

Minimising chlorate in the electrolytic disinfection of irrigation water

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To the background of discussions on problematical residues in plant-derived foods, the procedure for minimising chlorate content in the electrolytic disinfection of irrigation water is of great importance. As a source of adjustment recommendations was investigated, on the basis of a brine electrolysis plant (single chamber system), how much chlorate is produced during the electrochemical production procedure for the disinfectant solution and how its proportion changes during storage under warm greenhouse conditions. Investigated additionally was the effect the plant fertiliser ammonia has on disinfectant substances. Consequently, minimising chlorate in the electrolytic water disinfection could be achieved by using a cooling system for the electrolysis reactor and the disinfectant storage tank. Additionally recommended is a short term storage tank for the disinfectant solution. Regarding dosage of disinfectant solution, it was shown that ammonia markedly increases usage of disinfectant or chlorate input into the irrigation water. Thus, it is recommended that dosage is controlled by chlorine sensor so that alterations of chemical processes in water (e.g. chlorine loss) can be accounted.

Keywords

Water disinfection, brine electrolysis, hypochlorite, chlorate, chlorine loss

Where using surface water (esp. stored rainwater) for irrigation, and re-using surplus irrigation water (esp. in closed systems), the risk of spreading waterborne phytopathogens is increased (SALLY 2011). For this reason, there already exist several procedures for water disinfection that, in principal, are suitable for plant production. Hereby, we differentiate between physical disinfection procedures (heat, UV radiation, filtration) and chemical procedures (ozone, hydrogen peroxide, copper/silver ions, chlorine dioxide, chlorine) (VAN OS 2010). Compared with the physical procedures, the chemical procedures have the advantage of potential efficacy within the entire irrigation system and can thus prevent disease spread from plant to plant (WOHANKA et al. 2015).

However, problematical with chemical disinfection procedures is possible accumulation of undesirable by-products in the water or in the plants cultivated with the help of this water, or in plant parts washed by it. Thus, a comprehensive study with 1020 samples of plant-based foods showed that with around 10% of the samples, ≥ 0.01 mg chlorate (ClO_3^-) per kg of food was detectable (KAUFMANN-HORLACHER 2014). Chlorates (e.g. sodium chlorate or potassium chlorate) were used as non-selective herbicides. Since 2010, these chemicals are no longer approved as herbicides within the European Union (EUROPEAN COMMISSION 2008). Additionally, chlorate can cause damage to red blood cells and inhibit iodine uptake by humans (BfR 2014). For this reason, the European Food Safety Authority published a preliminary review of chlorate threshold values in food, based on an acute reference dose of 0.036 mg per kg bodyweight and day (EFSA 2015). The toxicological risk assessment is ongoing because the knowledge collected up until now is not enough to support a conclusive health-based evaluation.

As to how chlorate arrives in the food, among the reasons considered are application of chlorinated irrigation and washing water in pre- and post-harvest periods (KAUFMANN-HORLACHER 2014). There is already evidence in this respect that circulating irrigation water (NFT technique) with a chlorine disinfection using electrolytically produced hypochlorite (ClO^-) can lead to concentration of chlorate in tomatoes (DANNEHL et al. 2015a). The disinfection procedure applicable can feature application of commercially available chlorine bleach liquor (sodium hypochlorite) delivered in barrels, as well as disinfectant substances produced via on-site systems. Hereby, the latter, so-called electrolysis systems, have so far been mainly used for treatment of drinking and pool water (DYGUTSCH and KRAMER 2012). From salt (e.g. NaCl) water and electrical current is hereby produced a hypochlorite-content disinfectant solution with the necessary energy delivered into the brine (electrolyte or sole) via electrodes (anode and cathode). During the resultant material conversion (redox reaction), electrons (e^-) are transferred, whereby those from negatively charged ions (chloride) are deposited on the anode side (Eq. 1), and those from positively charged ions or neutral substances (water) are received on the cathode side (Eq. 2). Finally (Eq. 3), hydroxide (OH^-) and chlorine (Cl_2) react to give hypochlorite (ClO^-) and chloride (Cl^-).



In the context of brine electrolysis, two different chlorine compounds can be formed, depending on pH value of the water to be disinfected: hypochlorite (ClO^-) or hypochlorous acid (HClO). Both are, however, designated as free chlorine (CLARK and SMAJSTRLA 1992). The related dissociation reaction is reversible (Eq. 4). Hereby, the hypochlorite proportion reduces as the hypochlorous acid proportion increases, and vice versa. Furthermore, at $\text{pH} < 6$ the hypochlorous acid (HClO) has a high disinfection efficacy, and from $\text{pH} > 6$ the hypochlorite (ClO^-) increasingly has a reduced disinfection efficacy. From a $\text{pH} > 7.5$ the disinfection effect is regarded as insufficient (MEBALDS et al. 1996).



In an aqueous solution the disproportionation of three parts hypochlorite (ClO^-) occurs in a multi-stage reaction to one part chlorate (ClO_3^-) and two parts chloride (Cl^-). The overall reaction is given by equation 5. This process is accelerated, especially through high hypochlorite concentrations, UV radiation and heat (STRÄHLE 1999, GABRIO et al. 2004). Additionally, even during the electrochemical production of the solution, the reactions taking place through application of heat can produce a chlorate content of 2 to 8% (DYGUTSCH and KRAMER 2012).



The water to be disinfected can be different according to its proposed use and its chemical parameters. Thus, circulating nutritional solutions for hydroponic vegetable crops such as tomatoes or cucumbers, with 18 mg ammonia per litre (SONNEVELD and STRAVER 1988) contains a much higher concentration of plant nutrients compared to drinking water with < 0.05 mg ammonia per litre (BWB 2015). Additionally, the nitrogen fertiliser ammonia (NH_4^+) increases the chlorine loss in the water and oxidises with hypochlorous acid (HClO) in several partial steps, at first to chloramine (bound chlorine) with reduced antimicrobial efficacy (US NATIONAL RESEARCH COUNCIL 1987), then to nitrite and finally further to nitrate (NO_3^-) (BRYANT et al. 1992) which is a nitrogen source readily available to plants. Hereby, 1 mg of ammonia nitrogen ($\text{NH}_4\text{-N}$) binds around 10 mg of hypochlorous acid measurable as free chlorine (WOHANKA et al. 2015). The overall reaction is given by equation 6.



Focus of this paper is the procedural adaptation of the electrolysis technique, up until now mainly applied for disinfection of drinking and pool water, for use as disinfectant in irrigation water (esp. in closed irrigation systems). As a source of adjustment recommendations was investigated, on the basis of a brine electrolysis plant (single chamber system), how much problematical chlorate is produced during the production procedure for the disinfectant and how its proportion changes during storage under warm greenhouse conditions (summer season). Investigated additionally was the effect the plant fertiliser ammonia has on average disinfectant requirement, or chlorate input into the irrigation water.

Materials and methods

Featured in the investigation is a single chamber brine electrolysis plant (nt-BlueBox mini nt-CLE, newtec Umwelttechnik GmbH, Berlin, Germany) used for on-site disinfectant solution production in the experimental greenhouse of the Humboldt University at Campus Berlin Dahlem.

Brine electrolysis

During the electrochemical procedure for production of the disinfectant solution in an electrolysis reactor (Figure 1, C: single chamber system without membrane) a 3.5 % brine solution (sole) was subjected to a redox reaction for a 15 minute period through application of an electrical direct current ($\approx 9 \text{ A}$) at a low voltage range ($\approx 13 \text{ V}$). The brine solution (Figure 1, B) contained potassium chloride ($\text{KCl} \geq 99,5 \%$, p.a., ACS, ISO, Carl Roth GmbH + Co. KG, Karlsruhe, Germany). Because this was produced from the domestic water supply ($\approx 16 \text{ }^\circ\text{dH}$) the plant is equipped with a water softener (Figure 1, A) to avoid lime deposition on the titanium coated electrodes. Disinfectant solution was stored in a reserve tank (Figure 1, D) with content volume sensor. During withdrawal of the disinfectant solution (e.g. through a dosage system) this regulated the automatic refilling of the tank up to a predetermined volume. The functioning of the brine electrolysis system was based on two patents belonging to the plant constructor (GAO et al. 1997, GAO 2010).

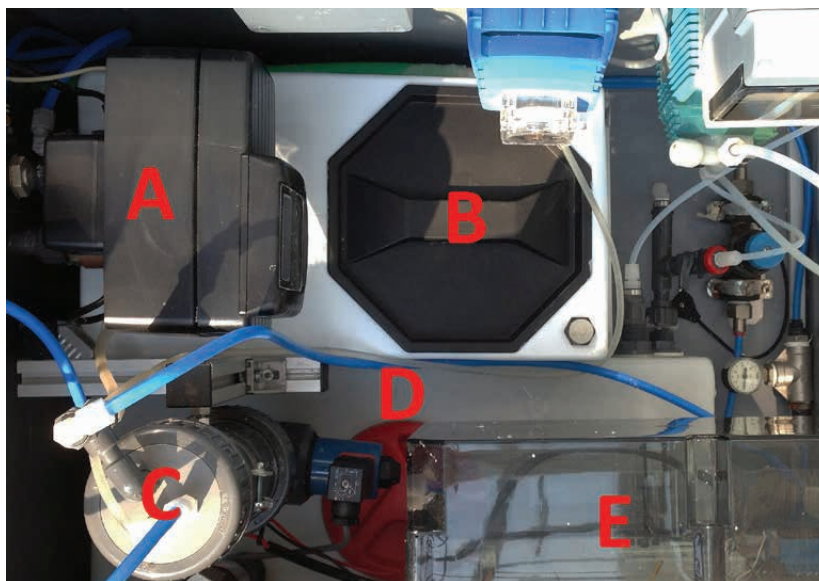


Figure 1: Brine electrolysis plant equipped with water softener (A), sole storage tank (B), electrolysis reactor (C), disinfectant storage tank (D) and control module (E) (Photo: I. Schuch)

Analysis of chlorate formation

To determine the influence of the length of time in storage on the chlorine and chlorate content of the disinfectant solution under warm greenhouse conditions (summer season), this was stored after electrolytically production for four weeks (August 2014) in the greenhouse. Hereby, it was assumed that the average day temperature of the disinfectant solution was the same as the daily average air temperature recorded at five-minute intervals within the greenhouse. Supplementing this, the set points (day/night) of the heating temperature were 17/20 °C and ventilation air temperature was 24/24 °C. The temperature recording in the greenhouse took place at 2 m height with a radiation-protected and ventilated climate recorder (P-TF-30, Positronik, Au in der Hallertau, Germany). The determination of free chlorine (Cl_2) in the disinfectant solution took place at the beginning ($n = 4$, double sample analysis), as well as at the end of storage via DPD method (CLESCERL et al. 1999) and photometer (Pocket Colorimeter II, Hach Lange GmbH, Düsseldorf/Berlin, Germany). Similarly, chlorate recording was via QuPpe method (ANASTASSIADES et al. 2013) and liquid chromatography coupled to mass spectrometry (1290 Infinity LC und 6460 Triple Quadrupole MS/MS, Agilent Technologies GmbH, Waldbronn, Germany).

Analysis of chlorine loss

In order to quantify the influence of ammonia on the chlorine loss in the irrigation water, a nutrient solution with an ammonia content of 10 mg/l (± 0.5 mg/l) and a pH of 6 was produced ($n = 2$). For this purpose, drinking water and rainwater (50 %/50 %) was dunged with a basic solution for hydroponic crops (SONNEVELD AND STRAVER 1988) and acidified with phosphoric acid (3 % H_3PO_4). Hereby, ammonia content was recorded via Nessler method (HANNA INSTRUMENTS INC. 2010) as well as photometer (HI 96733, Hanna Instruments GmbH, Vöhringen, Germany) and pH measurement via multi-parameter meter (HI 9811, Hanna Instruments GmbH, Vöhringen, Germany). In conclusion, the disinfectant solution was added to the nutrient solution in concentration steps of 5, 10, 50, 75 and 100 mg free

chlorine/l, so that after 10 minutes reaction time, the remaining free chlorine could be recorded using the DPD method (CLESERL et al. 1999) and photometer (Pocket Colorimeter II, Hach Lange GmbH, Düsseldorf/Berlin, Germany). Similarly, comparisons were made with drinking water and an ammonia content of < 0.05 mg/l (BWB 2015). All recording featured double sample analysis.

Results and discussion

The example of single chamber electrolysis plant used produced a disinfectant solution with an average free chlorine concentration of 4872 mg/l (± 612 mg/l) and 197 mg/l (± 154 mg/l) of chlorate (Figure 2), representing a limited chlorate proportion of around 4 % (based on the free chlorine). Hereby, the chlorate formation was possibly attributable to the endothermic production process within the electrolysis reactor (STRÄHLE 1999, GABRIO et al. 2004). In comparison, similar plants show a chlorate proportion of 2 to 8 % (DYGUTSCH AND KRAMER 2012). After four weeks storage (summer season), the disinfectant solution in the greenhouse with a storage temperature averaging 22.4 °C in total, ranging from 20.5 to 25.6 °C daily average (minimum 17 °C nights and 34.6 °C days according to the fundamental single measurements), showed a greater temperature divergence ($\Delta T = 17.6$ K). The content of free chlorine had decreased by almost 50 % to 2500 mg/l (± 375 mg/l). At the same time, the chlorate content increased to 1412 mg/l (± 212 mg/l) (Figure 2). This represented a high chlorate proportion of around 56 % (based on the free chlorine). This result should be checked for reproducibility and should also be supplemented by results from experiments with storage at lower temperature fluctuations in the greenhouse (e. g. winter season), as well as by any evidence of further by-products from the procedure such as chlorite (ClO_2^-) and perchlorate (ClO_4^-).

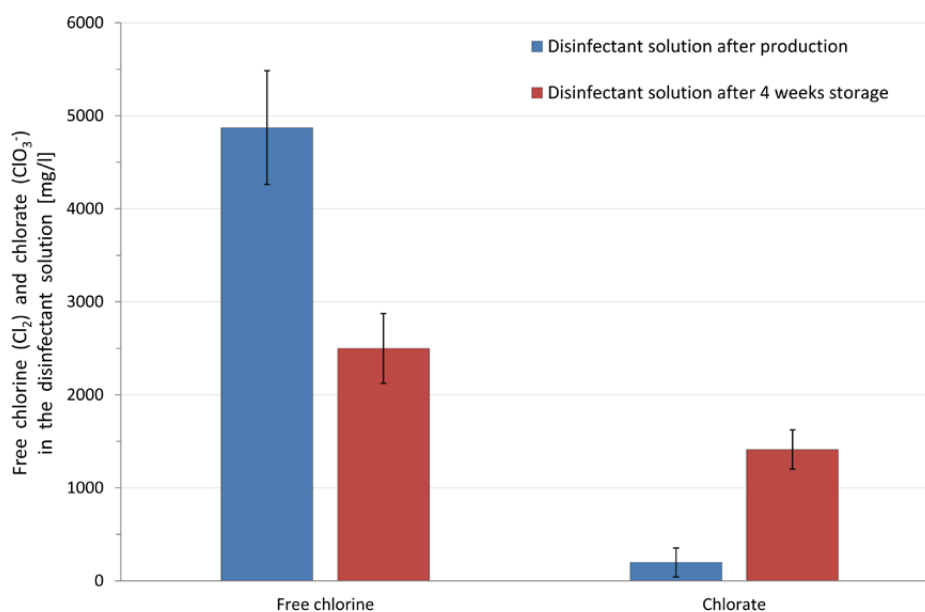


Figure 2: Influence of the length of time in storage on the free chlorine and chlorate content of the electrolytically produced disinfectant solution under warm greenhouse conditions (summer season, $T_{\text{max}} = 34.6$ °C); Mean values ($n = 4$) with standard deviation (\pm) of the free chlorine and chlorate content in the disinfectant solution after production (blue bars) and after 4 weeks storage (red bars)

According to the recordings, using fresh disinfectant solution and an antimicrobial dosage of 1 mg free chlorine/l (CLARK and SMAJSTRLA 1992) enables an arithmetical result of around 0.04 mg chlorate/l (± 0.03 mg/l) in treated irrigation water to be reached. Hereby, increasing or decreasing the disinfectant dose would influence the chlorate input to the same extent. On the other hand, should the disinfectant solution be stored for four weeks in the greenhouse (summer season) whereby the typical warm season temperature fluctuations occur within the greenhouse between day and night, the chlorate input would rise, on an arithmetic basis using the given disinfectant dosage, to 0.56 mg/l (± 0.08 mg/l).

The measured chlorine loss indicates a marked difference between the water with/without ammonia. Thus, as opposed to drinking water (< 0.05 mg NH_4^+ /l), in which the free chlorine was quite near the dosage applied (Figure 3, blue line), in irrigation water (10 ± 0.5 mg NH_4^+ /l) a rise in free chlorine did not take place until the chlorine loss limit was reached (Figure 3, red dashed line). In this respect, it is possible to deduce with the help of a regression line (Figure 3, red dotted line) supported by the parallelism for recording without ammonia, a loss ratio of ammonia to chlorine of around 1 : 7 (after 10 min reaction time). This is under the result of 1 : 10 for ammonia-nitrogen (WOHANKA et al. 2015) (without given reaction time). Hereby, we can assume that the period of time between chlorine addition and sampling influenced the measured value. Thus, chlorine loss measurements need to be supplemented with further reaction times (e.g. after 5, 20 and 30 min).

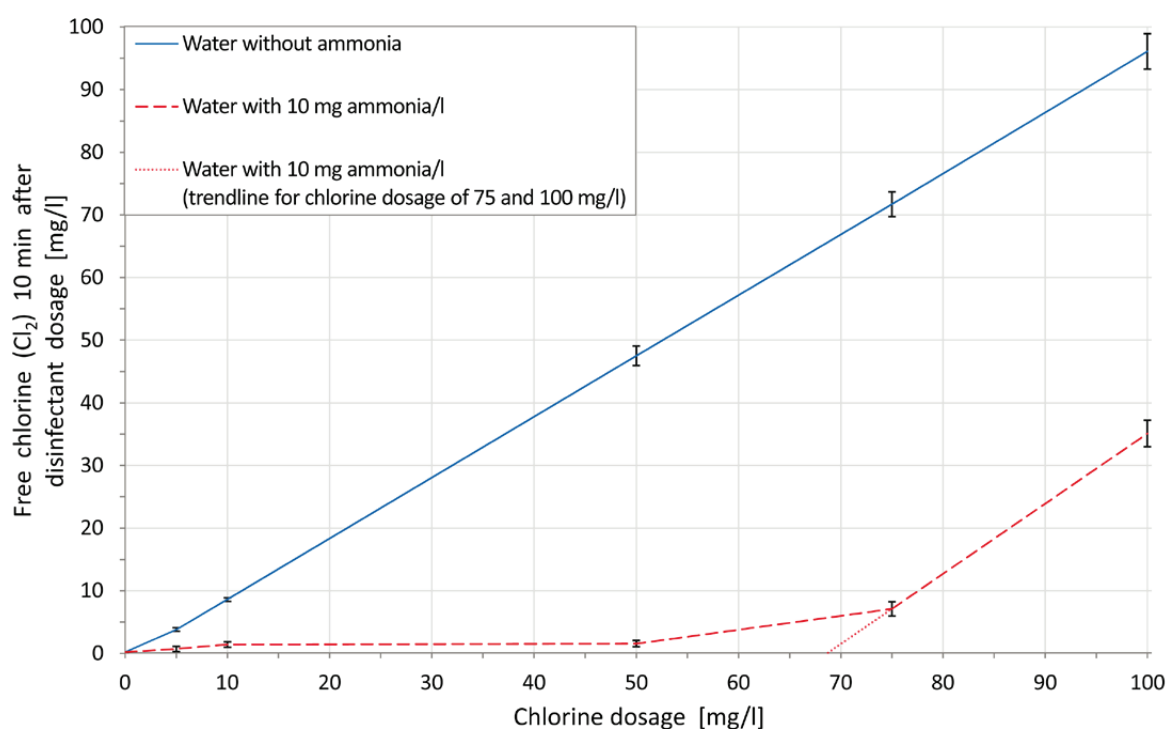


Figure 3: Chlorine loss, 10 min after dosage, in drinking water without ammonia (blue line) and irrigation water with ammonia (red dashed line); Calculation of the chlorine loss limit (red dotted line) for chlorine dosage of 75 and 100 mg/l; Mean values ($n = 2$) with standard deviation (\pm) of free chlorine

Chloramine (bound chlorine) is produced during reactions between chlorine and ammonia. In comparison with free chlorine this, however, has reduced antimicrobial efficacy (US NATIONAL RESEARCH COUNCIL 1987), although a longer half-life period whereby the chloramine decomposition under UV radiation reached a maximum of 0.2 mg/l h (WHITE 1992). Over and above this, the readily available nitrogen source for plants, nitrate (NO_3^-), can be produced from chloramine under nitrification conditions (BRYANT et al. 1992). In this respect, it can be assumed that continuous chlorination brings the fundamental microbiological degradation process (nitrification) to a standstill more rapidly than a discontinuous/sporadic disinfection of irrigation water.

With regard to the investigated loss ratio of ammonia to chlorine under nutrient recommendations for tomato/cucumber irrigation of 18 mg NH_4^+ /l (SONNEVELD and STRAVER 1988) and a reference value in irrigation water of 1 mg free chlorine/l, an increased requirement for disinfectant solution to the factor of 126 (based on a chlorine loss ratio 1 : 7) is to be expected. The chlorate input in irrigation water would be influenced to the same extent, whereby not a lot is known about chlorate uptake by plants. Official food surveillance shows, however, that relatively high chlorate contents, e.g. in tomatoes (0.2 mg/kg) and carrots (0.3 mg/kg), can occur (KAUFMANN-HORLACHER 2014). Hereby, it has been proved in the meantime that the electrolytic disinfection of irrigation water can lead to chlorate absorption by vegetables. In the corresponding study (DANNEHL et al. 2015A), tomato plants were continuously irrigated within a closed circuit on plastic channels (NFT) with a disinfectant solution produced on site and dosed discontinuously over a period of three months (1/week for 90 min) in concentrations of 1 mg (variant DI) or 2 mg free chlorine/l (variant DII) in fertiliser-containing irrigation water (contents including ammonia). In the result, no yield reduction was determined while antimicrobial efficacy was found to be improved (DANNEHL et al. 2015B). However, the chloride content in water rose following discontinuous introduction of disinfectant by 14 mg/l (DI) on average, or 21 mg/l (DII) (DANNEHL et al. 2015A), whereby the crop growing recommendation for hydroponic tomatoes of < 532 mg chloride/l (SONNEVELD and STRAVER 1988) was undersupplied, even after three months without water change. All the same, the tomatoes also tolerated, compared to other hydroponic crops (e.g. salad crops), higher chloride content in the irrigation water (SONNEVELD and STRAVER 1988). Furthermore, under the discontinuously applied disinfectant regime (as mentioned), chlorate content in the tomatoes rose to 0.22 mg/kg (DI) or 0.25 mg/kg (DII) (DANNEHL et al. 2015A) from which one can deduce, based on current knowledge, a toxicologically safe daily consumption of up to 10 kg tomatoes (based on a 70 kg bodyweight and in the absence of any other chlorate consumption) (EFSA 2015).

Conclusions

In principle, brine electrolysis systems are suitable for disinfection of irrigation water, when procedural techniques, storage conditions, dosage management and actual applications are coordinated. Required in the future to help in this respect are case studies with different crops, cropping systems, dosage scenarios and phytopathogens. The basic requirement for application in crop production is, however, that amounts of disinfection by-products (esp. chloride, chlorite, chlorate, perchlorate) in water, substrates and plants (esp. vegetable plants) do not exceed toxicologically acceptable limits.

During brine electrolysis, a certain amount of chlorate is produced by the electrochemical process whenever heat is applied. Therefore techniques should be tested for conducting any produced heat away from the procedure through e.g. fan, water-cooling or Peltier cooler.

A further procedure concerns storage of the disinfectant solution (esp. under warm conditions). With increasing storage time, the effective disinfectant proportion of the free chlorine decreases and the chlorate proportion increases. In the context of irrigation water disinfection it appears, that the on-site production of a fresh disinfectant solution according to short term requirement (e. g. one day's supply) is advantageous compared to storing industrially produced large volumes delivered in barrels. Additionally recommended is a cooling system for the disinfectant storage tank.

A further measure for minimising chlorate in water, or in the plants that are cultivated with the help of the water, concerns dosage management. Thus, a discontinuous application of disinfectant (e.g. 1 to 2 times per week) with higher doses of chlorine could lead to less chlorate input compared with continuous chlorine application at a lower dosage. Comparative tests on plant tolerance and infection reduction have yet to be conducted, however.

Regarding dosage of electrolytically produced disinfectant solution, it is shown that the plant fertiliser ammonia rapidly binds the free chlorine and thus markedly increases usage of disinfectant or chlorate input up to the chlorine loss limit. Thus, it is recommended that dosage to according to solution concentration with this concentration level controlled by chlorine sensor so that alterations of chemical processes in water (chlorine loss) can be accounted for when adjusting dosage. Additionally, as little ammonia fertiliser as possible under plant development requirements should be applied with substitution where required with other plant nutrient substances (e. g. nitrate).

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