information is clearly needed to determine the implications of the common practice of dual injection of fertilizer and Cl in horticulture.

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Table 4. Analysis of variance summary for Expt. 3 high applied chlorine (Cl) where free Cl was applied at two concentrations of 10 or 20 mg·L⁻¹ Cl ([Cl]) to 11 nutrient solutions at 200 mg·L⁻¹ nitrogen (N) and a deionized control (N source) and free Cl was measured after 2-min contact time.^z

	Free Cl (mg·L ⁻¹)
N source	***
[Cl]	***
N source × [Cl]	***
Block	NS

^zThe block was the measurement day (n = 2).

NS = not significant, * $P < 0.05$, ** $P < 0.01$, *** $P < 0.001$.

Fig. 3. Effect of sodium hypochlorite at 10 mg·L⁻¹ chlorine (Cl) and 20 mg·L⁻¹ Cl measured after 2 min in 11 nutrient solutions at 200 mg·L⁻¹ nitrogen (N) and a deionized control in Expt. 3 high applied Cl. Letters represent mean comparisons using Tukey's honestly significant difference at the $P = 0.05$ level, n = 2 replicates.

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