Recommendations for rationalizing cleaning-in-place in the dairy industry: Case study of an ultra-high temperature heat exchanger

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ABSTRACT

The objective of this work was to propose a new strategy, based on objective and rational arguments and calculations, that can be implemented by plant designers and operators in the dairy industry to reduce operating time and the volume and load of effluents. The strategy is based on the on-line and off-line use of sensors and tracers, the accuracy, relevance, and robustness of which were evaluated for each phase of the sequence used for cleaning an industrial sterilizer. The efficient duration of each phase of the cleaning sequence (management of the end of operation) and the sorting of the fluids (management of mixtures and destination of fluids) were determined in real time. As a result, significant reduction in total overall duration of the cleaning sequence, wastewater volume (waste volume was reduced by half), and detergent volume (caustic soda and acid was reduced by up to a few tens of kilograms per cleaning) was achievable.

Key words: cleaning-in-place, management, sensor, effluent

INTRODUCTION

Cleaning-in-place (**CIP**) systems are commonly used in the food industry for ensuring hygienic safety of foods and for recovering plant performance. In the dairy industry, the time of nonproduction dedicated to CIP is excessive and ranges from 4 to 6 h per day. Moreover, the cleaning operation leads to 50 to 95% of the waste volume sent to the purification station, representing from 0.5 to 5 L of water per liter of processed milk regardless of the type and size of the plant or equipment (Marty, 2001; Sage, 2005). These effluents represent nonaccidental losses of matter and DM ranging from 0.1 to 6.0 g of equivalent milk per liter of processed milk. With such characteristics, these effluents contain a matter amount equivalent to 200,000 t of milk and represent approximately 1% of the 22.5×10^6 t of milk produced each year in France (Räsänen et al., 2002).

At present, the industrial CIP procedure mainly lies on practical experience imposed with excessive safety margins in terms of duration and chemical consumption. For the past 20 yr, dairy industry operators and CIP designers have not taken advantage of knowledge acquired about mechanisms, kinetics, and sensors adapted to fouling, rinsing, or cleaning operations (International Dairy Federation, 1995, 1997; Alvarez, 2003). Some efforts could therefore be made to help the dairy industry reduce both the overall duration of the cleaning operation and the volume and load of the resulting waste. This could be done by reducing the duration of each sequence phase or by using adequate fluid sorting procedures, which would lead to reuse and increased value of the food matter while always maintaining the quality of hygiene of the equipment.

The objective of this work was to propose a new strategy, based on objective and rational arguments and calculations, that can be implemented in the dairy industry to reduce overall operating time and the volume and load of effluents. The strategy is based both on the critical analysis of the literature and on the on-line and off-line use of sensors and tracers, the accuracy, relevance, and robustness of which were evaluated for each phase of the cleaning sequence to allow the on-line determination of the end of each phase and the adequate time needed for fluid sorting. More generally, this study aimed to propose a decision making tool that consists of objective and relevant methods to help CIP designers and food industry operators manage daily cleaning operations more efficiently. To illustrate the proposed strategy, the industrial case of a UHT heat exchanger treating high-fouling dessert cream was studied. The critical analysis of the existing equipment, the proposition of a revised procedure, and the experimental validation of the suggested improvements were performed.
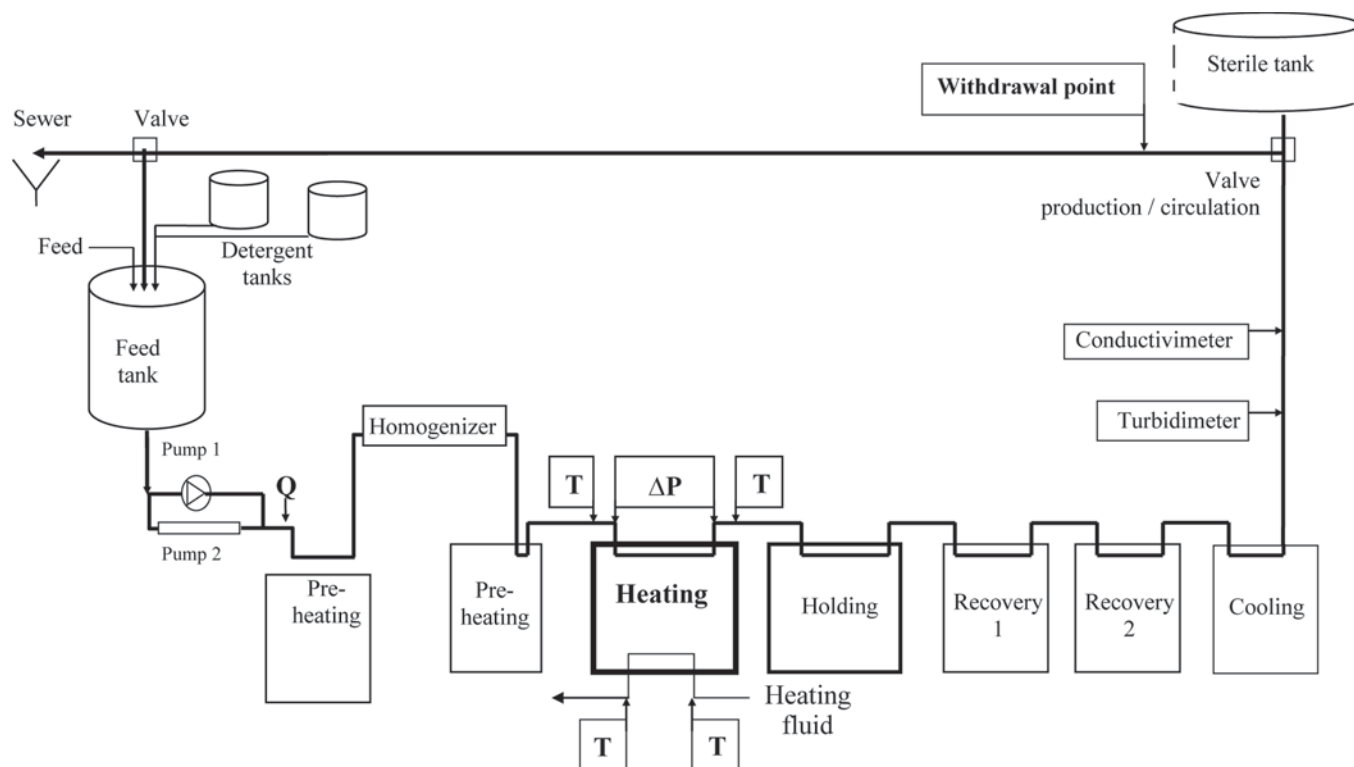
Figure 1. Schematic representation of the industrial UHT heat exchanger (ΔP = differential pressure sensor; Q = flow-rate sensor; T = temperature sensor).

MATERIALS AND METHODS

UHT Heat Exchanger

The industrial equipment studied was a tubular UHT heat exchanger (Invensys APV, Evreux, France) with 316 stainless steel tubes, each of them of 6.0 m long and consisting of 7 channels that were 14.0 mm in diameter. The sterilizer was composed of 5 zones (preheating, heating, recovery 1, recovery 2, and cooling; Figure 1). The study focused on the heating zone (inlet = 80°C; outlet = 128°C), which was the limiting zone because of its sharp fouling increase.

Several sensors were installed in the equipment (Figure 1). The flow-rate of the milk-based product (**Q**; 2.0 m^3/h) was measured with an electromagnetic flow meter (±0.5%; Magflo 6000, Danfoss, Trappes, France). A differential pressure sensor (2010 TD, 0–18 bar, ABB, Minden, Germany) was installed in the heating zone (accuracy = ±0.2% of the global scale of 10×10^5 Pa). Four 100-Ω platinum temperature sensors (±0.4°C) were installed at the entrance and outlet of the heating zone, 2 in the heating fluid and 2 in the dairy fluid compartments. A conductimeter (LMIT 08, 0–200 mS/cm, Henkel Ecolab, Nanterre, France) with an integrated 100-Ω platinum temperature sensor and a turbidimeter (Optec M, TTS Technologies, Saint Sébastien sur Loire,

France) was installed at the outlet of the cooling section. The calibration of the conductimeter was realized from physicochemical analyses performed on samples withdrawn from the equipment.

The industrial equipment allowed real time data registering and storing by a computer. All the data were collected every 20 s during the production step and every 5 s during the launching and cleaning sequence. Data were collected with the monitor PL7 Pro software (Schneider Automation, Brest, France) transferred to Access (Microsoft, Redmond, WA) and treated with Excel (Microsoft).

Operating Mode of the UHT Heat Exchanger

The sterilization was performed in 2 steps: the launching phase, during which dessert cream flushed water out from the equipment, and the production phase. At the end of the production phase, the cleaning sequence started using a decentralized CIP system. Cleaning solutions and water used during rinses were systematically discharged to the purification station after each use (i.e., it was a single-use system).

We studied the cleaning sequence that was the most time consuming and generated the most effluents. The first phase of this sequence was the flushing– first water

ALVAREZ ET AL.

rinse phase, during which water from the feed tank was used to push out the cream that was still present in the equipment. At the beginning of the phase, the nondiluted dessert cream was recovered to production and the diluted cream was sent to the sewer. The first rinse, which immediately followed flushing in a continuous mode, enabled the removal of food components that were slightly attached to the equipment and a reduction of the polluting load of the forthcoming cleaning solutions. The first alkaline cleaning phase (NaOH-1) was next, followed by an intermediate rinse; during this rinse, the detergent was washed out from the plant to avoid the neutralization of the forthcoming detergent. The next phases, in order, were the first acid cleaning phase (HNO$_3$-1); an intermediate rinse; the second alkaline cleaning phase (NaOH-2), an intermediate rinse; and the second acid cleaning phase (HNO$_3$-2). Last, during the final rinse phase, the detergent was washed out from the plant and traces of detergent were removed in order to fill in the plant with drinking water before the next production. The cleaning phases (NaOH, HNO$_3$) started with the direct addition of concentrated detergent solutions in the feed tank through a valve that induced sequential detergent injections and, consequently, oscillations of detergent concentration with time.

Table 1 gives the operating parameters for the different phases of the cleaning sequence. The nonproduction time of the sterilizer was 11.5 h/d (i.e., about half the functioning time); the CIP contributed to 88% of this nonproduction time and the launching phases contributed to approximately 12%.

Fluids

The milk-based fluid treated by the sterilizer was a complex chocolate dessert cream with a DM of 230 ± 4 g/kg. The water used was bore water. The detergent solutions were prepared from technical-grade caustic soda at 50% (Brenntag, Nantes, France) or nitric acid at 58% (Hydro Chemicals, Nanterre, France) without additives. The characteristics of the fluids were studied with a conductimeter and a turbidimeter. The sensitivity and detection thresholds of these sensors were determined for cream–water and detergent–water mixtures at the laboratory at 20 ± 2°C.

For the launching phase, DM (g/kg) was calculated from the turbidity (**Tur**) measured on-line according to

$$DM = 0.075\ Tur^2 + 1.34\ Tur - 1.76,\ r^2 = 0.97,\quad [1]$$

where Tur (%) was calibrated according to water = 0% and milk = 100%.

Table 1. Operating parameters and performances of the initial and revised cleaning sequence; the launching (17.2 ± 0.3 min) and production (162 ± 3 min) phases were similar in both cases

Item[1]	Initial cleaning sequence					Revised cleaning sequence			
	Duration (min)	Q[2] (L/h)	Te–To[3] (°C)	α[4]	Volume of effluents (L)	Duration (min)	Q (L/h)	α	Volume of effluents (L)
Flushing–first rinse	13.6 ± 0.2	2,000 ± 124 to 6,053 ± 246	124–102	17.6 ± 0.2	450 ± 15	13.6 ± 0.2	1,990 ± 10 to 5,845 ± 500	17.7 ± 0.4	542 ± 20
NaOH[-1]	18.2 ± 0.3	6,615 ± 150	100–89	5.1 ± 0.3	415 ± 15	16.1 ± 0.2	6,344 ± 555	3.6 ± 0.5	423 ± 10
IR	12.5 ± 0.3	6,902 ± 86	86–79	6.5 ± 0.1	1,433 ± 28	7.0 ± 0.3	6,984 ± 33	2.3 ± 0.4	815 ± 140
HNO$_3$[-1]	18.2 ± 0.2	6,966 ± 47	81–74	2.8 ± 0.5	469 ± 24	7.2 ± 0.2	7,002 ± 38	0.1 ± 0.5	469 ± 24
IR	16.8 ± 0.7	6,996 ± 10	79–66	4.6 ± 0.2	1,457 ± 20	7.0 ± 0.3	7,002 ± 42	0.3 ± 0.3	816 ± 30
NaOH[-2]	18.2 ± 0.3	6,892 ± 62	86–81	7.4 ± 0.5	456 ± 22	—	—	—	—
IR	12.5 ± 0.3	6,816 ± 60	86–79	6.9 ± 0.2	1,415 ± 34	—	—	—	—
HNO$_3$[-2]	18.2 ± 0.2	6,873 ± 156	81–74	0.5 ± 0.3	449 ± 21	7.2 ± 0.2	6,995 ± 40	0.0 ± 0.3	466 ± 21
FR	14.0 ± 0.2	6,977 ± 33	73–63	1.5 ± 0.3	1,734 ± 20	7.8 ± 0.4	6,984 ± 60	0.0 ± 0.2	907 ± 38
Total	142 ± 3				8,300 ± 200	66 ± 2			4,400 ± 300

[1]IR = intermediate rinse; FR = final rinse.
[2]Q = flow-rate of milk-based product.
[3]T = temperature sensor; e = entrance of the sterilizer (first value); o = outlet of the sterilizer (second value).
[4]α = fouling rate at the end of each phase.

Equation 1 was valid for 4.5 < temperature (T; °C) < 21.4 at Q ≅ 2 m^3/h and for DM ≤ 200 g/kg because the turbidimeter was saturated above this value.

For the launching and flushing–first rinse phases, the conductimeter saturated for values higher than 150 and 180 g/kg of DM, respectively. Below these thresholds, DM (g/kg) was correlated with conductivity (χ; mS/cm) according to

$$DM = 39.6\,\chi - 31.6, \; r^2 > 0.91 \qquad [2]$$

for DM (g/kg) > 0.2; 4.5 < T (°C) < 28.8; and 2 < Q (m^3/h) < 7.

For the cleaning phases and water rinses, the concentrations of NaOH (C_{NaOH}) and HNO$_3$ (C_{HNO3}) in grams per kilogram were correlated with the conductivity

$$C_{NaOH} = 0.24\,\chi - 0.21, \; r^2 = 0.98 \qquad [3]$$

for 0.063 < C_{NaOH} (g/kg) < 40.7; 40.0 < T (°C) < 63.3; and Q ≅ 7 m^3/h, and

$$C_{HNO3} = 0.22\,\chi - 0.19, \; r^2 = 0.99 \qquad [4]$$

for 0.006 < C_{HNO3} (g/kg) < 35.3; 35.0 < T (°C) < 57.0; and Q ≅ 7 m^3/h.

To calculate the DM or the detergent concentrations in a more precise manner for each phase, specific laws of equations 1 through 4 could be established from shorter ranges of conductivities (Alvarez, 2003).

At the end of intermediate rinses, the concentration in chemical oxygen demand (C_{COD}; g/kg) in the caustic soda solution was correlated with the Tur (%)

$$C_{COD} = 1.763 \times Tur - 1.631, \; r^2 = 0.97 \qquad [5]$$

for C_{COD} (g/kg) < 8.0; 40.2 < T (°C) < 59.0; and Q ≅ 7 m^3/h.

Concerning the sensors, no evolution of the conductimeter was observed for 8 mo and no maintenance (specific cleaning, zero adjustment, and so on) was necessary. However several difficulties were encountered with the turbidimeter. First, during the flushing–first rinse phase, turbidity and DM were not correlated; the turbidimeter was probably maintained fouled because of the high viscosity of the treated product. At the end of the first rinse phase, the sensor was cleaned because of the increase in flow-rate, but no appropriate correlation was proposed. This fouling phenomenon was not observed, however, in industrial equipment treating skim milk, in which the turbidimeter is an adequate sensor regardless of the mixing phases during which milk and water are mixed (Chareunphol, 2004).

Other difficulties were that a significant derivative of the turbidimeter (up to 40%) was observed during 8 mo, which necessitated verifications and calibrations, and that the soluble components of the deposit removed by the acid phase could not be quantified by the turbidimeter.

Data Treatment

Synchronizing the Time Scale. For relevance and technical reasons, sensors were installed in different zones of the equipment (Figure 1). Consequently, raw data issued from the acquisition and analyses performed on samples withdrawn were synchronized. Considering the fluid flow in the equipment to be similar to a plug flow reactor and taking into account the instantaneous variations of flow-rate, data were recalculated by referring to a single reference point, which was chosen to be the withdrawal point (Figure 1).

Fouling and Cleaning Kinetics. Several variables could be chosen to follow the fouling and cleaning evolution of the equipment. Thermal data of the heat-exchanger were not used because the specific heat capacity of the cream was unknown and the data related to the logarithmic average of the temperatures fluctuated greatly (Alvarez, 2003). However, a hydrodynamic balance enabled the calculation of the hydraulic diameter of the heat exchanger (D; meters; ±0.5%) during the cleaning phases. The fouling rate (α, %; ±0.5%) was then calculated according to Corrieu et al. (1986):

$$\alpha = \frac{D_0 - D}{D_0} \times 100, \qquad [6]$$

where the hydraulic diameter of the cleaned heat exchanger (D_0) = 14 × 10^{-3} m.

The fouling fraction removed by a phase ($\Delta\alpha$) was calculated according to

$$\Delta\alpha = \frac{\alpha_{beginning} - \alpha_{end}}{\alpha_0}, \qquad [7]$$

where $\alpha_{beginning}$ is the fouling rate at the beginning of the considered phase, measured with water; α_{end} is the fouling rate at the end of the considered phase, measured with water; and α_0 is the initial fouling rate at the end of the flushing–first rinse phase, measured with water.

The value of D was calculated experimentally from the measurement of the pressure decrease (ΔP) and the calculation of the Darcy number (Da). For an incompressible fluid flowing in a circular pipe, D can be calculated from

$$\frac{Da}{8} = \frac{\Delta P \times D}{4\ L \times \rho \times v^2},\quad [8]$$

where L is pipe length (m); ρ is fluid density (kg/m^3); and v is average velocity of the fluid in the pipe (m/s).

To take into account the pressure decrease generated by the pipe deviations, the equivalent hydrodynamic length L (99.6 m) was calculated from the real pressure decrease measured with water in a cleaned heat exchanger with D_0.

The Da is classically calculated from the Reynolds number (**Re**) following

$$\frac{Da}{8} = a \times Re^b, \quad [9]$$

where $a = 8$ and $b = 1$ in laminar regimen (Re < 2,000) and $a = 0.04$ and $b = -0.25$ (or $a = 0.023$ and $b = -0.2$) in turbulent regimen as proposed by Zigrang and Sylvester (1985) for smooth tubes, and $2 \times 10^3 < Re < 10^5$.

The Re was calculated from the generalized law

$$Re = \frac{\rho\ v^{2-n_s} D^{n_s}}{k_c \left(\dfrac{3\ n_s + 1}{4\ n_s}\right)^{n_s} 8^{n_s - 1}}. \quad [10]$$

For a Newtonian fluid (water, caustic soda, nitric acid, and so on), the structure index (n_s) = 1 and the consistency index (k_c) = μ.

From equations 8 through 10, it followed that

$$D^{5-3\ b\ n_s + 4\ b} = \frac{64\ L\ a\ 4^{2\ b - b\ n_s}\ Q^{2\ b - b\ n_s + 2}\ \rho^{1+b}}{k_c^{b\ n_s} \left(\dfrac{3\ n_s + 1}{4\ n_s}\right)^{b_e\ n_s} \pi^{2b - b\ n_s + 2}\ 8^{b\ n_s - b} \Delta P}. \quad [11]$$

This relationship, established from approximations, was simple to resolve and was proved to be pertinent (accuracy < 0.5%) by comparison with the numerical resolution of equation 8 using Newton's method with the Colebrook equation for the calculation of Da (Alvarez, 2003).

Values for ρ (kg/m^3) and dynamic viscosity (μ; Pa · s) were calculated for each fluid to take into account variations of temperature and concentrations.

For water and nitric acid, with 20 < T (°C) < 130,

$$\rho = 1{,}001 - 9.084 \times 10^{-2}\ T - 3.416 \times 10^{-3}\ T^2 \text{ and} \quad [12]$$

$$\ln(\mu) = -6.363 - 2.8 \times 10^{-2}\ T + 9.874 \times 10^{-5}\ T^2. \quad [13]$$

For caustic soda, with 0 < T (°C) < 150 and 0 < C (% mass) < 10,

$$\rho = 1{,}001.3 + 11.77\ C - 1.7941 \times 10^{-2}\ C^2$$
$$+ (-8.3446 \times 10^{-2} - 3.9428 \times 10^{-2}\ C + 9.3318$$
$$\times 10^{-4}\ C^2)\ T + (-3.4666 \times 10^{-3} + 2.8429 \times 10^{-4}$$
$$C - 7.727 \times 10^{-6}\ C^2)\ T^2 \text{ and} \quad [14]$$

$$\ln(\mu) = -6.3563 + 5.725 \times 10^{-2}\ C + 5.984 \times 10^{-4}$$
$$C^2 + (-2.836 \times 10^{-2} - 1.877 \times 10^{-4}\ C + 7.75$$
$$\times 10^{-7}\ C^2)\ T + (1.055 \times 10^{-4} + 9.134 \times 10^{-6}$$
$$\times\ C - 6.12 \times 10^{-7} \times C^2)\ T^2. \quad [15]$$

Values for C_{NaOH} and C_{HNO3} were evaluated using equations 3 and 4. The temperatures at the pressure sensor limits were calculated considering a linear profile of the temperature all along the heating zone; ρ and μ were then average values between the entrance and outlet of the heat exchanger.

During the chemical cleaning phases and water rinses, α was then calculated from D values. Because the characteristics of the cream (ρ, k_c, n_s) and mixtures of water and cream were not known at high temperature, α could not be calculated with accuracy during production and during the phases during which cream and water were mixed (end of launching, beginning of the flushing–water rinse phase).

End of Operation

The flushing–first rinse phase was considered to be finished when the matter (cream, deposited foulants) was no longer removed; the arbitrary threshold was DM <1.0 g/kg.

The intermediate rinse phases ended when the detergent concentration was lower than an arbitrary threshold set to 100 mg/kg corresponding to an on-line conductivity ≤1.1 and <1.4 mS/cm for NaOH and HNO$_3$, respectively. In this extreme case, the neutralization of the acid or alkaline for the forthcoming cleaning would lead to a very low decrease in concentration (0.8 and 0.2%, respectively). For safety reasons, the final rinse phase was considered to be finished when $\chi \leq 0.91$ mS/cm, corresponding to values close to the water conductivity, and $C_{HNO3} < 20$ mg/L. This criterion was validated by pH measurement indicating values higher than 5.8 (i.e., in the range of water for human consumption; 4.5 < pH ≤9.0).

Table 2. Appropriate sensors used to determine the end of each phase and the sorting of the fluids during the cleaning sequence[1]

Phase of cleaning	End of phase	Sorting
Flushing–first rinse phase	Conductivity (turbidity)	Conductivity (turbidity)
NaOH	ΔP, α, C_{COD}	
HNO_3	α, calcium, C_{COD}	
Water rinses (intermediate and final)	Conductivity	Conductivity

[1]ΔP = pressure decrease; α = fouling rate; C_{COD} = concentration in chemical oxygen demand.

During the chemical cleaning phases, the calculation of D did not allow quantifying the cleanability of the exchanger because of a lack of accuracy (60 μm). However, α was used to evaluate the efficient cleaning time corresponding to $\alpha \leq 0.5\%$. Moreover, the acid cleaning was considered to be finished when calcium concentration was $<36.5 \pm 2.0$ mg/kg.

Table 2 summarizes the appropriate sensors used to determine both the sorting of the fluids and the end of each phase during the cleaning sequence.

Masses, Volumes, and Concentrations of Effluents

For the launching phase, the effluent DM was calculated from the turbidity measurement, and for the flushing–first rinse phase, it was evaluated from the conductivity measurements. The DM values could be expressed in C_{COD} (g/kg) from

$$C_{COD} = \frac{DM}{1.13}. \qquad [16]$$

Values for C_{NaOH} and C_{HNO3} were calculated from the conductimeter (equations 3 and 4). Regardless of the phase, mass (kg) and volume (m^3) losses during the cleaning were calculated by numerical integration according to the trapeze method ($\pm 2\%$).

Analyses

The conductivity ($\pm 2\%$) was measured at 20°C with a 6660 conductimeter (Metrohm AG 9101, Herisau, Switzerland) and the turbidity ($<10\%$) with a turbidimeter (Hach 2100 AN IS, Namur, Belgium; range = 0–10,000 nephelometric turbidity units). Density ($\pm 1\%$) was measured from 20 to 50°C using a densitometer (DM A48 AP Paar, Roucaire, Rennes, France), and dynamic viscosity of Newtonian fluids ($\pm 1\%$) was obtained with a D8 Haake viscosimeter (Karlsruhe, Germany). The DM (<0.5 g/kg) was measured after drying at 105°C for 7 or 15 h. The C_{COD} ($\pm 2\%$) was measured by Nanocolor Test 29 cuvettes and a PF 10 pocket filter photometer (Macherey-Nagel, Hoerdt, France). This method, rapid and requiring a small volume of sample (2 mL), was correlated with the results given by AFNOR (1988) (Dresch, 1998). Calcium concentration was measured by atomic absorption spectrometry (Varian AA 300, Les Ulis, France) according to Brulé et al. (1974). Titration of NaOH and HNO_3 ($<2\%$) was performed with HCl (1 N) and NaOH (1 N) according to 2 methods: 1) titration acid-base with phenolphthalein, and 2) titration of pH on a dosage bench (Schott Geräte TR155, TA20 and T100, Hofheim, Germany; electrode = INGOLD 90 433, Mettler-Toledo, Urdorf, Switzerland).

The frequency of sample withdrawal varied from 10 s to several minutes according to the phase kinetics. Two industrial experiments were performed to analyze the different phases of the cleaning sequence and to propose revisions, and 2 more were performed to validate the improvements.

RESULTS AND DISCUSSION

Critical Analysis of the Monitoring of the Sterilizer CIP

No information is given about the launching and production phases because the industrial operators did not choose to change any operating parameters because the changes might have altered the final cream characteristics. Nonetheless, the differential pressure sensor gave relevant information about fouling evolution during the production phase.
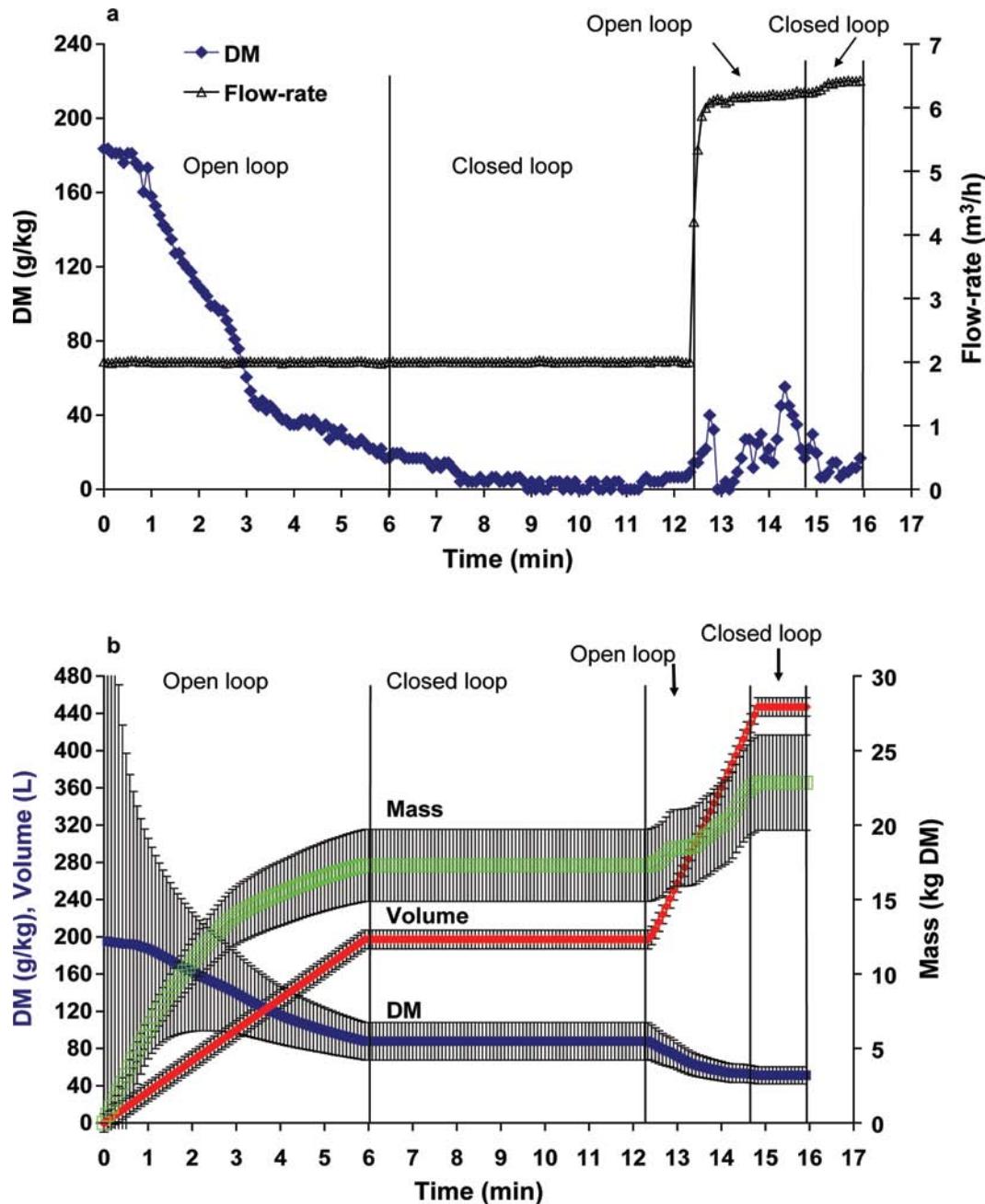
Flushing–First Rinse Phase

The flushing–first rinse phase was carried out in 2 cycles, both performed successively in open and closed loops, the second cycle being performed at a high flow-rate (6 instead of 2 m^3/h) (Figure 2a; Table 1). During this phase, DM sharply decreased during the first minutes and leveled off for $7.5 <$ time (min) < 12.3. After 12.3 min, the increase of Q and then of the shear stress at the sterilizer surface favored the soil removal and the increase in DM, as previously noted by Thor and Loncin (1978) and Tissier et al. (1999). A subsequent increase of waste DM was observed. The threshold at the end of this phase, set to 1 g/kg by the

ALVAREZ ET AL.

industrial operator, was not reached, suggesting that rinsing time (duration and waste volume) should have been increased. The matter losses during these 2 phases were evaluated at 22.5 ± 3.0 kg of DM in 450 ± 15 L of effluent (Figure 2b; Table 1).

The critical analysis of this phase showed 3 possible improvements. 1) The treatment of the conductivity enabled the sorting of the fluids for further valorization. For example, sorting the fluids at 13.6 min can lead to the separate collection of approximately 320

L of effluent with an average DM of 60 g/L and 130 L of effluent with an average DM of 29 g/L (Figure 2b). Moreover, knowing the acceptable water content of the end product, a small but not negligible quantity of little-diluted cream recovered during the very beginning of the phase could be reintroduced in the production line. 2) The DM information showed that it could be interesting to increase the flow-rate right at the beginning of this step. Moreover, the final part of the first rinse phase, operated in a closed loop, could be

Figure 2. The flushing–first rinse phase: a) flow-rate and DM with time; b) matter losses, volume, and DM of the discharged effluent. Color version available in the online PDF.

changed to an open loop because the beginning of the first alkaline cleaning phase that follows immediately was operated that way. To choose adequate parameters (flow-rate, running time, open or closed loop) for this phase (DM <1 g/kg), the balance between energy consumption and saved time should then be estimated on the basis of newly collected experimental results. 3) Instead of increasing the duration of this phase to reach the industrial goal of DM <1 g/kg, the effect of small amounts of matter on the cleaning efficiency of NaOH should be studied. For example, stopping the operation at 12 min (instead of 16 min) would lead to the addition of 13 kg of DM in the NaOH solution (DM collected from 12 min to the end of the water rinse phase; i.e., 16 min) and then to an increase of C_{COD} in the NaOH solution (from 7.4 g/kg to 22.4 g/kg in the studied case). Such added pollution may be not detrimental to the NaOH solution efficiency because the presence of fat and proteins solubilized in the caustic soda solution was previously shown to decrease surface tension characteristics of the solution, leading to interesting cleaning properties of the detergent solution (Alvarez et al., 2007; Gésan-Guiziou et al., 2007).

Chemical Cleaning Phases. The chemical cleaning procedure consisted of 4 phases. At the beginning of each phase, the concentrated detergent was directly injected into the feed tank. The first 4 min, operated with an open loop, yielded approximately 450 L of effluents that was disposed of at the purification station (Table 1).

During the NaOH-1 phase, a significant pollution load (C_{COD} = 3–10 g/kg), not removed by the flushing–first rinse operation, was expulsed with water from the equipment (Figure 3). However, for the first 4 min of the NaOH-2 and acid phases, the effluent was mainly clean water (results not shown); the first 4 min could therefore have been operated in a closed loop so as to save 450 L of water per phase, corresponding to a total amount of 1.35 m^3 of water per cleaning. After 5 min, the flow of the chemical cleaning solution was detected through the sudden increase of C_{NaOH} or C_{HNO3} (equations 3 and 4), followed by oscillations resulting from the sequential injection of the concentrated detergent in the feed tank (Figures 3 and 4a). The oscillations, the period of which was equal to the mean residence time in the closed loop (approximately 5 min), were attenuated by progressive mixing of the detergent in water contained in the closed loop. During the first 2 min of the NaOH-1 phase (4–6 min after the beginning of the phase), ΔP (Figure 3) and α (Figure 4a) increased simultaneously with the increase of OH$^-$ ions in the heating zone of the sterilizer. Such an increase was a result of the swelling of the deposit in contact with caustic soda, as previously reported for a UHT

plate sterilizer of chocolate dessert cream (René et al., 1988) and for various plants heat-treating dairy fluids (Gallot-Lavallée and Lalande, 1985; Gallot-Lavallée et al., 1988; Bird and Fryer, 1991; Gillham et al., 1998). Then, ΔP and α decreased progressively, showing that cleaning continued, and stabilized after approximately 10 min of cleaning (Figures 3 and 4a). The turbidity signal was difficult to use or interpret because it fluctuated with time (Figure 3), even though it attenuated after 10 min. The cleaning did not seem to be efficient during the following 8 min (average α = 5.1 ± 0.3%; Figure 4a; Figure 3) and could have been be shortened.

During the HNO$_3$-1 phase, α decreased after approximately 3 min of cleaning (average α = 2.8 ± 0.5%; Figure 4a), as reported by Corrieu et al. (1986) with a UHT sterilizer fouled with milk. The fast cleaning rate was confirmed by the fast calcium removal, the concentration of which become equal to that of the water after 5 to 6 min (e.g., approximately 1 min after the flow of the first H$^+$ ions; Figure 5). The overall amount of removed calcium was around 270 g. These results were in agreement with Van Asselt (2002), who shows that the overall amount of calcium contained in the fouling of a tubular sterilizer of whole milk is removed after less than 5 min. Thus, the acid cleaning duration could have been sharply reduced from 18 min to 6 to 7 min.

During the NaOH-2 phase and regardless of the cleaning time, the maximum C_{COD} value was very low (less than 40 mg/kg), with an average value of 10 mg/kg (results not shown). Because the removed fouling amount was approximately 250 times lower than that measured during the NaOH-1 phase, the necessity of the NaOH-2 phase was questionable. It was likely that the small existing fouling would not alter the microbiological safety of the end product because it probably stayed in the end of the heating section where the temperature was more than 100°C. Nonetheless, a risk is still possible, and the residual deposit could act as an agent for future fouling. If this phase has to be retained, its duration should be significantly reduced from 18 to 10 min as for the NaOH-1 phase.

During the NaOH-2 phase, the average α level at the end of the cleaning (α = 7.4 ± 0.5%) was higher than after the NaOH-1 and HNO$_3$-1 phases (Figure 4a; Table 1). No simple explanation could be given with regard to residual fouling because the sterilizer could be considered to be clean at the end of the first alkaline and acid phases. The same trend was observed with water at the end of the alkaline rinses (intermediate rinses; Figure 4a). There are then unexpected variations of α when there is a change of fluid in the sterilizer. One can suppose that the differential pressure sensor or the removal of the fouling layer could be sensitive to a change in
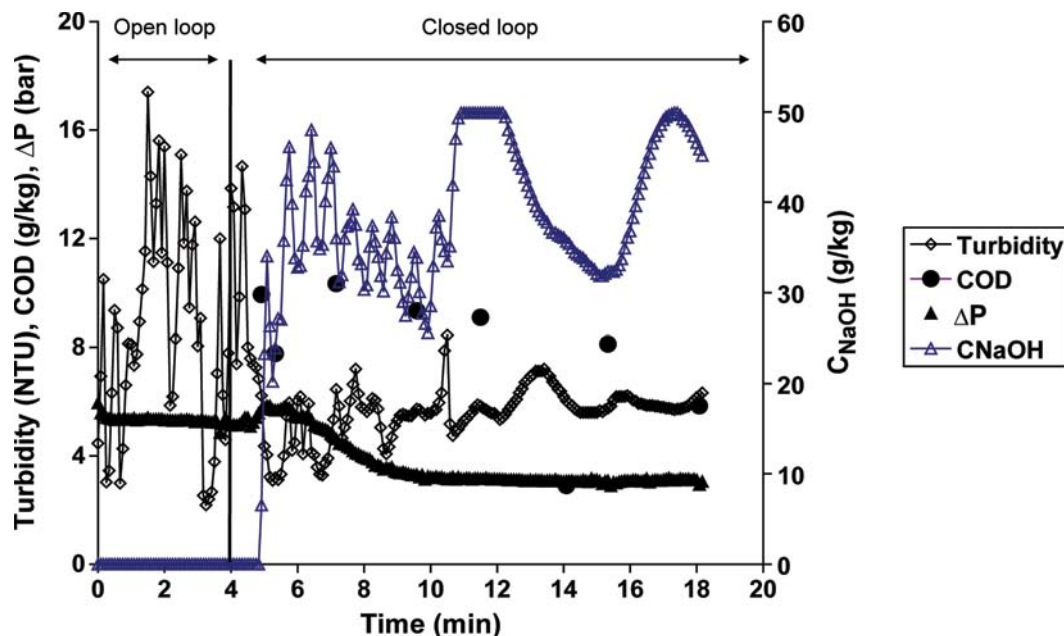
Figure 3. The first alkaline cleaning phase: turbidity (in nephelometric turbidity units), chemical oxygen demand (COD), concentration of caustic soda (C_{NaOH}), and pressure drop in the heating zone of the sterilizer (ΔP) with time. Color version available in the online PDF.

operating conditions (e.g., temperature, flow-rate, and physical and chemical characteristics of the fluid) or a change in physical and chemical characteristics of the fouling materials extracted from the sterilizer surface. It was likely that, because of the effect of OH- ions or even water, the residual deposit hydrates and swells as shown previously during the NaOH-1 phase but without being totally removed during the NaOH-2 phase. These observations are in agreement with the results and discussion reported by Perlat et al. (1986), who observed an increase of the fouling rate of the heating zone of a plate UHT sterilizer fouled by milk from the very beginning to the end of the alkaline cleaning. Such a phenomenon was observed only when NaOH was the chemical reactant used for the first phase of cleaning (Perlat et al., 1986); in case of an alkaline cleaning operated after an acid cleaning, the phenomenon of swelling was not observed, contrary to our results.

The HNO_3-2 phase was similar to the HNO_3-1 phase. The calcium concentration (maximum 500 mg/kg, compared with 4,500 mg/kg in the HNO_3-1 phase) became equal to that of the water from 5 to 6 min and α decreased rapidly to reach a stable level of 0.5 $\pm$ 0.3% (Figure 4a; Table 1). The total amount of calcium removed, around 17 g, was 16 times less than that removed during the HNO_3-1 phase. The current duration of 18 min could have been decreased to 6 to 7 min.

Rinsing of Detergent. During the intermediate rinses, the detergent concentration sharply decreased after 4.6 $\pm$ 0.3 min and 4.8 $\pm$ 0.5 min for NaOH and

HNO_3, respectively (Figures 4a and 6). It then leveled off, indicating that the flushing operation was over. Regardless of the operation, the critical detergent concentration of 0.1 g/kg was reached after less than 11 min (Figure 4a). For the final water rinse (results not shown), an acid concentration lower than 20 mg/kg (0.91 mS/cm) was reached after less than 9 min, indicating that the duration of the final rinsing phase could have been significantly reduced.

The current management of the intermediate and final rinsing steps generated losses evaluated at approximately 1,500 L of water (Table 1) containing 20 kg of caustic soda and 14 kg of nitric acid. Reducing the rinsing durations would reduce the volume of effluents, and the recovery of the detergent solution could also be realized. As for the flushing–first rinse phase, the effluent could be separated into 2 fractions by using the conductivity sensor signal; at the very beginning, a diluted detergent solution could be recovered. The lower the sorting threshold of caustic soda concentration, the higher the dilution and the volume of recovered detergent, the stronger the readjustment of detergent concentration, and the bigger the storage capacity required. The other fraction to be recovered (very diluted detergent solution) could be either disposed of and sent to the purification station or recovered as flushing–first rinse water, but in that case, it would be necessary to control the sorting of the cream from launching water so as to avoid any traces of detergent in the manufactured product.
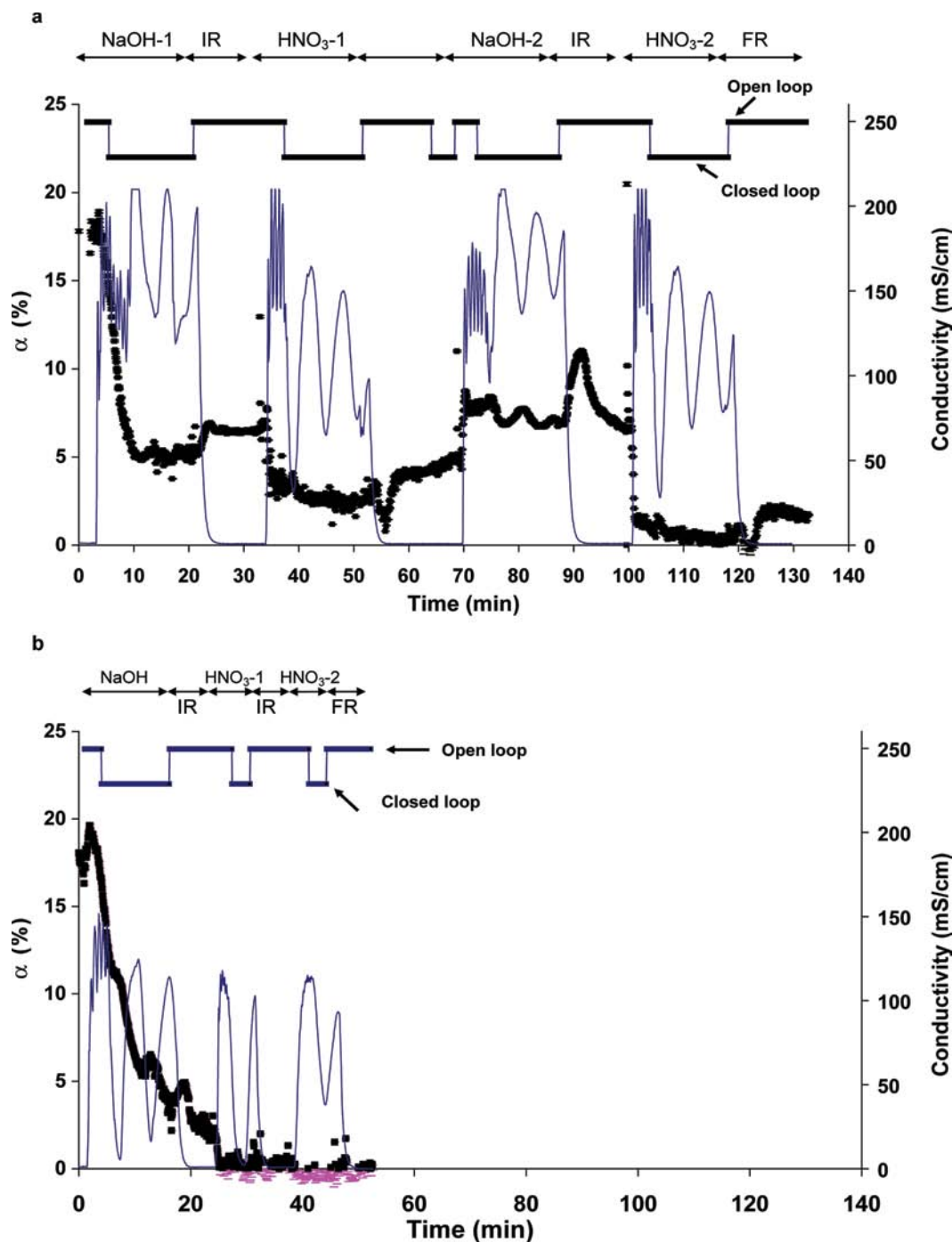
Figure 4. Sequence of rinsing and cleaning phases during a) the initial industrial procedure and b) the revised sequence [line = conductivity; square = fouling rate (α); IR = intermediate rinse; FR = final rinse]. Color version available in the online PDF.

Industrial Validation of Revised Sequence

The preceding critical analysis of the initial industrial cleaning sequences made it possible to propose completely revised sequences. Among all the modifications suggested, many of them were accepted by the industrial operators. Their relevance was then quantified and checked on an industrial scale. The operating conditions of each phase and the volume of the effluents disposed of to the purification station during both sequences are reported in Table 1.

Synthesis of the Proposed Improvements. The new parameters of the revised sequences to be checked were as follows (Table 1). For the flushing– water rinse
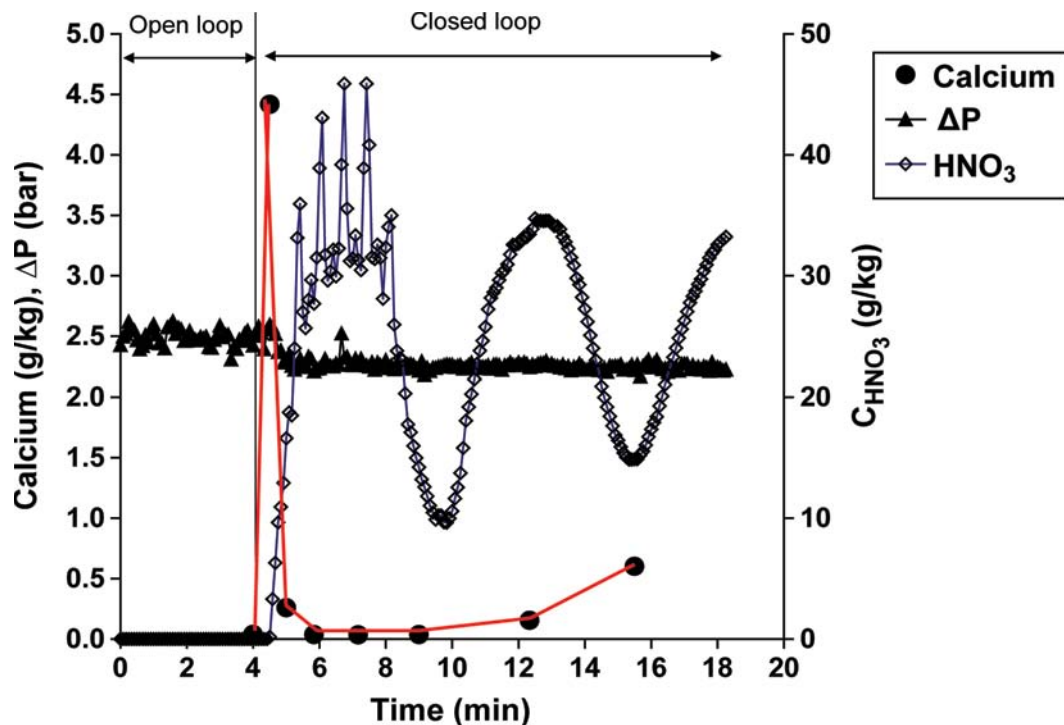
Figure 5. The first acid cleaning phase: calcium content, concentration of nitric acid (C_{HNO3}), and pressure drop in the heating zone of the sterilizer (ΔP) with time. Color version available in the online PDF.

phase, the overall duration of the phase remained unchanged and the last step, originally operated in a closed loop, was operated in an open loop. The duration of the NaOH-1 phase was reduced from 18.2 ± 0.2 to 16.1 ± 0.2 min. For both the HNO_3-1 and HNO_3-2 phases, the initial duration of 18.2 ± 0.2 min was reduced to 7.2 ± 0.5 min and the first 4 min were operated in a closed loop. The NaOH-2 phase was suppressed with the suppression of the following intermediate rinsing. Because the NaOH-2 phase was suppressed, the HNO_3-2 phase was likely useless, but the industrial partner decided to retain it. Regarding intermediate and final rinses, the automatic program was adapted to control the end of operation on-line with the conductivity sensor. For safety reasons, a calculated delay was inserted for controlling the minimum and maximum duration of the operation so as to mitigate any dysfunction of the sensor. The durations of the intermediate rinsing steps after NaOH and HNO_3 cleanings were reduced from 12.5 ± 0.3 min and 16.8 ± 0.7 min, respectively, to 7.0 ± 0.3 min for each. The duration of the final rinsing was reduced from 14.0 ± 0.2 min to 7.8 ± 0.2 min. The conductivity and pH at the outlet of the plant were consistent with those of drinking water ($\chi \leq 1{,}000$ μS/cm; $4.5 < pH \leq 9.0$).

Efficiency of the Industrial Revised Sequence. The gains induced by means of the industrially revised sequence were considerable in duration and overall ef-

fluents volume, both divided by 2 (Figures 4a and 4b; Table 1). The losses of NaOH and HNO_3 were reduced with the revised sequence: 11 ± 3 compared with 43 ± 4 kg of DM and 16 ± 3 compared with 28 ± 1 kg of DM for NaOH and HNO_3, respectively.

With the revised cleaning sequence (operated twice for the current study at industrial level), the α at the end of the final rinsing ($\alpha = 0.0 \pm 0.2\%$) was equal to or lower than initial α determined during the launching phase (2.8–$5.4 \pm 0.6\%$) and much lower than α reached after the production phase. These low α values demonstrated the high efficiency of the revised sequence. The NaOH-1 phase removed the major part of fouling ($0.5 \leq$ the fouling fraction removed by a phase ≤ 0.9), and the residual fouling fraction was removed with the subsequent acid cleanings (HNO_3-1 and HNO_3-2 phases). When the NaOH-2 phase was not operated, the HNO_3-2 phase removed no fouling fraction, confirming the usefulness of that phase (Table 1).

Compared with the initial sequence, α at the end of the final rinsing was smaller in the revised sequence ($\alpha = 0.0 \pm 0.2\%$ compared with $1.5 \pm 0.3\%$; Table 1; Figure 4); this was unexpected because the revised sequence included only 1 shortened alkaline cleaning. This difference was not the outcome of a different initial level of fouling (similar at the end of the first rinsing phase) or of different thermal and hydrodynamic cleaning and rinsing conditions, but originated from a gap
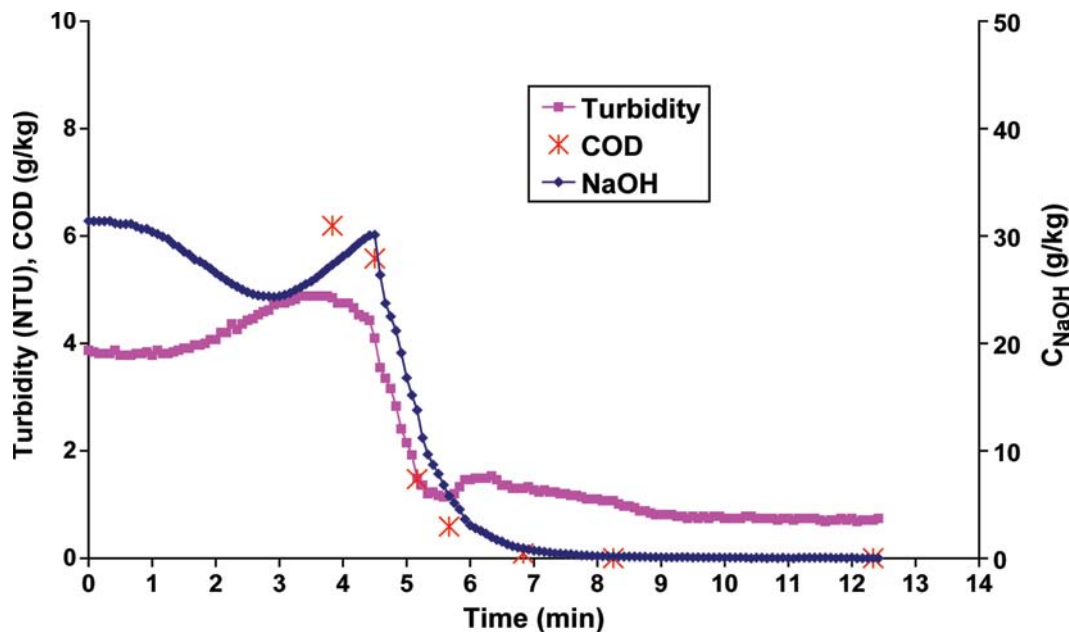
Figure 6. The first intermediate water rinse after the first alkaline cleaning phase: turbidity (in nephelometric turbidity units), chemical oxygen demand (COD), and concentration of caustic soda (C_{NaOH}) with time. Color version available in the online PDF.

in detergent concentrations. These concentrations were much smaller for the revised sequence owing to large concentration variations of mother solutions injected. For NaOH, the concentration was in the range of 0 to 30 g/kg versus 0 to 50 g/kg for the initial sequence; for HNO_3, the concentration was in the range of 0 to 25 g/kg versus 0 to 45 g/kg for the initial sequence. Such differences could generate significant differences of efficiency. According to Timperley and Smeulders (1988), the cleaning duration of a deposit of whole milk (72°C) is multiplied by approximately 2 when alkaline detergent increases from 30 to 50 g/kg. According to Bird and Bartlett (1995), the cleaning duration of stainless steel tubes fouled with whey (97°C) is multiplied by 5 when the caustic concentration increases from 5 to 20 g/kg. This trend was also observed after the alkaline rinsing: the average caustic concentration was close to 20 g/kg in the revised sequence compared with 34 g/kg in the initial sequence and this leads to a fouling rate (α) 2 times smaller with the revised sequence. This leads to the idea that the removal of a larger fraction of fouling by the alkaline cleaning at lower concentration made easier the access of H^+ ions to the wall, where the deposit, mainly inorganic, was eliminated by the acid action. This kind of reasoning, based upon the average concentration, applied to the highly fluctuating values of concentration within the sterilizer.

Despite the satisfactory industrial results given by the revised sequence, further improvements, listed as follows, might still be made. 1) For improving cleaning,

the caustic concentration should be controlled carefully after having experimentally defined the optimal value. 2) The HNO_3-2 phase is useless and can be removed. 3) The order of cleaning sequences should be validated in terms of cleaning kinetics and efficiency. Indeed, Perlat et al. (1986) reported that for a UHT sterilizer of skim milk, the cleaning duration of the preheating zone (80–120°C) was 2 times faster when the acid cleaning was operated before alkaline cleaning; moreover, only the order acid-base was efficient for cleaning the heating zone (120–140°C). The enhanced cleaning rate with the order acid-base was also demonstrated by Jeurnink and Brinkman (1994) for cleaning a tubular evaporator (70°C) fouled with whey. 4) The relationship between sterilizer fouling level (related to production duration) and chemical cleaning rate and duration should be studied. It would help control the combination of production duration and cleaning duration for better UHT sterilizer management. 5) Supplementary savings of matter should be validated by a more adequate management of the sorting and use of fluids (e.g., methane bioproduction, external substrate of denitrification, and so on; Sage, 2005). The experiment requires a new plant for recovering the fluids, which was not industrially possible in the present study.

In this study, alkaline cleaning was realized according to single-use CIP mode. During cleaning, NaOH is polluted with suspended solids and soluble compounds (Figure 3). It would be of interest to study alkaline phase kinetics and duration by using reused NaOH

treated by microfiltration (so as to eliminate suspended solids) to quantify the effect of NaOH solutions with lower surface tension as shown by Alvarez et al. (2007) for UF membrane cleaning.

Strategy for Rationalizing Cleaning

This work opens the way to a general procedure for the rational management of CIP, which requires 4 main steps. To propose improvements, the first compulsory step consists of knowing the general scheme of the plant and the geometrical process characteristics from the CIP tanks to the CIP backflow and the waste valves. This knowledge eventually allows the fixing of equipment design mistakes that have created dead-zones sources resulting in rinsing and cleaning difficulties, and the determination of the time scales to be synchronized and the volumes involved in the plant.

In the second step, the appropriate sensors or tracers and their signal treatment methods for each phase of the process must be selected. Two main sensors should be exploited: the turbidity sensor, which allows the end of water flushing–first rinse phases to be determined and could also be useful for operating the sorting of fluids during the launching phase, and the conductivity sensor, which determines the time of the end of water flushing–first rinsing phases as well as the rinsing phases. It could also be appropriate for sorting both the cream during the launching and first rinse phases and the cleaning solutions (NaOH, HNO$_3$) during the rinses. Both sensors are important for knowing the nature and the concentration of each fluid flowing in the equipment in real time, the knowledge of which opens the way to a better control of the process. Special attention should be paid to the treatment of the signal of the pressure-difference sensor, which quantifies the fouling rate throughout the process. It quantifies the state of the sterilizer and the cleaning efficiency at any processing time, in particular at the beginning and the end of each phase and of the entire sequence. The off-line analyses of both C_{COD} and calcium concentrations are also useful for evaluating the amount of fouling matter removed from the food plant surface by the alkaline and acid cleanings, respectively.

In the third step, the database must be designed. This allows all the various data collected from the sensors or tracers during the different phases (launching, production, cleaning, rinsing) to be acquired. The adequate selection of the acquisition frequency must be adapted to each phase and to each step of the phase according to its duration and to the kinetics of concern.

The final step consists of exploiting the data according to the calculation modes selected for synchronizing the time scales; calculating the parameters useful for the evaluation of fouling, rinsing, and cleaning kinetics; and determining the operation ends and the sorting of the fluids mixtures and cumulated volume, effluents load. Such data exploitation allows an on-line management to be implemented that performs better than a temporization. Also, the data is the source of ways in which the production and cleaning modes of the equipment can be improved.

CONCLUSIONS

The duration of each phase of the cleaning sequences of industrial plants and equipment is often set by temporization with excessive empirical safety margins. This study shows that critical analysis of the management of an industrial plant (a sterilization plant that processes dessert cream, coupled with a single-use CIP plant) can lead to the definition of appropriate tools (sensors, tracers, data treatment) to help CIP designers and food industry operators manage daily cleaning operations more efficiently. In particular, the on-line measurement of turbidity and conductivity; the determination of C_{COD}, C_{NaOH}, and C_{HNO3}, and calcium content; and the calculation of fouling rate from the signal of the pressure difference sensor installed in the heating zone enable the determination of the effective end of operations and the sorting of fluids for further use from those sent to the purification station.

The critical analysis of the industrial sterilizer single-use CIP opened the way to the revision of the whole sequence, which led to the reduction of the total duration of the process the the reduction of effluents produced by more than half. Because of the efficiency of production and the quality of the sterilized cream, this new procedure has been used for more than 5 yr and similar approach has also been successfully applied for managing the operation of a skim milk plate pasteurizer (Chareunphol, 2004).

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