

Optimization of CIP Process for Ozone Sanitization Retrofit

James R Jackson¹ and Arturo Enriquez²

1. Mazzei Injector Company, LLC., Bakersfield, CA, USA
2. Laboratorios Sanox SA de CV, Querétaro, Querétaro, MEXICO

Abstract

The inclusion of ozone sanitization in Clean-In-Place (CIP) processes promises multiple benefits to the food processing and beverage industries. Chief among these is the reduction in plant down time during the CIP procedure, a benefit which goes directly to the bottom line as increased plant production.

Over the years, numerous white papers have lauded the inclusion of ozone sanitization in the CIP process. These case histories credit ozone sanitization with a rapid reduction in bio-film and demonstrate a significant return on the investment made in the ozone system; yet today only a handful of plants include ozone a part of their CIP procedure.

The lack of growth in ozone CIP sanitization suggests that integrating ozone into a CIP process requires a level of knowledge and commitment beyond that required for other ozone applications. This paper examines the intricacies of optimizing a beverage CIP system in preparation for retrofit with an ozone sanitization system; illustrating how the benefits of ozone sanitization are highly dependent on the skill at which the ozone system is integrated into the existing CIP process.

Key Words: Ozone; CIP, Clean-In-Place; HACCP; Beverage; Alicyclobacillus

Introduction

The beverage industry became a stakeholder in ozone with the introduction of bottled water as an alternative beverage to fruit juice and carbonated soft drinks. Ozone's ability to sanitize bottles and bottle fillers without imparting a chemical aftertaste made it the ideal bottled water sanitizer, leading to intense lobbying of the US Food and Drug Administration (FDA) to approve the use of ozone for bottled water sanitation. In November of 1982, the FDA declared ozone as GRAS for the treatment of bottled water (21 CFR § 184.1563).

The successful use of ozone for bottled water sanitation led many in the beverage industry to expand ozone's use to the disinfection of their process water systems. Today, many beverage

plants rely on ozone as their primary process water disinfectant. Chemical sanitization, however, continues to dominate the industry's Clean-In-Place (CIP) process.

CIP Process

CIP Cleaning

Dairies and Food processing facilities are required to meet the 3-A Sanitary Standards (3-A SSI) which are published by 3-A SSI, Inc., a non-profit organization composed of equipment manufacturers, food processors, representatives from the Department of Agriculture (USDA), the FDA and public health officials. The 3-A SSI standard defines a Clean-In-Place (CIP) process as: “The removal of soil from product contact surfaces in their process position by circulating, spraying, or flowing chemical solutions and water rinses onto and over the surfaces to be cleaned. Components of the equipment, which are not designed to be cleaned in place, are removed from the equipment to be Cleaned-Out-Of-Place (COP) or manually cleaned”.

The standard CIP process begins at the end of a product run and uses a timed sequence of events. A typical beverage CIP system consists of three stainless steel storage vessels: one for hot water rinse, one for cold water rinse and one for the detergent solution. The rinse and detergent solutions are pump through a specific CIP circuit to clean and disinfect lines, storage tanks and bottle fillers that have been taken out of service for cleaning. An automated chemical feed station delivers the required chemical concentrate during the cleaning processes. (Figure 1: Typical chemical CIP sequence).

Sequence	Description	LPM	Minutes	°C	Notes
1	Preheat detergent and rinse water		36		Steam heating
2	Water rinse to drain	150	10	30	Ambient water
3	Hot Detergent	150	30	86	
4	Hot water rinse	150	10	86	Until < 750 μs
5	Hot water disinfection	150	30	86	To drain
6	Ambient water rinse	150	≥ 5	30	To drain until < 750 μs and < 40 °C

Figure 1. Typical chemical CIP sequence.

CIP Sanitation

Food and beverage sanitation standards are not defined by 3-A SSI, but instead fall under FDA and USDA guidelines as well the manufacturers own standards. In addition to their own Good Manufacturing Practices or GMP's, food and beverage manufacturers follow the Hazard

Analysis and Critical Control Point (HACCP) seven principles designed to prevent food borne illness. These 7 principles, accepted by all segments of the food and beverage industry are:

1. Conduct a hazard analysis.
2. Determine critical control points (CCPs).
3. Establish critical limits.
4. Establish monitoring procedures.
5. Establish corrective actions.
6. Establish verification procedures.
7. Establish record-keeping and documentation procedures.

Each segment of the food industry has its own unique basket of target organisms they must inactivate to ensure against food borne illness and premature spoilage. Egg producers strive to eliminate salmonella, while the produce and beef industries monitor for *E. coli*. The beverage industry, also concerned with pathogens, faces some unique challenges in preventing contamination with molds, yeasts and thermophilic microorganisms.

Alicyclobacillus, for example, is an aerobic, non-pathogenic spore forming bacteria found in soil. When present, it produces the metabolic by-products of guaiacol, 2,6-di-bromophenole and 2,6-di-chlorophenole. Concentrations of these by-products in the parts per trillion can cause smoky and stale off-flavors in fruit juices and sweetened beverages. Tolerant to low pH and high temperatures, the pasteurization process activates the spore form of the organism which can then proliferate at temperatures up to 70°C.

Though tolerant to low pH and high temperatures, *Alicyclobacillus* is inactivated through exposure to ozone and its metabolic by-products, if present in the process water, are rapidly oxidized to non-objectionable aldehydes and organic acids (Figure 2).

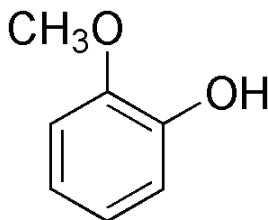


Figure 2. Guaiacol molecular structure.

Ozone CIP

Pilot Study

Laboratorios Sanox SA de CV (Sanox), a chemical company which specializes in CIP systems, contacted Mazzei Injector (Mazzei) to discuss the use of ozone as an alternative to peracetic acid sanitation or heat sterilization at their customer plants. A review of the published literature provided by Mazzei convinced Sanox to pilot ozone at a local bottling plant in Merida, YUL, Mexico.

Pre-ozone CIP Evaluation and Optimization

Prior to piloting with ozone, the plant's existing CIP process was evaluated. Equipment inspections and a review of the current CIP system indicated the following deficiencies and concerns:

1. Non-wetted surfaces within vessels (shadowing) following CIP spray.
2. Stagnant dead legs in pipeline system.
3. Non-turbulent flow within pipelines.
4. High energy costs from the use of hot caustic solution and rinse water.
5. Programming issues with the CIP central PLC controller.
6. Safety concerns on the handling and storage of peracetic acid (blend of acetic acid + hydrogen peroxide) to disinfect syrup lines and storage vessels.

The issue of shadowing and the existence of dead legs in the process pipeline was of serious concern, because it meant that some internal surfaces of the process piping and vessels were not adequately cleaned and sanitized during the CIP procedure. To correct these deficiencies, portions of the process piping were modified to eliminate dead legs and additional spray heads were installed in those tanks with shadowing. The optimum orientation of all tank spray heads was confirmed through visual inspection following a CIP trial rinse.

Turbulent flow is required within a pipeline to ensure that the CIP detergent lifts and removes product residue (soil) from pipe surfaces and to prevent the redeposit of soil to downstream areas of low velocity. In the critical sections of the CIP loop, pipeline was changed to ensure a minimum fluid velocity of 1.5 m/second.

To reduce energy costs and eliminate safety concerns on the use of peracetic acid, an ambient temperature, surfactant enriched detergent followed by an ozone rinse was proposed for adoption plant wide following validation through pilot testing in a limited plant area. PLC reprogramming was offered as part of the proposed ozone pilot.

CIP Process: Pre-Ozone Cleaning

Soil remaining on beverage pipe and vessel surfaces following a product run is composed of simple carbohydrates, primarily sucrose and fructose sugar solutions. Removing this soil is an

essential step in preparing the surface for sanitation, because remaining soil residue increases ozone demand, making it difficult to achieve a dissolved ozone residual at the end of the CIP loop. If any soil remains on the surface following the final ozone rinse, it then serves as a precursor substrate for bio-film formation.

At the start of the ozone pilot study, the plant was using a hot caustic (sodium hydroxide) solution to clean surfaces in preparation for sanitization (Figure 1). Commercial caustic is frequently utilized as the CIP detergent because of the low cost of commercial sodium hydroxide. In solution, the sodium hydroxide ionizes into sodium and hydroxyl ions. When heated, these ions rapidly hydrolyze sugar rings, breaking them down to more soluble forms. When the primary sugar is sucrose, the initial reaction is the hydrolysis of the disaccharide ring to fructose and glucose. Continued contact with the caustic solution results in hydrolysis and solubilization of these disaccharides (Figure 3).

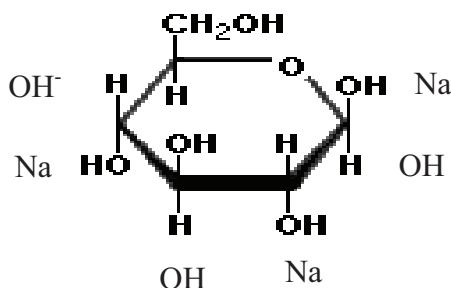


Figure 3. Hydrolysis of glucose.

There are multiple drawbacks to utilizing commercial caustic as the CIP detergent. First, insoluble precipitates are often formed during the CIP cleaning cycle, fouling surfaces. When present, these alkaline deposits react with molecular ozone, making it impossible to obtain a downstream dissolved ozone residual in a reasonable amount of time. Second, there is a time lag as well as a high energy cost to heat the caustic solution to its active cleaning temperature. The time lag expands bottling line downtime, reducing plant productivity; the high energy costs reduce profitability. Finally, the high dosage required to effectively clean surfaces with commercial caustic significantly reduces the cost benefit of using a commodity cleaning solution. Applied at more than 3 times the feed rate of specialty cleaning solutions, the use cost of caustic soda approaches the cost of specialty cleaning agents which designed to remove soil without the fouling and high energy cost associate with commercial caustic cleaning.

The alternative to hot caustic cleaning, surfactant based detergent, contains a blend of wetting agents and dispersants. These surfactant solutions remove soil by hydrating, lifting and suspending the surface residue into solution. The surfactant molecules are manufactured with a hydrophilic chemical group at one end and a hydrofobic chemical group at the other. The hydrophobic end is talyored to penetrate the soil. Once soil is penetrated, polar surface charges at the surfactant's hydrophilic end repulse the hydrated soil away from the surface (Figure 4).

Surfactant based solutions are more costly than commercial caustic, however, low dosage requirements, the ability to clean at ambient water temperatures and the absence of alkaline precipitates make surfactant solutions ideal detergents to prepare surfaces for ozone CIP sanitization.

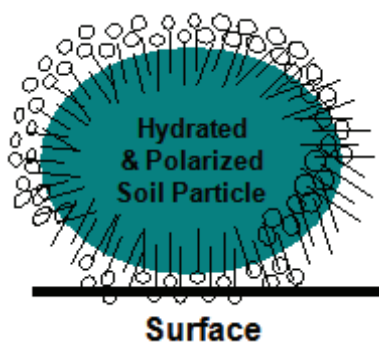


Figure 4.

CIP Ozone Retrofit

Initial Results

The ozone retrofit consists of an oxygen fed, 30 gram per hour ozone generator provided by Guardian Manufacturing, an Air Sep AS-12 oxygen concentrator and a Mazzei GDT™ Process ozone transfer and degas skid. Dissolved ozone controllers and ambient air monitors for the generator, work space and points of application were provided by Orbisphere and Analytical Technology. PLC programming to modify and integrate ozone into the CIP operation was provided by Sanox S.A.

To minimize space and pump energy requirements, the ozone contact and degasification system was designed to be operated as a side stream (Figure 5. GDT Process side stream ozone contacting system for Sanox S. A. ozone pilot).

Initial ozone pilot runs conducted at 30°C water temperatures limited the distance the system could reach with a dissolved ozone residual. It was concluded that the starting ozone dosage was insufficient to handle the demand and decay rate of the entire CIP pilot loop. With all available funding spent, the CIP ozone pilot team decided to increase gas solubility and reduce the ozone decay rate by chilling the CIP rinse water to 14°C using the plant's Carbo Cooler, a standard piece of bottling equipment which is designed to simultaneously chill and carbonate beverages.

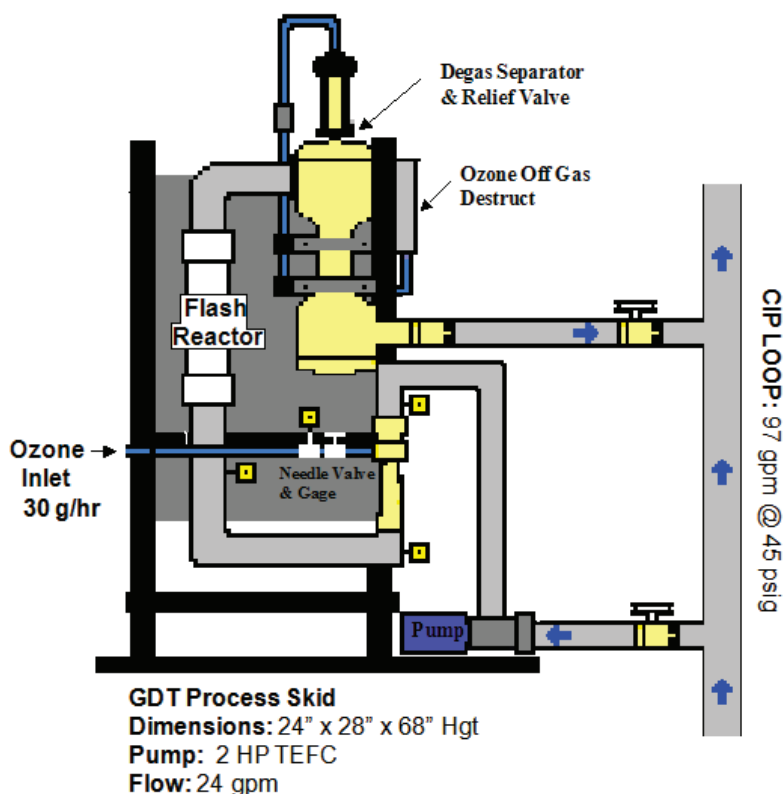


Figure 5. GDT Process side stream ozone contacting system for Sanox S. A. ozone pilot.

CIP Ozone Retrofit

Microbiological Data

The microbiological counts obtained on system rinse water following an ozone wash are shown in Figures 6, 7 and 8. Colony forming units (CFU's) on all systems tested were well within the plants microbiological control targets for bacteria, fungi and yeast; most systems showed 0 CFU's on post ozone rinse samples held for up to 120 hours.

The counts obtain from filler valves and Canes (valve stems) are surface swab cultures. The CFU's present is a consequence of the fillers not rotating during the CIP cycle. Those surfaces not exposed to the CIP solutions undergo a second manual cleaning.

Figures 9, 10 and 11 compares the plant's standard CIP cycles with the new ambient detergent - ozone CIP process.

Filler No. 3			Total Aerobic Count (u.f.c./1ml.)					Fungus (u.f.c./20 ml o 100 ml.)					Yeast (u.f.c./20 ml. o 100 ml.)					
			Target	24 hrs.	48 hrs	72 hrs	96 hrs	120 hrs	Target	48 hrs	72 hrs	96 hrs	120 hrs	Target	48 hrs.	72 hrs	96 hrs	120 hrs.
CIP Rinse Water	12:20:00	12/12/07	< 25	1	1	1	1	1	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Syrup	12:22:00	12/12/07	< 25	zero	zero	zero	zero	zero	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Carbo Cooler	12:24:00	12/12/07	< 25	zero	zero	zero	zero	zero	< 5	zero	1	1	1	< 10	zero	zero	zero	zero
Filler V-11	12:26:00	12/12/07	< 25	zero	zero	zero	zero	zero	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Filler V-32	12:26:00	12/12/07	< 25	1	1	1	1	1	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Filler V-57	12:26:00	12/12/07	< 25	1	1	1	1	1	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Frotis Valve No. 6	12:20:00	12/12/07	< 10	1	3	5	5	5	< 5	zero	1	1	1	< 10	zero	1	1	1
Frotis Valve No. 6	12:20:00	12/12/07	< 10	zero	zero	zero	zero	zero	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Cane #62	12:21:00	12/12/07	< 10	zero	zero	zero	zero	zero	< 5	zero	1	1	1	< 10	zero	zero	zero	zero
Cane # 63	12:21:00	12/12/07	< 10	zero	zero	zero	zero	zero	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero

Figure 6.

Syrup tanks			Total Aerobic Count (u.f.c./1ml.)					Fungus (u.f.c./20 ml o 100 ml.)				Yeast (u.f.c./20 ml. o 100 ml.)			
			Target	24 hrs	48 hrs	72 hrs	96 hrs	Target	48 hrs	72 hrs	96 hrs	Target	48 hrs.	72 hrs	96 hrs
Enj. Tq -4 (Before O3)	10:50	12/14/07	< 25	5	5	5	5	< 5	1	1	1	< 10	1	1	1
Enj. Tq -4 (After O3)	12:54	12/14/07	< 25	zero	zero	zero	zero	< 5	zero	zero	zero	< 10	zero	zero	zero

Figure 7.

Gallon Fillers			Total Aerobic Count (u.f.c./1ml.)					Fungus (u.f.c./20 ml o 100 ml.)					Yeast (u.f.c./20 ml. o 100 ml.)					
			Target	24 hrs	48 hrs	72 hrs	96 hrs	120 hrs	Target	48 hrs	72 hrs	96 hrs	120 hrs	Target	48 hrs.	72 hrs	96 hrs	120 hrs.
Galonera V 4 Rinse	14:30	12/12/07	< 25	zero	7	9	9	9	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Galonera V 6 Rinse	14:30	12/12/07	< 25	zero	zero	2	2	2	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Galonera V12 Rinse	14:30	12/12/07	< 25	zero	5	7	9	9	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero
Galonera V17 Rinse	14:30	12/12/07	< 25	zero	zero	1	2	2	< 5	zero	zero	zero	zero	< 10	zero	zero	zero	zero

Figure 8.

CIP w/hot water disinfection					
Sequence	Description	LPM	Minutes	°C	Notes
1	Preheat detergent and rinse water		36		Steam heating
2	Water rinse to drain	150	10	30	Ambient water
3	Hot Detergent	150	30	86	
4	Hot water rinse	150	10	86	Until < 750 μ s
5	Hot water disinfection	150	30	86	To drain
6	Ambient water rinse	150	≥ 5	30	To drain until < 750 μ s and < 40 °C

Figure 9.

CIP w/peracetic acid disinfection					
Sequence	Description	LPM	Minutes	°C	Notes
1	Water rinse to drain	150	10	30	
2	Hot Detergent	150	30	86	
3	Water rinse	150	10	30	To drain until < 750 μ s
4	Peracetic acid (disinfection)	150	20	30	To drain
5	Water rinse	150	5	30	To drain until < 750 μ s and < 40 °C

Figure 10.

CIP w/ambient temperature and ozone disinfection					
Sequence	Description	LPM	Minutes	°C	Notes
1	Water rinse to drain	150	10	30	
2	Surfactant Detergent	150	30	30	
3	Water rinse	150	10	30	To drain until < 750 μ s.
4	Ozone	150	20	14	Recirculate for 20 minutes, then discharge. Future will return to storage for future step 1 rinse.
5	Water rinse	150	5	30	Rinse until < 750 μ s.

Figure 11.

Ozone Retrofit Savings

The final modification, a diversion of the ozone rinse water from drain to the (former) hot water storage vessel will be completed by the end of August. The diversion will allow the plant to recycle the water for use in the pre and post detergent rinse cycles, saving over 1,500 gallons of water per CIP cycle or more 7,500 gallons per day.

The switch from hot detergent-hot water to ambient temperature (surfactant) detergent – ozone is saving over 47 million BTU's (British Thermal Units) per day, resulting in a fuel savings of \$1,384 day (Figures 12 and 13).

CIP Process	Daily CIP	Hot Minutes	Flow lbs/min	Fluid °F	Daily BTU's
Hot	5	70	342.8	186.8	59,808,727
Ambient (O3)	5	70	342.8	86	12,676,744
Daily BTU Saving:					47,131,983

Figure 12.

BTU Savings	LP BTU/Gal	LP Gallons	Boiler % Eff	LP Gallons	Cost per Gallon	Daily Savings
47,131,983	91,000	518	70	740	1.87	\$1,384

Figure 13.

The final and most significant savings is the recovery in lost production time. When utilizing the hot detergent-hot water CIP process, the plant must wait 36 minutes for the caustic detergent and hot water tanks to reach the 86°C application temperature. At a plant average of 5 CIP cycles per day, the switch to the ambient temperature surfactant detergent - ozone CIP process has added an additional 180 minutes of bottling time per production day. The monetary value of this recovered time is considered confidential and has not been released, however the plant typically produces 14,000 bottles per hour, consequently the recovered time results in an additional 2.5 million units of bottled beverage in a typical production day.

References

1. The 3A Sanitary Standards, Incorporated web site: www.3-a.org
2. The International Society of Beverage Technologist web site: www.bevtech.org.
3. The Coca Cola Bepensa Bottling group, bottling facility in Merida, YUL, Mexico.