# Sodium Chlorite Treatments in Irrigation Water Are a Significant Source of Chlorate and Perchlorate in Hop Cones

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**Abstract. Chlorine-based disinfectants used for cleaning and maintaining hop (*Humulus lupulus*) irrigation systems may contribute to chlorate ($ClO_3^-$) and perchlorate ($ClO_4^-$) residues in hop cones, thus raising regulatory concerns in various export markets. Because of their associated risk of disrupting iodine uptake in the thyroid gland, $ClO_3^-$ and $ClO_4^-$ are oxyanions designated as contaminants in vulnerable populations. This study evaluated their impact on hop cone contamination. A 2-year field study (2023 and 2024) at Washington State University examined the following four drip irrigation cleaner treatments: 1) continuous hydrogen peroxide + peroxyacetic acid; 2) continuous 2 ppm sodium chlorite; 3) continuous 2 ppm sodium chlorite with two late-season 25-ppm shock treatments; and 4) two late-season 25-ppm sodium chlorite shock treatments. A residue analysis was performed using liquid chromatography-tandem mass spectrometry. Continuous sodium chlorite application led to $ClO_3^-$ residues up to 47 mg·kg$^{-1}$, which far exceeded the European Union limit (0.05 mg·kg$^{-1}$). The $ClO_4^-$ levels peaked at 0.25 mg·kg$^{-1}$, which was below the 0.75 mg·kg$^{-1}$ limit. Hydrogen peroxide-treated hops still contained $ClO_3^-$, suggesting other contamination sources. A soil analysis showed $ClO_4^-$ accumulation but no detectable $ClO_3^-$. Interestingly, the use of sodium chlorite resulted in higher hop cone yield. Chlorine-based cleaners significantly increase $ClO_3^-$ and $ClO_4^-$ residues in hops. Alternative treatments reduced but did not eliminate $ClO_3^-$ and $ClO_4^-$ contamination in hop cones.**

Hops (*Humulus lupulus*) are a vital component of the brewing industry because they provide unique flavor, aroma, and stability in beer (Lafontaine et al. 2022; Turner et al. 2011). Globally, hop is primarily cultivated in the United States and Europe. The United States accounted for approximately 44% of global production in 2022, followed by Germany (32%), China (6%), and the Czech Republic (4%) (European Commission DG AGRIE.2 2023; US Department of Agriculture National Agriculture Statistics Services 2023). In the United States, most hop production occurs in the Pacific Northwest (PNW) states of Washington (74%), Idaho (16%), and Oregon (13%) (Hop Growers of America 2023). Unlike Europe, nearly all US hop production relies on irrigation during dry summer conditions because precipitation predominantly occurs during the dormant season (Morton et al. 2017).

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The data supporting this study's findings are available on GitHub at https://github.com/Gonzalezhopresearch/Hop-Chlorate-Study. Additionally, data can be obtained upon reasonable request by contacting the corresponding author.
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A drip irrigation system (DIS) is widely used in hop cultivation, particularly in Washington, where more than 96% of hop producers rely on this method to deliver the approximately 762 mm of water required during the growing season (Evans 2003; Washington State Department of Agriculture 2024). While DIS ensures precise water application and reduces crop health issues, a primary drawback is emitter clogging caused by the small orifice diameter (0.5–1.2 mm) (Zhou et al. 2017). Emitters can become blocked by suspended particles, chemical precipitation, and biofilm formation. Biofilm, an extracellular polymeric substance (EPS) formed by microbial growth, poses significant challenges. The DIS biofilm accumulation impacts irrigation uniformity, reduces service life, and increases operational costs (Wang et al. 2022; Zhou et al. 2017).

Chlorine-based products are widely used to reduce microbial populations in fresh-cut produce (Praeger et al. 2018) and mitigate biofilm formation in DIS by reducing microbial contamination (Waters et al. 2014). Historically, the PNW hop industry has relied on these products to maintain irrigation system efficiency (Hop Growers of Washington 2025). While effective, the extensive use of chlorine-based cleaners has raised concerns about the accumulation of chemical residues such as chlorate ($ClO_3^-$) and perchlorate ($ClO_4^-$) in agricultural products, including hop cones (Debelder 2020; European Food Safety Authority Panel on Contaminants in the Food Chain 2015). These residues can form through chemical reactions involving chlorine-based products such as sodium chlorite and oxidizing agents present in soils (Sandin et al. 2013; Srinivasan and Viraraghavan 2009). Once formed, soil particles are weakly adsorbed by these highly soluble compounds; therefore, they are quickly mobilized into water systems (Acevedo-Barrios et al. 2024; Dasgupta et al. 2006; Hutchison et al. 2013). The accumulation of $ClO_3^-$ and $ClO_4^-$ in crops poses health risks by disrupting iodine uptake in the thyroid gland, particularly affecting vulnerable populations such as infants, children, and pregnant women (European Food Safety Authority Panel on Contaminants in the Food Chain 2015).

It has been determined that $ClO_4^-$ is substantially more potent in reducing iodine uptake by the thyroid than $ClO_3^-$. Based on in vitro studies of the effects of various anions on the sodium/iodine symporter (NIS), $ClO_4^-$ was significantly more potent than $ClO_3^-$ for reducing iodine uptake in thyroid cells. The $IC_{50}$ values, which represent the concentration required to inhibit 50% of iodine uptake, were 0.43 μM for $ClO_4^-$ and 131 μM for $ClO_3^-$ in COS NIS-6 cells, a human NIS-expressing cell line. These findings indicate that $ClO_4^-$ is approximately 305-times more potent than $ClO_3^-$ as an inhibitor of NIS (Van Sande et al. 2003). Animal studies further support these observations, with research indicating that $ClO_4^-$ is 10-times more potent than $ClO_3^-$ at reducing thyroid function (European Food Safety Authority Panel on Contaminants in the Food Chain 2015). The higher potency of $ClO_4^-$ was recognized by lawmakers in the European Union (EU) when they established tolerable daily intake doses (TDI) for $ClO_3^-$ and $ClO_4^-$ . The TDI for $ClO_3^-$ was set to 3 $ug \cdot kg^{-1}$ and 0.3 $ug \cdot kg^{-1}$ for $ClO_4^-$, acknowledging the 10-times higher potency of $ClO_4^-$ in their decision (European Food Safety Authority Panel on Contaminants in the Food Chain 2015).

Studies have documented $ClO_3^-$ and $ClO_4^-$ residues in various foods often exceeding regulatory thresholds. For instance, high levels of $ClO_4^-$ were detected in South Korean dairy milk and infant formula in 2009 (Her et al. 2010). In Europe, $ClO_3^-$ residues in plant-based foods frequently exceeded the EU maximum residue level (MRL) of 0.01 $mg \cdot kg^{-1}$, with leafy vegetables and processed products showing the highest contamination (Kaufmann-Horlacher et al. 2013). More recent studies in the United Kingdom and Ireland reported $ClO_3^-$ residues in melons, peas, pineapples, and dairy products, highlighting the global extent of the issue (Twomey et al. 2023).

To address growing concerns over elevated levels of $ClO_3^-$ and $ClO_4^-$ in imported foods, the EU enacted Regulation 2020/749, which established a default MRL of 0.05 $mg \cdot kg^{-1}$ for $ClO_3^-$ in imported dried tea, herbal infusions, and hops. Additionally, Regulation (EU) 2020/685 set an MRL of 0.75 $mg \cdot kg^{-1}$ for $ClO_4^-$ in imported hops (European Commission 2020a, 2020b). These stringent MRLs established by the EU have introduced significant trade challenges, particularly for the US hop industry, which exports nearly one-third of its hop production to the EU. In 2022 alone, US hop exports to the EU totaled nearly $211 million, while imports from the EU amounted to $36.8 million (US Department of Agriculture Foreign Agricultural Service 2022).

Recent findings underscore the urgency of addressing $ClO_3^-$ and $ClO_4^-$ contamination in hops. For instance, German hop samples tested in 2023 revealed $ClO_3^-$ levels of 0.27 $mg \cdot kg^{-1}$ attributable to chlorine-based DIS cleaning products (EURL-SRM and CVUA Stuttgart 2023). Although PNW hop growers have transitioned to alternative cleaning agents, such as hydrogen peroxide and peroxyacetic acid (Hop Growers of Washington 2025), the legacy of chlorine-based treatments remains underexplored. Importantly, no studies have investigated the direct impact of chlorine-based drip line cleaning products on $ClO_3^-$ and $ClO_4^-$ residues in hop cones.

This study aimed to evaluate the influence of chlorine-based drip line cleaning products on $ClO_3^-$ and $ClO_4^-$ levels in hop cones. Beyond simply identifying contamination levels, this research aimed to provide critical insights into the persistence of these residues and the efficacy of alternative cleaning strategies. By examining the relationship between irrigation treatment and residue accumulation, the study sought to inform industry best practices and facilitate the sustainable production of high-quality hops.

## Materials and Methods

**Experimental site.** The experiment was conducted in 2023 and 2024 at the Washington State University Irrigated Agriculture Research and Extension Center near Prosser, WA, USA (lat. 46.2568°, long. 119.7326°, elevation 269 m). The hopyard was established with cultivar Cascade in 2009 following standard commercial practices, including installing a high trellis system with a height of approximately 5.49 m and 4.6 m between rows. The research site comprised 12 rows that could host three research plots of seven plants spaced 1.7 m apart. The same research site was used during both seasons. In 2024, the plots were laid out to minimize overlaying plots from the previous year (Fig. 1). The crop was irrigated using drip irrigation with an output of 2.01 L per hour and emitters were spaced 61 cm apart. The top 30 cm of soil at the experiment site was silt loam composed of 40% sand, 52% silt, and 8% clay, with a pH of 6.7 across the field.

The experiment site experiences a semiarid climate characterized by hot, dry summers and cool, moist winters. The climate of the area is premium for hop production because it receives approximately 2873 annual growing degrees (base of 45 °F). The region receives an average annual precipitation of approximately 203 mm, mainly between October and March. Summers (April through September) are typically warm, with average high temperatures reaching approximately 32.8 °C, but it can be as high as 45.6 °C in June and July. Winters are cooler, with average lows of approximately −3.8 °C (NOAA National Weather Service 2024). Weather parameters were collected using Washington State University's AgWeatherNet (https://weather.wsu.edu) and a weather station 600 m west of the research site.

**Drip line cleaner treatments.** The impact of chlorine-based drip line cleaners was assessed over 2 years at two adjacent research plots. Four treatment regimens were applied through the drip irrigation system using injection pumps at a ratio of 200:1. Each plot was equipped with its own drip tubing, while the rest of the tubing leading up to a mainline was blank tubing. The blank tubes from each treatment were connected to a mainline fed by an injection pump. The control treatment used Jet-Ag (US Environmental Protection Agency registration no. 81803-6), which is a hydrogen peroxide (26.5%) and peroxyacetic acid (PAA; 4.9%) formulation commonly used for irrigation system maintenance. The three chlorine-based treatments used 15% sodium chlorite (EnviroChlorite 15; US Environmental Protection Agency reg. no. 63838-21), which generates chlorine dioxide under acidic conditions and can degrade into $ClO_3^-$ in light and heat. The treatments were as follows:

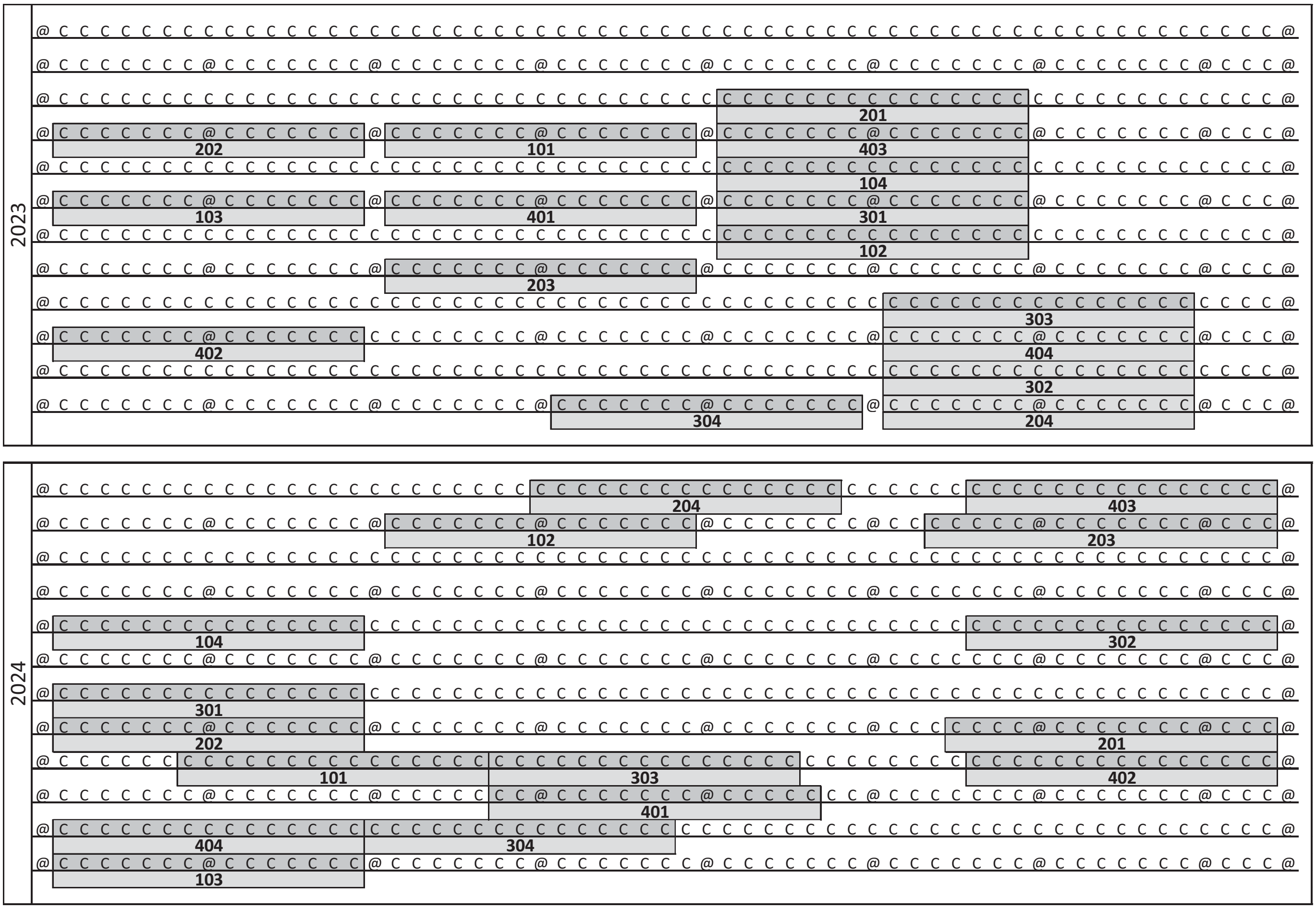


Fig. 1. Research ploy layout during the 2023 and 2024 hop growing seasons. "C" represents the plants and "@" represents the pole locations. The bolded values represent the treatment (first value) and the replication (third value).

- T1 (control – grower standard): Hydrogen peroxide (10.4 ppm) + PAA (2 ppm) applied at every irrigation event.
- T2 (continuous sodium chlorite): Sodium chlorite (2 ppm) was applied at every irrigation event, simulating past biofilm control methods.
- T3 (sodium chlorite + shock treatment): Sodium chlorite (2 ppm) was applied at every irrigation event, with two 15-min shock treatments of 25 ppm sodium chlorite between July and August.
- T4 (shock treatment only): Two 15-min shock treatments of 25 ppm sodium chlorite applied in July and August.

To investigate whether there was an carryover effect in the levels of $ClO_3^-$ and $ClO_4^-$ in the hop cones and soil after 1 year, research plots that were treated in 2023 were harvested, pelletized, and evaluated for $ClO_3^-$ and $ClO_4^-$ in 2024. The 2023-treated plots were irrigated with just water throughout the 2024 growing season.

**Agri-management and sampling.** Soil moisture was maintained near the soil's field capacity throughout the season. Fertilization was managed based on soil test recommendations, ensuring appropriate nutrient availability throughout the growing season. Pest and disease management adhered to integrated pest management principles and incorporating regular field scouting and applying control measures as necessary. Harvest occurred when cones reached optimal maturity, as indicated by a cone dry matter content between 24% and 26% and a hop storage index below 0.30, ensuring peak quality and aroma potential. In 2023, the hop cones were harvested on 30 and 31 Aug. The hop cones of hop plants treated in 2024 were harvested on 4 Sep, and the hop cones of hop plants treated in 2023 were harvested on 7 Sep. Harvest consisted of cutting down the hop bines into a 6.10-m trailer, which was transported to the picking facility; then, the cones were separated from the bine using a Wolf 170 picker (Wolf Harvesters, Lublin, Poland). Between plots, the picker was thoroughly cleaned to minimize cross-contamination. The hop cones were dried the same day they were harvested using a forced-air batch kiln and allowed to dry until the water content read between 8% to 10%. The dried cones were stored at $-18\,°C$ until they were pelletized.

In Sep 2023 and 2024, a 2-kg subsample of hop cones was collected from the entire population. Using liquid nitrogen, the hop cones were pelletized under controlled conditions to maintain the pelletizer's internal head temperature below $55\,°C$. The pelletized cones were vacuum-sealed in mylar bags, flushed with nitrogen, and stored at $-18\,°C$ until analysis. Samples from the 2023 harvest were submitted for analysis on 2 Feb 2024, while those from the 2024 harvest were submitted on 15 Sep 2024. A total of 300 g of pelletized hop cones per experimental unit were shipped via next-day air for analysis. The $ClO_3^-$ and $ClO_4^-$ analyses were performed on 4 Mar 2024 for the 2023-treated samples, and on 19 Nov 2024 for the 2024-treated samples.

Soil samples were collected from the top 15 cm of the soil profile in each plot by extracting six cores that were then thoroughly mixed to create a composite sample. A 50-g subsample was placed in a polyethylene bag and stored at 4 °C until the analysis could be completed. Soil samples were collected on 31 Oct 2024, nearly 2 months after harvest. The soil samples were analyzed on 9 Jan 2025.

During Jun 2023 and 2024 and Jul 2023 and 2024 of the growing seasons, three 30-mL irrigation water samples were collected from each plot using 50-mL polypropylene tubes. The samples were stored at −25 °C to preserve integrity until analysis. The three aliquots from each season were pooled into a single composite sample, resulting in one representative sample per season. All samples were shipped via overnight courier on 3 Jan for analysis.

**Determining combined and free chlorine concentrations in irrigation water.** The irrigation water was indirectly evaluated for the presence of chlorine-based products by measuring free chlorine and total chlorine in each treatment at the time of application. The assessment used an eXact® Micro 20 with Bluetooth® Photometer (Industrial Test Systems, Rock Hill, SC, USA) in conjunction with DPD-1 and DPD-4 reagents. This method aligns with the 4500-Cl G DPD colorimetric method described in the 20th edition of *Standard Methods for the Examination of Water and Wastewater* (Eaton 1998). Free chlorine measures the active components (HOCl and $OCl^-$) in water that are available for disinfection, and total chlorine is the sum of free and combined chlorine, which measures chlorine that reacted with organic or nitrogen compounds to form chloramines, providing a more complete picture of chlorine levels (US National Research Council Safe Drinking Water Committee 1980).

Although sodium chlorite, the chlorine-based product used in this study, breaks down into chlorine dioxide, there is an overlap between free chlorine and chlorine dioxide, which is detected by the instrument using the DPD colorimetric test (Gordon et al. 1988; Palin 1975; Tkacova and Bozikova 2014). The handheld unit provided an accuracy of ±3 ppm ±5% of the free and total chlorine readings at 525 nm and 638 nm at a water temperature of 25 °C. The device measured free chlorine concentrations ranging from 0.01 to 6.5 ppm and total chlorine from 1 to 200 ppm with optimal accuracy within this range. Following the manufacturer's guidelines, free and total chlorine levels were measured in the irrigation water while the treatments were applied in July and August before harvest.

**Determining the chlorate and perchlorate concentrations in pelletized hop cones, irrigation water, and soil.** The pelletized hop cones and irrigation water were evaluated for $ClO_3^-$ and $ClO_4^-$ by Columbia Laboratories (ISO/IEC 17025 accredited), located in Portland, OR, USA, and soil samples were evaluated by the Smoke, Wine, and Grape Chemistry Laboratory at Oregon State University in Corvallis, OR, USA. Both laboratories used the quick method for analyzing high polar pesticides (EURL-SRM version 7) to assess $ClO_3^-$ and $ClO_4^-$ in the various matrices (Anastassiades et al. 2012). The quick method for analyzing high polar pesticides method described in the EURL-SRM guidelines is a highly efficient and robust procedure for analyzing polar pesticides, such as $ClO_3^-$ and $ClO_4^-$, in plant-origin matrices, including hop cones. The method involves cryogenic milling of 2 g of dried hop cones to ensure homogeneity and analyte accessibility. This is followed by extraction using 10 mL of acidified methanol enhanced with isotopically labeled internal standards to account for matrix effects and analyte recovery. After extraction, the samples were subjected to high-speed cryogenic centrifugation to separate the analytes from interfering components. In cases of high oil content, the samples can be cleaned using dSPE with C18 sorbent to remove lipids and proteins. The processed extracts are filtered and subsequently analyzed using liquid chromatography-tandem mass spectrometry with specific ion chromatography techniques to achieve high sensitivity and precision when detecting trace levels of $ClO_3^-$ and $ClO_4^-$. The only difference when assessing $ClO_3^-$ and $ClO_4^-$ in the hops and soil matrix was that in soil, 20 g was used while still using 10 mL of water for the extraction.

**Experiment design and statistical analysis.** The experiment was conducted using a completely randomized design, with four replicates randomly assigned throughout the research site. Outlier detection was performed using the interquartile range (IQR) method and identifying one outlier in the hop cone $ClO_4^-$ dataset. The outlier was detected after segmenting the values into their perspective treatment groups and explicitly observed in T1 (Fig. 2). The outlier significantly inflated the group's mean and standard deviation, potentially skewing the analysis and leading to erroneous inferences when comparing the means. To mitigate this, the outlier value was recalculated using automated data imputation, which uses singular value decomposition to automatically select the optimal dimension for low-rank approximation based on the data. Normality was assessed using the Shapiro-Wilk test. Only the $ClO_4^-$ analysis of hop cones treated with drip line cleaners in 2024 was normally distributed among the datasets. However, this dataset failed to meet the assumption of equal variances across groups, as determined by Levene's test. Because the dataset violated both the assumptions of normality and equal variances, nonparametric methods were applied for statistical analysis, specifically, the Kruskal-Wallis test. The Wilcoxon method, a nonparametric version of the Student's method, was used to compare the treatment means.

A two-way analysis of variance (ANOVA) of the dry cone yield dataset was conducted because it complied with each of the required assumptions. The statistical analysis was conducted using JMP (version 17; SAS Institute Inc., Cary, NC, USA), and the Figures and Tables were crafted using SigmaPlot (version 15; Grafiti, LLC, Palo Alto, CA, USA) and Excel (Microsoft, Redmond, WA, USA), respectively.

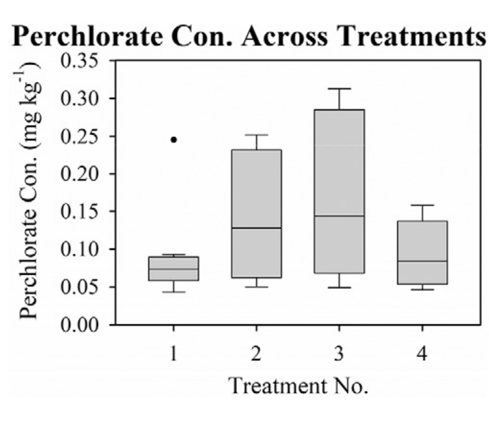


Fig. 2. Box plot of hop cone perchlorate concentration displaying outliers by treatment number.

## Results

**Weather at the research site.** The weather conditions at the research site during the 2023 and 2024 hop-growing seasons were generally similar, with only minor differences. The average annual temperature varied by approximately 4%, with means of 16.6 °C in 2023 and 16.0 °C in 2024. However, temperature extremes were more frequent in 2024, with 26 d exceeding 35 °C and 4 d surpassing 40 °C, compared with 20 d over 35 °C and only 2 d above 40 °C in 2023. Precipitation also differed between the 2 years. During the hop-growing season, 2023 received 62.5 mm of rainfall, which was 20% more than the 50.3 mm recorded in 2024. Other weather parameters remained broadly consistent across both years (Table 1).

**Hop cone yield.** The 2-year dry hop cone yield was significantly influenced by the different drip line cleaner treatments ($P = 0.0064$) (Fig. 3A) but did not differ significantly between years ($P = 0.1564$) (Fig. 3B). Hop plants treated with two 15-min flush shock applications of 25 ppm sodium chlorite (T3 and T4) produced an average dry hop cone yield of 3622 $kg \cdot ha^{-1}$, which was 27% higher than the yield from plants treated with T1 and T2 (mean = 2546 $kg \cdot ha^{-1}$) (Fig. 3A). Notably, hop plants subjected to sodium chlorite treatments in both 2023 and 2024 had a mean yield of 3084 $kg \cdot ha^{-1}$, which was 37% greater than the yield from plants not treated with drip line cleaners in 2024 (mean = 1953 $kg \cdot ha^{-1}$) (Fig. 3C).

**Irrigation water chlorine, chlorate, and perchlorate analysis.** Free chlorine concentrations in the irrigation water were not detected during the application of hydrogen peroxide and PAA (T1) in the 2023 or 2024 season. This aligns with the absence of chlorine-based products in these treatments, as expected based on the methodology. Conversely, free chlorine was consistently detected during the application of sodium chlorite (T2 and T3) at a concentration of 2 ppm. The average free chlorine concentration in irrigation water for the 2023 season was 2.04 ppm; however, in 2024, the average was 1.67 ppm. The slight reduction observed in free chlorine levels in 2024 reflected the substantially lower values recorded in July. When the irrigation water was treated with a shock application of sodium chlorite at 25 ppm, the average total chlorine concentration was 28 ppm across all evaluations. This result is consistent with the expected output for treatment with 25 ppm sodium chlorite (Table 2). Furthermore, irrigation water samples collected during July and August in both years and analyzed in Jan 2025 showed no detectable levels of $ClO_3^-$ or $ClO_4^-$ (Limit of Quantification = 10.0 $\mu g \cdot kg^{-1}$), confirming that these compounds were not introduced into hop cones via irrigation water.

**Hop chlorate analysis.** In 2024, significant differences were found in the $ClO_3^-$ concentrations measured in pelletized hop cones harvested from plots subjected to the various drip line cleaner treatments ($P = 0.0053$). Hop plants subjected to the continuous application of sodium chlorite, T2 and T3 (mean = 44.64 $mg \cdot kg^{-1}$), produced hop cones with an estimated 300-times greater $ClO_3^-$ concentration than those subjected to hydrogen peroxide + PAA (T1; mean = 0.15 $mg \cdot kg^{-1}$). The additional two applications of 25 ppm sodium chlorite at the end of the season added to T3 did not significantly increase hop cone $ClO_3^-$ levels in both 2023 and 2024. The mean $ClO_3^-$ concentration of hop cones subjected to the continuous application of sodium chlorite, T2 and T3, was eight-times greater than those subjected to the two 25-ppm sodium chlorite flush treatments at the end of the season (T4; mean = 5.45 $mg \cdot kg^{-1}$). The $ClO_3^-$ concentration of hop cones subjected to the continuous application of hydrogen peroxide + PAA (T1; mean = 0.15 $mg \cdot kg^{-1}$) was nearly 37-times lower than those subjected to the two applications of 25 ppm sodium chlorite flush at the end of the season (T4; mean = 5.45 $mg \cdot kg^{-1}$) (Table 3).

Table 1. Average weather parameters measured during the 2023 and 2024 hops growing seasons.

| Year | Month | 1.5-m Avg air temperature (°C) | 1.5-m Max air temperature (°C) | 1.5-m Avg relative humidity (%) | Avg solar radiation ($W \cdot M^{-2}$) | Total precipitation (mm) | 2-m Avg wind speed (kph) | Wind direction |
|---|---|---|---|---|---|---|---|---|
| Monthly summary | | | | | | | | |
| 23 | 3 | 5.0 | 17.2 | 64.4 | 144.8 | 20.8 | 7.5 | W |
| 23 | 4 | 13.1 | 29.2 | 51.2 | 230.7 | 0.8 | 8.6 | W |
| 23 | 5 | 18.5 | 34.9 | 53.5 | 288.0 | 5.8 | 7.6 | W |
| 23 | 6 | 20.4 | 35.7 | 47.5 | 308.1 | 4.6 | 7.1 | W |
| 23 | 7 | 24.0 | 37.6 | 43.1 | 331.2 | 4.1 | 7.4 | NW |
| 23 | 8 | 22.5 | 40.2 | 53.1 | 237.4 | 10.2 | 7.2 | NW |
| 23 | 9 | 16.9 | 31.2 | 62.0 | 185.6 | 7.1 | 7.4 | NW/W |
| 23 | 10 | 10.8 | 26.7 | 70.9 | 121.4 | 9.1 | 6.3 | NW |
| 24 | 3 | 7.6 | 24.5 | 61.2 | 171.5 | 18.0 | 9.1 | W |
| 24 | 4 | 10.5 | 25.6 | 55.2 | 225.3 | 6.9 | 8.7 | W |
| 24 | 5 | 14.9 | 31.4 | 50.9 | 284.2 | 7.9 | 9.0 | W |
| 24 | 6 | 19.4 | 34.6 | 44.3 | 305.6 | 4.6 | 9.0 | W |
| 24 | 7 | 24.7 | 40.4 | 42.1 | 313.7 | 1.3 | 7.4 | W |
| 24 | 8 | 21.9 | 40.3 | 51.1 | 256.7 | 0.0 | 7.5 | NW |
| 24 | 9 | 18.9 | 36.7 | 55.8 | 194.8 | 0.0 | 7.1 | NW |
| 24 | 10 | 10.7 | 26.9 | 64.9 | 130.4 | 11.7 | 6.9 | W |
| Annual summary | | | | | | | | |
| 23 | | 16.7 | 40.2 | 56.1 | 230.8 | 62.5 | 7.3 | W |
| 24 | | 16.1 | 40.4 | 53.2 | 235.2 | 50.3 | 8.1 | W |

N = north; NW = northwest; W = west.

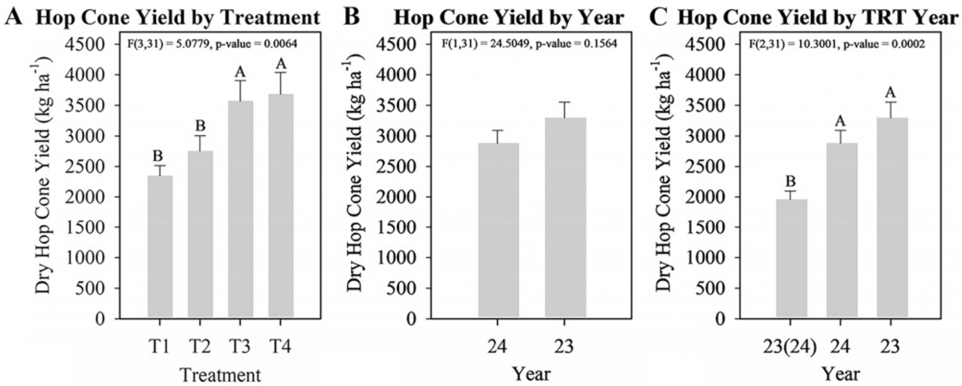


Fig. 3. Dry hop cone yield analysis among four drip line cleaner treatments across 2023 and 2024 and between the years. The drip line cleaner treatments were evaluated separately during the 2023 and 2024 seasons. Differences among the groups were determined using a fixed one-way analysis of variance at a significance level of 0.05. Comparisons between groups were conducted using Student's *t* test.

In 2023, the $ClO_3^-$ concentrations measured among the plots subjected to the four drip line cleaners were significantly different ($P$ = 0.0048), mimicking trends similar to those in 2024. The $ClO_3^-$ concentration of hop cones subjected to a continuous application of sodium chlorite, T2 and T3 (mean = 21.67 $mg\cdot kg^{-1}$), was 181-times greater than those treated with hydrogen peroxide + PAA (T1; mean = 0.12 $mg\cdot kg^{-1}$). The $ClO_3^-$ concentration of hop cones subjected to the 25-ppm sodium chlorite flush treatments at the end of the season (T4; mean = 2.15 $mg\cdot kg^{-1}$) was nearly 18-times greater than those subjected to the continuous application of hydrogen peroxide + PAA (T1; mean = 0.12 $mg\cdot kg^{-1}$) (Table 3).

The research plots treated with the four drip line cleaners in 2023 were re-evaluated in 2024 to investigate the trajectory of the $ClO_3^-$ levels in the hop cones over a 1-year span. Even though the 2023 research plots were not subjected to the drip line cleaner treatments in 2024, a significant difference was found in the $ClO_3^-$ levels measured in the hop cones ($P$ = 0.0117). The $ClO_3^-$ concentration of hop cones subjected to T2 and T3 (mean = 2.52 $mg\cdot kg^{-1}$) was 10.6-times greater than that of hop cones subjected to T1 and T4 (mean = 0.28 $mg\cdot kg^{-1}$) (Table 3).

**Hop perchlorate analysis.** In 2024, the $ClO_4^-$ concentrations measured from hop cones exposed to the drip line cleaner treatments differed significantly ($P$ = 0.0057). The highest $ClO_4^-$ levels were found in hop cones subjected to a continuous application of sodium chlorite throughout the entire season (T2 and

Table 2. Free and total chlorine levels in irrigation water during treatment applications of hydrogen peroxide ($H_2O_2$) peroxyacetic acid (PAA), or sodium chlorite ($NaClO_2$) in the 2023 and 2024 growing seasons.

| 2023 | | | | | 2024 | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Date | TRT no. | Line cleaner product | Free chlorine (ppm) | Total chlorine (ppm)[i] | Date | TRT no. | Line cleaner product | Free chlorine (ppm) | Total chlorine (ppm)[i] |
| 17 Jul | T1 | $H_2O_2$ + PAA | 0 | | 07/09 | T1 | $H_2O_2$ + PAA | 0 | |
| 17 Jul | T2 | $NaClO_2$ | 1.83 | | 07/09 | T2 | $NaClO_2$ | 2.16 | |
| 17 Jul | T3 | $NaClO_2$ | 1.9 | | 07/09 | T3 | $NaClO_2$ | 1.79 | |
| 24 Jul | T1 | $H_2O_2$ + PAA | 0 | | 07/16 | T1 | $H_2O_2$ + PAA | 0 | |
| 24 Jul | T2 | $NaClO_2$ | 2.01 | | 07/16 | T2 | $NaClO_2$ | 1.89 | |
| 24 Jul | T3 | $NaClO_2$ | 2.1 | 28 | 07/16 | T3 | $NaClO_2$ | 1.98 | 28 |
| 31 Jul | T1 | $H_2O_2$ + PAA | 0 | | 07/23 | T1 | $H_2O_2$ + PAA | 0 | |
| 31 Jul | T2 | $NaClO_2$ | 2.18 | | 07/23 | T2 | $NaClO_2$ | 0.8 | |
| 31 Jul | T3 | $NaClO_2$ | 2.27 | | 07/23 | T3 | $NaClO_2$ | 0.66 | |
| 7 Aug | T1 | $H_2O_2$ + PAA | 0 | | 07/30 | T1 | $H_2O_2$ + PAA | 0 | |
| 7 Aug | T2 | $NaClO_2$ | 1.98 | | 07/30 | T2 | $NaClO_2$ | 0.66 | |
| 7 Aug | T3 | $NaClO_2$ | 2.01 | | 07/30 | T3 | $NaClO_2$ | 0.93 | |
| 14 Aug | T1 | $H_2O_2$ + PAA | 0 | | 08/06 | T1 | $H_2O_2$ + PAA | 0 | |
| 14 Aug | T2 | $NaClO_2$ | 1.97 | | 08/06 | T2 | $NaClO_2$ | 2.32 | |
| 14 Aug | T3 | $NaClO_2$ | 2.01 | | 08/06 | T3 | $NaClO_2$ | 2.43 | |
| 17 Aug | T1 | $H_2O_2$ + PAA | 0 | | 08/13 | T1 | $H_2O_2$ + PAA | 0 | |
| 17 Aug | T2 | $NaClO_2$ | 1.97 | | 08/13 | T2 | $NaClO_2$ | 2.16 | |
| 17 Aug | T3 | $NaClO_2$ | 2.01 | 28 | 08/13 | T3 | $NaClO_2$ | 1.86 | 28 |
| 24 Aug | T1 | $H_2O_2$ + PAA | 0 | | 08/20 | T1 | $H_2O_2$ + PAA | 0 | |
| 24 Aug | T2 | $NaClO_2$ | 2.08 | | 08/20 | T2 | $NaClO_2$ | 1.83 | |
| 24 Aug | T3 | $NaClO_2$ | 2.24 | | 08/20 | T3 | $NaClO_2$ | 1.85 | |

[i] Total chlorine concentration value measured from T3 during a flush shock treatment reflects a 25-ppm sodium chlorite application.
T1 = treatment 1; T2 = treatment 2; T3 = treatment 3; TRT = treatment.

Table 3. Chlorate concentrations ($mg \cdot kg^{-1}$) of pelletized hop cones subjected to four drip line cleaners and evaluated in 2024 and 2023, and plots that were treated in 2023 and evaluated in 2024. The bars represent the mean chlorate concentration, and the error bars are standard errors (*SE*s). The analysis of variance was conducted using the Kruskal-Wallis test at a significance level of 0.05, and the comparisons among the groups were performed using the Wilcoxon method.

| Hop cone chlorate analysis ($mg \cdot kg^{-1}$) | | | | |
|---|---|---|---|---|
| | | 2024 Hops | 2023 Hops | 2023 Hops |
| TRT no. | TRT description | Evaluated in 2024 | Evaluated in 2023 | Evaluated in 2024 |
| | | Mean ± *SE* | | |
| T1 | Control – hydrogen peroxide + PAA | 0.15 ± 0.02 c | 0.12 ± 0.05 c | 0.24 ± 0.06 b |
| T2 | Continuous sodium chlorite | 42.05 ± 4.98 a | 19.15 ± 5.54 a | 1.63 ± 0.46 a |
| T3 | Continuous sodium chlorite + shock | 47.23 ± 6.74 a | 24.18 ± 3.24 a | 3.42 ± 1.3 a |
| T4 | 15-min shock sodium chlorite | 5.46 ± 1.44 b | 2.15 ± 0.4 b | 0.32 ± 0.07 b |
| $\chi^2$ | | 12.7279 | 12.9044 | 11.0074 |
| *P* value | | **0.0051** | **0.0048** | **0.0117** |

Different letters following the mean ± *SE* represent a significant difference among the groups.
PAA = peroxyacetic acid; T1 = treatment 1; T2 = treatment 2; T3 = treatment 3; T4 = treatment 4; TRT = treatment.

T3), with an average $ClO_4^-$ concentration of 0.255 $mg \cdot kg^{-1}$. The $ClO_4^-$ concentration of hop cones subjected to T2 and T3 was 58% higher than that of hop cones subjected to T1 and T4 (Table 4).

Unfortunately, a $ClO_4^-$ analysis of hop cones was not conducted in 2023. However, in 2024, a $ClO_4^-$ analysis of hop cones that were subjected to the drip line cleaners in 2023 was conducted. The ANOVA did not support a significant difference in $ClO_4^-$ concentrations among the drip line cleaner treatments ($P = 0.4707$). The mean $ClO_4^-$ concentration measured in the hop cones subjected to the four drip line cleaners was 0.067 $mg \cdot kg^{-1}$ (Table 4).

**Soil and irrigation water chlorate and perchlorate analyses.** A $ClO_4^-$ analysis of soil subjected to four different drip line cleaner treatments revealed a statistically significant difference among the treatment means in 2023 ($P = 0.0031$), but not in 2024 ($P = 0.0838$). Soil treated with a continuous application of 2 ppm sodium chlorite plus two 25-ppm sodium chlorite shock treatments at the end of the season produced the highest $ClO_3^-$ level (0.005 $mg \cdot kg^{-1}$). The lowest $ClO_4^-$ level (0.0043 $mg \cdot kg^{-1}$) was found in soil that was treated with two 15-min shock treatments at 25 ppm at the end of the season. However, there was no statistical difference between T1 and T2. The mean soil $ClO_4^-$ concentrations across the four drip line cleaners were 0.0025 $mg \cdot kg^{-1}$ in 2024 and 0.0046 $mg \cdot kg^{-1}$ in 2023 (Table 5). Across both years, analyses of soil $ClO_3^-$ from plots subjected to the drip line cleaner treatments revealed extremely low $ClO_3^-$ levels, with values below the limit of calibration (0.05 $\mu g \cdot kg^{-1}$). Additionally, analyses of $ClO_3^-$ and $ClO_4^-$ of the irrigation water in 2023 and 2024 collected during June and July revealed no detectable levels (10.0 $\mu g \cdot kg^{-1}$) of these oxyanions.

Table 4. Perchlorate analysis ($mg \cdot kg^{-1}$) of pelletized hop cones subjected to four drip line cleaners evaluated in 2024 and from plots treated in 2023 and evaluated in 2024. The analysis of variance was conducted using the Kruskal-Wallis test at a significance level of 0.05, and the comparisons among the groups were performed using the Wilcoxon method.

| Hop cone perchlorate analysis ($mg \cdot kg^{-1}$) | | |
|---|---|---|
| TRT | 2024 Hops evaluated in 2024 | 2023 Hops evaluated in 2024 |
| | Mean ± *SE* | |
| T1 | 0.080 ± 0.004 b | 0.060 ± 0.008 |
| T2 | 0.220 ± 0.016 a | 0.060 ± 0.004 |
| T3 | 0.255 ± 0.032 a | 0.083 ± 0.019 |
| T4 | 0.125 ± 0.018 b | 0.063 ± 0.009 |
| $\chi^2$ | 12.3167 | 0.9307 |
| *P* value | **0.0064** | 0.4453 |

Different letters following the mean ± standard error (*SE*) represent a significant difference among the groups.
PAA = peroxyacetic acid; T1 = treatment 1; T2 = treatment 2; T3 = treatment 3; T4 = treatment 4; TRT = treatment.

## Discussion

**Hop cone yield.** Although hop cone yield was not part of the main scope of this study, it is worth noting that plants subjected to chlorine-based line cleaners throughout the hop-growing season produced significantly higher hop cone yields than those treated with hydrogen peroxide (Fig. 3C). However, this was at the expense of significantly higher $ClO_3^-$ levels in the hop cones. There is limited literature linking sodium chlorite applications to increased crop yield. The primary agricultural use of sodium chlorite has been as a disinfectant and antimicrobial agent. Its role in enhancing crop yield through direct application remains underexplored in scientific studies. However, it is noteworthy that chloride ions, which are related to compounds like sodium chloride, have been studied to determine their effects on plant growth. Research suggests that chloride can improve nitrate utilization and nitrogen use efficiency in plants, potentially leading to increased biomass (Rosales et al. 2020).

**Hop cone chlorate and perchlorate analyses.** The findings indicated that using chlorine-based products in hop production can substantially increase $ClO_3^-$ and $ClO_4^-$ residues in hop cones. The $ClO_3^-$ concentrations reached up to 47 $mg \cdot kg^{-1}$ (Table 3), significantly exceeding the EU MRL of 0.05 $mg \cdot kg^{-1}$ (European Commission 2020b). The $ClO_4^-$ levels, although lower, reached a maximum of 0.25 $mg \cdot kg^{-1}$ (Table 4), which remains relevant given the potency of $ClO_4^-$ potency to inhibit iodine uptake. Continuous applications of sodium chlorite (T2 and T3) as a drip line cleaner resulted in $ClO_3^-$ levels up to 300-times higher than those observed under the grower standard treatment using hydrogen peroxide and peroxyacetic acid (T1). Although $ClO_4^-$

Table 5. Chlorate and perchlorate analyses of soil subjected to four drip line cleaners, which were treated in 2023 and evaluated in 2024 and those that were treated and evaluated 2024. The analysis of variance was conducted using the Kruskal-Wallis Test at a significance level of 0.05, and the comparison among the groups was made using the Wilcoxon Method.

| 2024 Soil chlorate and perchlorate analysis ($mg \cdot kg^{-1}$) | | | |
|---|---|---|---|
| Application year | TRT | Perchlorate | Chlorate |
| 2023 | | Mean ± *SE* | |
| | T1 | 0.0044 ± 0.00003 c | <LoC |
| | T2 | 0.0047 ± 0.00008 b | <LoC |
| | T3 | 0.0050 ± 0.00006 a | <LoC |
| | T4 | 0.0043 ± 0.00001 c | <LoC |
| | $\chi^2$ | 13.8274 | |
| | *P* value | **0.0031** | |
| 2024 | T1 | 0.0014 ± 0.00049 | <LoC |
| | T2 | 0.0030 ± 0.00119 | <LoC |
| | T3 | 0.0042 ± 0.00091 | <LoC |
| | T4 | 0.0015 ± 0.00040 | <LoC |
| | $\chi^2$ | 6.6538 | |
| | *P* value | 0.0838 | |

LoC (0.05 $\mu g \cdot kg^{-1}$) = limit of calibration; T1 = treatment 1; T2 = treatment 2; T3 = treatment 3; T4 = treatment 4; TRT = treatment.

residues remained below the EU MRL of 0.75 $mg \cdot kg^{-1}$ (European Commission 2020a), their relative increase underscores the potential unintended consequences of chlorine-based disinfectants in agricultural systems. These results support the hypothesis that chlorine-based cleaning agents contribute to $ClO_3^-$ and $ClO_4^-$ accumulation in hops and highlight the importance of exploring alternative practices to meet regulatory standards and ensure food safety.

Despite the use of hydrogen peroxide and peroxyacetic acid (T1), both of which are nonprecursors for $ClO_3^-$, $ClO_3^-$ concentrations in T1-treated hop cones still exceeded the EU MRL, with measurements of 0.12 $mg \cdot kg^{-1}$ in 2023 and 0.15 $mg \cdot kg^{-1}$ in 2024 (Table 3). Similarly, $ClO_4^-$ concentrations reached 0.08 $mg \cdot kg^{-1}$ in T1-treated hop cones in 2024 (Table 4). These findings suggest that factors beyond direct chlorine-based treatment may contribute to the presence of $ClO_3^-$ and $ClO_4^-$. Given that PNW hop growers have not used chlorine-based line cleaners for at least the past 3 years, additional research is needed to determine potential sources of these residues (Hop Growers of Washington 2025).

Our findings also demonstrated that replacing chlorine-based line cleaners, such as sodium chlorite, with hydrogen peroxide and peroxyacetic acid over 1 year could reduce $ClO_3^-$ levels in hop cones by up to 88% (Table 3). Although $ClO_4^-$ levels in hop cones were not measured in the first year of this study, second-year data suggest that its reduction may also be substantial, with a potential decrease of up to 61% under hydrogen peroxide treatment (Table 4).

**Soil chlorate and perchlorate analysis.** A soil analysis further supported the hypothesis that sodium chlorite application contributes to $ClO_4^-$ accumulation. The $ClO_4^-$ levels in the top 15 cm of soils treated with sodium chlorite throughout the season reached 0.0042 $mg \cdot kg^{-1}$, while those treated with hydrogen peroxide and peroxyacetic had $ClO_4^-$ levels that reached 0.0014 $mg \cdot kg^{-1}$. These concentrations are consistent with naturally occurring background levels (Andraski et al. 2014). No detectable $ClO_3^-$ levels were found in the top 15 cm of soil across the four treatments. These findings align with our hop cone $ClO_3^-/ClO_4^-$ analyses, where hop cone $ClO_3^-$ levels were 140-times greater than in $ClO_4^-$ (Tables 4 and 5). This phenomenon has been attributed to an innate inability of plants to hyperaccumulate $ClO_4^-$ in leaves and other organs once a maximum level has been reached, leading to the plant's exudation, transformation, or transpiration of $ClO_4^-$. In lettuce treated with $ClO_4^-$ ions, the levels in leaves reached 750 $ug \cdot kg^{-1}$ after 4 weeks and then decreased to just 20 $ug \cdot kg^{-1}$ in the fifth week, suggesting the potential of plant $ClO_4^-$ exudation, transformation, or transpiration from leaves (Yu et al. 2004). The undetectable $ClO_3^-$ levels in the soil treated with sodium chlorite suggest that hop plants have an excellent affinity for taking up $ClO_3^-$, as noted by the high levels of $ClO_3^-$ found in hop cones (as high as 47.23 $mg \cdot kg^{-1}$).

## Conclusion

This study provides critical insights into the impact of chlorine-based drip line cleaners on $ClO_3^-$ and $ClO_4^-$ accumulation in hops. The results demonstrate that continuous application of sodium chlorite through irrigation significantly increases $ClO_3^-$ and $ClO_4^-$ residues in hop cones, with $ClO_3^-$ levels surpassing the European Union's MRL of 0.05 $mg \cdot kg^{-1}$ and $ClO_4^-$ concentrations remaining below the regulatory threshold of 0.75 mg·kg. Our findings confirm that hydrogen peroxide and peroxyacetic acid-based cleaners effectively reduce $ClO_3^-$ and $ClO_4^-$ contamination compared with sodium chlorite treatments. Additionally, residual $ClO_4^-$ levels in untreated plots from the previous season indicate potential long-term persistence; therefore, further investigations of environmental and agronomic factors that influence residue accumulation are warranted. Future research should explore $ClO_3^-$ and $ClO_4^-$ transformation pathways in soil and plant systems, assess the carryover effect over several years, and evaluate industry-wide strategies to minimize their accumulation in hops. By addressing these challenges, the hop industry can ensure sustainable cultivation practices, maintain international market access, and align with evolving regulatory frameworks while safeguarding consumer health.

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