

## Optimization of the cip system rinse stage for effluent reduction

Otimização da etapa de enxágue de sistema cip para redução de efluentes

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### Resumo

*O leite é um dos principais alimentos e possui considerável riqueza nutritiva. Nesse sentido, o consumo de produtos alimentícios industrializados tem aumentado significativamente e com isso é importante conhecer e melhorar os processos de higienização da indústria. A limpeza no local, clean in place (CIP), é uma tecnologia útil para limpeza de equipamentos e tubulações, pois evita tanto a parada da produção por longo período quanto a desmontagem dos mesmos e é composta de diversas etapas, como limpeza com detergente alcalino, enxágue, aplicação de ácido, enxágue, sanitização e enxágue. Neste trabalho foi avaliado comparativamente o consumo de água e a consequente geração de efluente na etapa de enxágue do detergente alcalino utilizando vazão constante e vazões pulsadas e ainda proposto um ajuste ótimo do valor da vazão pulsada para minimização do consumo de água e do efluente gerado. Os ensaios foram conduzidos em um protótipo de sistema CIP e executados com base em um planejamento composto central (PCC) alterando as variáveis: amplitude, período e duração da etapa de maior valor de vazão. Utilizou-se a técnica de superfície de resposta para avaliar os efeitos de cada variável sobre o consumo de água. Os resultados experimentais obtidos mostraram que existe uma condição ótima para operação do enxágue de forma pulsada com amplitude 1,5 L/min, período 138 segundos e duração da etapa de maior valor de vazão de 53 segundos. Além disso, verificou-se que o enxágue com vazão pulsada produz uma economia de aproximadamente 14,52% em relação a operação a vazão constante.*

**Palavras-chave:** CIP; Enxágue; Vazão pulsada; Efluentes; Superfície de resposta; Otimização

### Abstract

*Milk is one of the main foods and possesses considerable nutritional richness. In this sense, the consumption of industrialized food products has increased significantly and with this it is important to know and improve the processes of hygiene of the industry. Clean in Place (CIP) is a useful technology for cleaning equipment and pipes because it avoids dismantling and is made up of several steps, such as cleaning with alkaline detergent, rinsing, applying acid, rinse, sanitize and rinse. The objective of this work was to evaluate the water consumption and the consequent generation of effluent in the alkaline detergent rinsing stage using constant flow and pulsed flow rates and also to propose an optimal configuration of the pulsed flow to minimize the consumption of water and effluent generated. The tests were conducted in a CIP system prototype and executed based on a central composite planning (CCP) changing the variables amplitude, period and duration of the high part. The response surface technique was used to evaluate the effects of each variable on water consumption. It was verified that there is an optimum condition for the operation of the rinse in pulsed form with amplitude 1.5 L / min, period 138 seconds and duration of the high part of 53 seconds. In addition, it has been found that the rinse with pulsed flow produces an economy of approximately 14.52% in relation to the constant flow operation.*

**Keywords:** CIP; Rinse; Pulsed flow; Effluents; Response surface; Optimization

## 1 Introduction

Milk is one of the main foods consumed in the world. Highlighting its nutritional richness and its nutritional load due to the proteins, minerals and vitamins present in its composition. Dairy products, consumed in liquid form or in the form of their derivatives, such as cheese, yogurts, butters and fermented dairy products, have an important contribution to the international agricultural trade (SOUZA, *et al.* 2015).

In Brazil, the amount of raw milk purchased, cooled or not, in the first quarter of 2018 totaled approximately six billion liters. Of this production, about 99.9% of the volume is processed in 1,917 establishments inspected at the municipal, state or federal level. Also in the first quarter of 2018 the state of Minas Gerais is the largest dairy producer, followed by the states of Rio Grande do Sul and Goiás (IBGE, 2018).

Dairy products are sensitive to heating, which requires care to avoid changes in processed products. Proteins can coagulate and embed into the surfaces of hot equipment or tubing used to heat milk (BYLUND, 2003). Consumption of processed food products has been growing steadily in recent years. In this sense, the food industry is concerned with the quality of the final product and its preservation after processing (BANSAL; CHEN, 2006).

Industrial production requires on-site cleaning systems to remove soils through the circulation of chemicals. This on-site cleaning technology, known by its acronym (CIP), is automatic and does not require the dismantling of equipment and pipes to promote hygiene. Industries that rely heavily on CIP are those that require high levels of hygiene, such as food, beverages and pharmaceuticals. (ANDRADE, 2008; FRYER *et al.*, 2006). However, CIP systems require significant quantities of water, detergent and energy. In addition, CIP can generate large quantities of effluents, with considerable costs for the treatment and disposal of this industrial waste, and also provoke an environmental burden for society (LYNDGAARD *et al.*, 2014).

In the food industry, the sanitation process comprises cleaning and sanitizing steps. Cleaning consists of pre-washing with water to remove dirt, followed by the use of chemical agents such as alkaline detergents, usually sodium hydroxide (NaOH) with concentration of 0,5 or 1 wt%. In addition, the use of acids to remove organic and mineral residues from equipment surfaces (ANDRADE; MACÊDO, 1996). At the end of the application of the alkaline detergent and at the end of the use of the acid the equipment and / or tubing for the removal of chemical residues is rinsed (ANDRADE, 2008).

The dairy effluent has pH between 2 and 12, as a result of the use of acid and alkaline detergents to clean the processing plants. Low and high pH values interfere with the activity of microorganisms that break organic pollutants in the biological treatment stage of the sewage treatment plant, transforming them into sludge (BYLUND, 2003). Considering the limitation of freshwater on the planet, increasing water consumption to meet human, agricultural and industrial demand and the concern to reduce environmental impacts by the food industries, it is important to adopt strategies to rationalize water use and to minimize the generation of effluents (FIESP/CIESP, 2006).

In this way, minimizing the consumption of water and consequently the environmental effects, is a critical point for the industry. Therefore, evaluating the steps of the CIP and investigating alternative ways of cleaning should be constant efforts to significantly evolve in the quest for sustainability (CARRERA, 2015). In this respect, understanding the cleaning of dairy plants and optimizing the operating conditions applied in CIP can reduce the consumption of water, energy and detergents, while maintaining process hygiene (FRYER E ROBBINS, 2005; WILSON, 2005).

To this end, the simulated-use tests seek to represent the processing conditions typical of the food industry in the laboratory. To accomplish this task, it is necessary to develop methodologies and equipment to simulate the various conditions and stages present in the industrial hygiene procedures (ANDRADE, 2008). Thus, the optimization of the various factors affecting the pulsed rinse is fundamental to design an efficient hygiene in the CIP system. The shear stress and the reactions between the fluid flow and deposits formed are important in removing incrustations (GEORGIADIS *et al.*, 1998). The use of

pulses in the flow slowly when imposed to a steady flow of liquid increases the shear stresses impelled to a surface and improves the cleaning (AUGUSTIN e BOHNET, 2001; GILLHAM *et al.*, 2000).

The most efficient way of evaluating the influence of variables and how their interactions affect the volume of water consumed in a CIP process is through statistical experimental designs. Response surface methodology (RSM) is a useful statistical tool to study the effect of different factors and the impact of their interactions on the response of interest and to effectively determine the levels of the variables for the optimal region of the response (KUMARI, 2014).

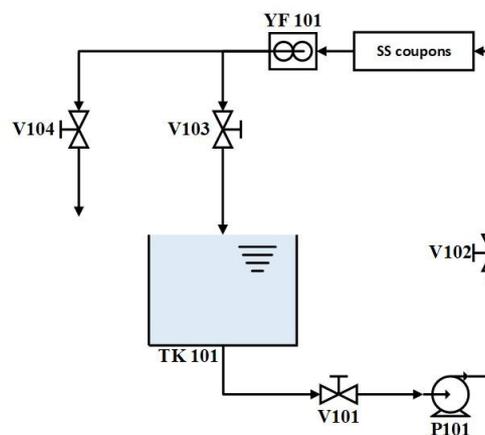
This technique has been widely used in the modeling and optimization of treatment of industrial and urban effluents (TAK *et al.*, 2015; THIRUGNANASAMBANDHAM *et al.*, 2015; PI *et al.*, 2015; NAVES *et al.*, 2017). Central composite planning (CCP) is one of the most used configurations for the design of the response surface, since it is capable of generating nonlinear objective functions as complete quadratic models. Planning is an array of responses for experimental conditions composed of three groups of points: factorial combination of the variables with  $2^k$  experiments, added to  $2k$  axial points and  $n$  central points, where  $k$  is the number of controlled variables present in the research (MONTGOMERY, 2009; LI *et al.*, 2015; NAVES *et al.*, 2017).

In this context, the objectives of this research were: (1) to evaluate the amount of water spent in the alkaline detergent rinsing stage using a CIP prototype using constant flow rates compared to the consumption of water for rinsing by means of pulsed flows, (2) to develop a mathematical expression for the volume of water spent as a function of the variables in the pulsed flow and (3) to propose parameters for pulsed rinsing using the surface response technique to achieve greater efficiency in the use of water and thus reduce the effluent load generated in the rinsing stage of dairy units.

## 2 Material and methods

The experiments were conducted using the system shown in Figure 1. The prototype represents a typical system for processing milk and derivatives, formed by a reservoir of approximately 25 L,  $\frac{1}{2}$  HP centrifugal pump, turbine type flow sensor, 1.2 meter pipe length for transporting material and 304 stainless steel cylindrical test coupon, polishing # 4, length 15 cm and diameter  $\frac{1}{2}$  "in diameter; electronic data collection system, electronic conductivity meter, Gehaka pHmeter and a microcontroller from the Arduino family. It was used as cleaning solution, alkaline detergent, sodium hydroxide (NaOH) with concentration 0.5 wt% and potable water for system rinsing. The experiments were carried out in Laboratory of the Federal University of Uberlândia - Santa Monica Campus.

Figure 1 - Schematic representation of the CIP prototype



Source: Authors

The Table 1 presents the items, equipment and accessories used in the prototype of the CIP system with their respective terminologies.

Table 1 - Components of the experimental system

Equipment	Terminology
Centrifugal Pump ½ HP	P101
Hand valves	V101, V102, V 103 e V104
Turbine flow sensor	YF 101
Tank	TK 101

To perform cleaning with alkaline detergent in the dairy plant prototype, 20 L of 0.5 wt% sodium hydroxide solution were used. This solution was stored in the tank TK 101.

Then, the circulation of the alkaline detergent solution was circulated within the system for approximately 30 minutes. Subsequently, the alkaline detergent solution was run out of the system, the solution being transferred to another separate storage tank. After the previous stage, drinking water was supplied to the tank in order to adequately represent the typical scenario verified in the industrial facilities that use this type of cleaning.

After each step of rinsing the detergent, the water used in the CIP prototype was discarded, being measured in real time the respective conductivity. The rinsing process was maintained until the final conductivity reached the value of drinking water  $0.0735 \pm 0.008$  mS / cm with a 95% confidence level without significant variations in its value. In addition, the removal of sodium hydroxide from the system was verified by means of the phenolphthalein indicator reaction to evaluate the necessary rinsing time.

The variables of interest in rinsing with pulsed flow were amplitude and pulse period. It is important to point out that within a period the duration of the highest flow value may be different from the duration of the lower threshold, thus setting up asymmetric periods. This duration of the upper part is another parameter that can be changed. For comparison purposes, the water consumption and the amount of effluent generated with rinse were investigated at a constant flow rate of 6 L / min.

The manipulation of pulse characteristics such as amplitude, period and duration of the high stage was performed through the microcontroller, which adjusted the power of the pump to produce the pulses of interest. The controller used in this process employed proportional and integral control modes. The choice of the tuning parameters of the flow controller used as criterion the use of proportional gain and integral time values small enough to guarantee the desired performance.

In order to analyze and plan the experimental scenario, a central composite planning (CCP) was used orthogonally, with 6 experiments at the central point totaling a total of 20 experiments. The STATISTICA software was used to analyze the collected data and to create the response surfaces. The results are presented with mean value  $\pm$  error with 90% confidence interval were performed in triplicate.

The Table 2 shows the actual values and the coded levels of the variables: amplitude ( $X_1$ ), period ( $X_2$ ) and duration of the upper part ( $X_3$ ) studied in the CCP.

Table 2 - Levels of the real and codified variables used in the CCP

Variables	Levels				
	-1,525	-1	0	1	1,525
Amplitude [L/min]	1,5	2	3	4	4,5
Period [s]	62	75	100	125	138
Duration [%]	19,5	30	50	70	80,5

In order to calculate the amount of water spent in the rinse in the different configurations studied, the curve was integrated of the electronically collected flow values as a function of time, as shown in Equation 1. The division by 60 was done to correct the units, since the sampling was done in seconds, resulting in dimensional consistency the equation.

$$V = \frac{\int_0^t Q dt}{60} \quad (1)$$

Since V is the volume of water used in rinsing [L] and Q the respective volumetric flow rate [L / min].

### 3 Results and Discussion

Based on the experiments described in the methodology, the volume of water spent for each rinsing configuration. Table 3 shows the experimental design and the volume obtained for each condition.

Table 3 - Central composite planning (CCP)

Amplitude [L/min]	Period [s]	Duration [%]	Volume [L]
-1,000	-1,000	-1,000	58,12
-1,000	-1,000	1,000	45,14
-1,000	1,000	-1,000	40,72
-1,000	1,000	1,000	39,08
1,000	-1,000	-1,000	62,70
1,000	-1,000	1,000	48,54
1,000	1,000	-1,000	55,00
1,000	1,000	1,000	54,16
-1,525	0,000	0,000	45,92
1,525	0,000	0,000	54,16
0,000	-1,525	0,000	50,96
0,000	1,525	0,000	45,80
0,000	0,000	-1,525	53,00
0,000	0,000	1,525	43,96
0,000	0,000	0,000	45,96
0,000	0,000	0,000	45,48
0,000	0,000	0,000	45,70
0,000	0,000	0,000	47,30
0,000	0,000	0,000	44,24
0,000	0,000	0,000	48,52

The influence of the parameters on the effectiveness of the rinsing was evaluated with statistical analysis to identify the significant variables in the process. The results of the analysis of variance are presented in Table 4. The factors were evaluated by the ANOVA table for the volume of water spent were significant for  $p < 0.05$ .

Table 4 - Analysis of variance for volume of water spent

Factor	SS	df	MS	F	p	Comment
X <sub>1</sub>	196,88	1	196,88	60,81	0,000015	Significant
X <sub>2</sub>	88,23	1	88,23	27,25	0,000390	Significant
X <sub>3</sub>	148,93	1	148,93	46,00	0,000048	Significant
X <sub>1</sub> <sup>2</sup>	36,27	1	36,27	11,20	0,007402	Significant
X <sub>2</sub> <sup>2</sup>	13,51	1	13,51	4,17	0,068372	
X <sub>3</sub> <sup>2</sup>	14,57	1	14,57	4,50	0,059923	Significant
X <sub>1</sub> X <sub>2</sub>	57,14	1	57,14	17,65	0,001826	Significant
X <sub>1</sub> X <sub>3</sub>	0,02	1	0,02	0,01	0,941954	
X <sub>2</sub> X <sub>3</sub>	76,01	1	76,01	23,48	0,000676	Significant
<b>Residual</b>	32,38	10	3,24			
<b>SS Total</b>	663,93	19				

The correlation coefficient  $r^2$  was 0.9512, which indicates satisfactory data adequacy and that the model can be used to predict responses within the range of values studied. The second order polynomial equation presented the contribution of each variable in the volume spent, Equation 2.

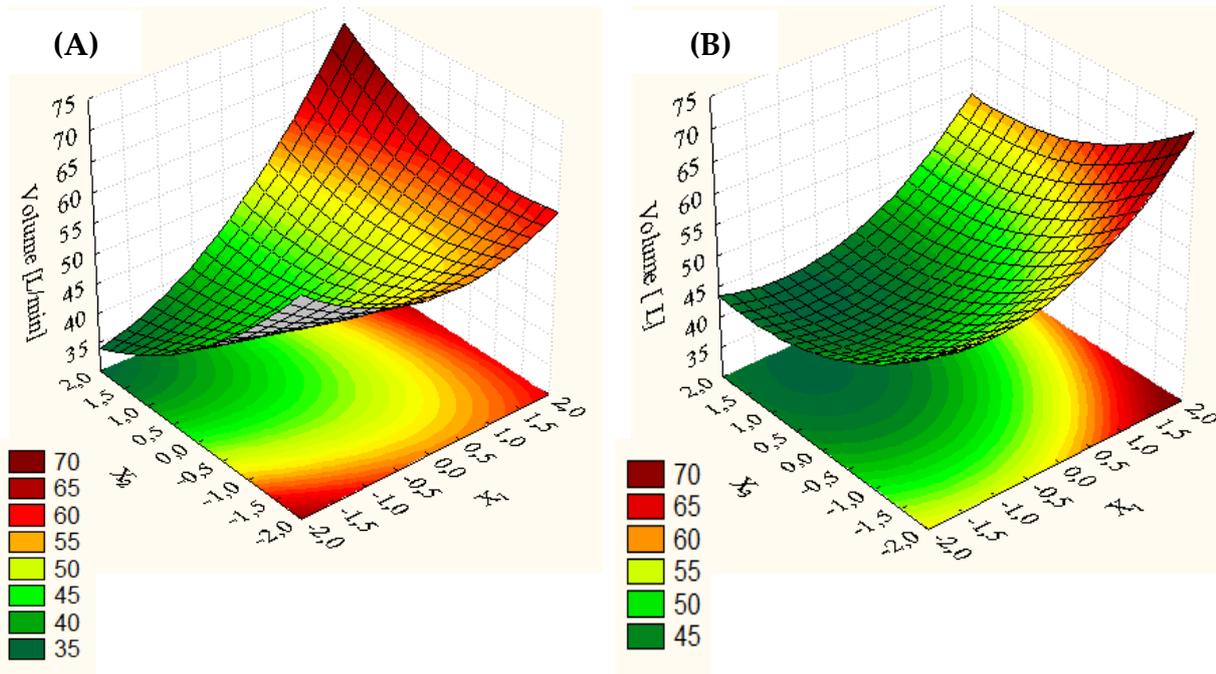
$$V = 46,12 + 3,95X_1 + 1,83X_1^2 - 2,64X_2 - 3,43X_3 + 1,16X_3^2 + 2,67X_1X_2 + 3,08X_2X_3 \quad (2)$$

In this analysis it was verified that the three variables studied and their combinations produce significant effects on the response, the most significant variable being the pulse amplitude both in its linear form and in the quadratic form, followed by the duration of the high part also in the linear form.

The increase in amplitude contributed to increase the volume spent, while the increase in the duration of the high part participated negatively, in the sense of reducing the amount of water spent. Thus, the greater the amplitude the greater the volume being spent, while the increase in the duration of the upper part of the pulse indicates a minimization of consumption. The effect of periods of longer duration may favor the reduction in the consumption of rinsing water in dairy units.

Figure 2 shows the response surfaces for the volume of water used in the rinse as a function of the studied variables. It is noticed that smaller amplitudes decrease the consumption of water. This behavior is consistent with expected, since the increase in amplitude implies a greater amount of water used in the process and consequently produces an increase in volume. However, the period and duration of the high part in smaller magnitudes did not contribute to volume reduction. In this case, the variables period and duration of the high stage minimize the water consumption when they are employed with higher values.

Figure 2 - Response surface of the volume spent. (A) Volume as a function of amplitude and period (B) Volume as a function of the duration of the high part and amplitude



Source: Authors

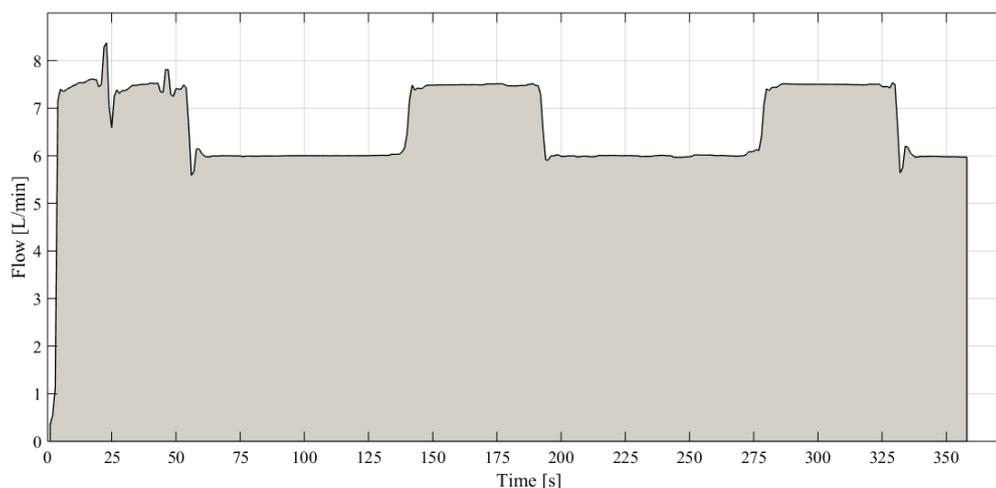
In order to investigate the best operational region, we estimated the optimal values for the parameters under study using the mathematical model shown in Equation 2. The values found to minimize the volume of water spent are presented in Table 4. Thus, the most economical rinse occurs with periodic pulses with 6 L / min in the lower part and 7.5 L / min in the upper part. In addition, the best setting for the pulse are periods of 138 s with duration of the high part 38% of the time, that is, approximately 53 seconds.

Table 4 - Optimum parameters to minimize rinse volume

Coded variables		
X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>
-1,5	1,5	-0,55
Real variables		
Amplitude [L/min]	Period [s]	Duration [%]
1,5	138	38

Using the optimum values found, the volume of 36.56 L (Equation 2) was estimated, with the amount of water required for rinsing. To verify the real consumption of the system, and to analyze the suitability of the response to the proposed objective, the system was operated with the most favorable condition, in which 39.32 L. The actual volume spent was calculated through Equation 1, which is numerically equal to the area below the curve, as shown in Figure 3.

Figure 3 - Profile of the periodic pulses in the optimal condition



Source: Authors

The consumption of the rinse made at the constant flow rate (6 L / min) was compared with the same operation performed with pulsed flow in the optimum condition with flow rate of 6 L / min and amplitude of 1.5 L / min, period 138 s and duration of high 53 seconds. Thus, it was found that the use of pulsations in the water flow used in the rinse increases the shear stress applied to the pipe walls and, therefore, reduces the amount of water spent as verified by Gillham *et al.*, 2000. Table 5 shows the comparative consumption between the flushes and the generated economy.

Table 5 - Comparison of consumption with constant flow and pulsed flow

	Constant Flow [6 L/min]	Pulsed Flow [6 a 7,5 L/min]	Economy / washing [L]
Volume [L]	46,00	39,32	6,68

Considering that the rinsing operation was evaluated in a test tube of 1.2 meters and that this stage is carried out daily in dairy products, rinsing with pulsed flow is an advantageous strategy in the cleaning of pipes since the industries have pipelines of dimensions much greater than which makes the water economy quite significant over a year, as well as contributes to the reduction of the effluent load generated.

## 4 Conclusions

In this work the alkaline detergent rinse was studied in a CIP system prototype, typically used in the dairy industry, using pulsed flows to reduce water and effluent consumption. Twenty combinations of parameters, amplitude, period and duration of the upper part were analyzed. The experimental results and the statistical analysis indicated that the magnitude of the amplitude is the variable with greater significance for water saving.

It was also estimated the most feasible operational configuration to minimize water consumption and effluent generation. For this, a flow rate of 6.0 L / min was established with periodic amplitude of 1.5 L / min, period of 138 seconds and duration of the high part of 53 seconds. Thus, the results obtained showed that the proposed alternative was able to reduce the water consumption and consequently the effluent quantity generated in about 6.68 L at each washing stage, representing an economy of approximately 14.52% in relation the conventional operation.

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