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Control of Chlorine and Hypochlorite Residues in the Dairy Chain

Chlorine-based products are widely used in both dairy farming and dairy processing as effective cleaners, sanitizers, and teat disinfectants. The use of chlorinated products in pre-milking disinfection practices in the form or teat dips or sprays, or in equipment sanitation without a post-rinse final sanitizing step, can potentially contribute to residues in milk. Also, if chlorine containing products are not used as recommended, or if the equipment has improper drainage, both residues and derivatives can enter into dairy products such as fluid milk, butter, and milk powder. The residues in milk potentially most likely arising from use of chlorine-based cleaning and sanitizing solutions are chlorite, chlorate, perchlorate, trichloromethane, cyanurate and chlorhexidine.

Potential most likely residues and derivatives

During use, most active-chlorine compounds found in chlorinated cleaners and disinfectants decompose slowly in water over time but can react rapidly when in contact with organic matter. In addition to inorganic disinfectant by-products such as chlorate and perchlorate, chlorine will form chlorinated organic compounds upon interacting with organic matter (Resch & Guthy, 2000). Table 1 summarises the use of the main disinfectant agents and the residues and derivatives that might be originating.

Table 1: Usage of main disinfactant agents and potential resulting residues and derivates.

Disenfection Agent	Use	Residues[1] and Derivates[2]
Chlorhexidine	Teat dip	chlorhexidine[1]
Chlorine dioxyde	Teat dip and hard surface sanitizer	chlorite[2], chlorate[2]
Hypochlorous acid (HClo)	Hard surface sanitizer	chlorite[2], chlorate[2], trichloromethane[2], cyanurate (when HClO is produced from sodium dichloroisocyanurate)[2]
Sodium hypochlorite	Chloralkaline detergents, hard surface disinfectant, towel sanitizer	chlorate[2], perchlorate[2], haloacetates[2]

[1] Residues are defined as unaltered residual chemical product. [2]Derivatives are defined as secondary products formed during the cleaning and sanitation processes as a result of breakdown of the original formula and/or its reaction with both organic and inorganic molecules naturally present in the environment.

- Chlorite (ClO_2^-) can be formed when chlorine dioxide (ClO_2) reacts with water. Chlorite in water may move into groundwater. Neither chlorine dioxide nor chlorite build up in the food chain (Agency for Toxic Substances and Disease Registry [ATSDR], 2004).
- Chlorate (ClO_3^-) may be formed during the decomposition of hypochlorite solutions during storage (Garcia-Villanova, 2010) or as a residual by-product of chlorine dioxide formation (Yang, 2013). Formation is dependent on the concentration, temperature and pH of the solution (McCarthy, 2018). Entry of chlorate into the dairy production chain will depend on multiple factors such as the level of chlorate formed in the stored hypochlorite solution, and on the efficiency of removal of chlorate residues during equipment rinsing cycles.
- Perchlorate (ClO_4^-) is a ubiquitous environmental contaminant, with both natural and industrial sources. Industrial sources of perchlorate include fertilizers and chlorine-based sanitizers. Perchlorate has been detected in hypochlorite solutions used for water disinfection (European Food Safety Authority, 2014) and cleaning and disinfection of milking equipment (Sanchez, 2008). It has low reactivity, high persistence and exhibits good solubility in water. The contribution of hypochlorite used in cleaning and disinfection of milking equipment to perchlorate in raw milk was considered insignificant in a controlled study (Rice, 2007), but it is not known whether this conclusion is also valid for milk collection procedures at dairy farms in practice.

- Trichloromethane (TCM): Contact of chlorine with organic material, e.g., milk, can result in the formation of total organic chlorine (Ryan, 2013) that consists of both volatile and non-volatile chlorinated organic compounds. The most important of the volatile organic chlorine group is TCM which can accumulate in the fat-rich portions of dairy products such as cream and butter. Resch and Guthy (2000) showed that the formation of TCM can occur in recycled cleaning and sanitizing solutions when rinsing (pre- and post-washing) with water was omitted from the on-farm milking machine wash procedure.

- Cyanurate is the anion of cyanuric acid. It may be formed through dechlorination of sodium dichloroisocyanurate when that is used for the formation of hypochlorous acid (Wahman, 2018). Cyanuric acid is a -United States Food and Drug Administration -accepted component of feed-grade biuret, a ruminant feed additive.
- Chlorhexidine has been detected in nature, including water bodies (Pereira-Marostica, 2023). While chlorhexidine is relatively non-toxic at normal use concentrations, it has been found to bioaccumulate and have a low degradation profile. A study by Middleton et al., (2003) estimated the mean elimination half-life for chlorhexidine after intramammary injection to be 11.5 days.
- Haloacids: monochloroacetic, dichloroacetic (DCA), and trichloroacetic (TCA) acids, are the result of the reaction of active-chlorine disinfectants reacting with organic matter during cleaning and disinfection and may accumulate in high-fat dairy products such as cheese and butter.

Toxicity and human health risk

- Chlorite and Chlorine Dioxide: exposure to concentrated forms, greater than those that can be present in potable water and milk, may cause minor irritation in the mouth, oesophagus, or stomach. There is no evidence of reproductive or carcinogenic health effects (ATSDR, 2004).
- High doses of both chlorate and perchlorate have been associated with inhibition of iodine uptake in humans (McCarthy, 2018; Braverman, 2005) causing reduced production of thyroid hormones and hypothyroidism. The contamination of infant formula with chlorate is a major concern due to the vulnerability of infants, which have lower tolerance than adults; however, there is little evidence that current residues in milk pose a health risk (Li, 2021).
- TCM: Oral consumption of low levels of these compounds poses a minimal risk to human and animal health. The major effect from acute inhalation exposure to TCM in humans is central nervous system depression. Acute ingestion, outside normal dietary exposure, can result in liver and kidney damage, high-dose, long-term studies have also shown carcinogenicity. However, it is clear from the current weight of available evidence that exposures to low concentrations of TCM, even over time, do not pose significant carcinogenic risks (ATSDR, 2024).
- Cyanurate has been found to be readily and quantitatively eliminated from the body unchanged and no teratogenic, mutagenic, or reproductive toxicity has been found on laboratory studies. Cyanuric acid has low acute oral toxicity and is considered non-genotoxic (World Health Organization, 2009).
- Chlorhexidine absorption from the digestive tract or through the skin is negligeable. It has been linked with rare allergic reactions, respiratory distress, and ototoxicity (Below 2017; Hirata 2002).
- DCA poses very little risk in acute dosages. Chronic administration in rats resulted in kidney and liver damage, testicular degeneration, and vascular changes in the brain and spinal cord (ATSDR 2004). Compared to DCA, the toxicology of TCA is largely unknown (Anand 2014).

Legislation and maximum residue limits

Regulations for the control of the presence of chemical derivatives throughout the food chain are established by country specific health agencies. In many countries, outside of the United States, the European Union and New Zealand, the regulation of chlorine derivatives in milk is not readily available.

Sanitizers and disinfectants are considered to be antimicrobial pesticides in the United States according to the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) and are regulated under the jurisdiction of the United States Environmental Protection Agency (US EPA). The US EPA-approved label indicates at what concentration sanitizers and disinfectants can be used without risk of significant remaining residues, which products will require a potable water rinse, and which will not. Sodium, potassium, and calcium hypochlorite, three of the most common sources of active chlorine in chlorinated cleaners, are exempted from the requirement to establish a residue tolerance in the United States due to their chemical nature and structure and a long history of safe public use in the disinfection of water supplies. Furthermore, sodium hypochlorite is one of the few substances that has been designated as "Generally Recognized as Safe" (GRAS) for use in the disinfection of food crops (referred in the Code of Federal Regulations as 40 CFR 180.2).

All biocidal products containing approved active substances are evaluated for safety and efficacy before they are allowed to be sold in the European Union under Regulation (EU) 528/2012. However, products that were on the market before 2000 can continue to be sold while the authorities are performing a full evaluation of the active substances they contain. The use of chlorinated compounds is restricted in the food industry in certain European countries, such as for example Germany (Expert Group for Technical Advice on Organic Production, 2016). EU Regulation 396/2005 sets a default maximum residue limit (for pesticides) of 100 µg/kg where no specific maximum residue limit has been set, as is the case with chlorate, which is a banned pesticide. Perchlorate is considered to be a contaminant rather than a pesticide. For infant formula, follow-on formula and other foods for infants and young children, a default maximum limit applies for perchlorate of 10 µg/kg, as consumed.

The contaminant chlorate is regulated in New Zealand, with maximum limits set in infant formula and follow-on formula and an action limit set in milk. Under New Zealand legislation the dairy manufacturer is the 'Risk Management Programme Operator' and accountable for the production of safe food in accordance with regulatory requirements. The contaminant chlorate is monitored through New Zealand's official 'National Chemical Contaminants Programme'.

Risk mitigation

Where the primary source of chlorine derivative residues in milk is cleaning or sanitizing solutions, the critical overall parameter required to achieve a residue reduction in milk is the appropriate use of the cleaning or sanitizing product according to manufacturer label recommendations regarding storage, concentration and post-rinse needs. Generally, preventing or mitigating residue occurrence is necessary as, once present, the removal of these residues is impractical at later stages in the food manufacturing process.

Mitigation factors include the correct identification of the causes of high residue in milk on farms or in processing, the effective transfer of the information for corrective action to the farmer or factory manager, in the case of on-farm or processing issues, respectively, continued sampling and analysis and finally prompt feedback of the results to the concerned actor. It is critical that prevention and control of residues is addressed without compromising microbiological hygiene, which is of prime importance to deliver food products that are safe for consumption. Precautionary practices relating to the use of sodium hypochlorite in cleaning/disinfection practices have been developed by Gleeson and O'Brien (2016). These include utilizing adequate and label compliant levels of sodium hypochlorite in the cleaning product, ensuring the final volume of rinse water is sufficient to remove any residues, minimizing the storage time of cleaning products containing chlorine, purchasing such products as close to their manufacture date as possible, and storing chlorinated products in cool and dark environments.

Conclusion

Use of detergents, sanitizers and udder health disinfectants, serve an important role in safeguarding the safety and quality of dairy products as well as animal health and welfare. While dairy product manufacturers must be mindful of their responsibilities to consumers to produce food that is safe to eat and endeavour to meet low residue levels, it is absolutely critical that dairy foods are microbiologically safe, and that microbiological quality is not compromised.

More details are available from the Bulletin of the IDF N° 529/2024 on Control of Chlorine and Hypochlorite Residues in the Dairy Chain (International Dairy Federation, 2024).

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