Indian J Dairy Sci 77(4): 329-337
https://doi.org/10.33785/IJDS.2024.v77i04.005

RESEARCH ARTICLE

Development and experimental performance of a cleaning-in-place system for three stage scraped surface heat exchanger

Pooja N. Bhagat[1], P. Barnwal[1](✉), Ankit Deep[1], Anup D. Gadwe[1] and P. Behare[2]

Received: 22 August 2023 / Accepted: 17 November 2023 /

Abstract: Aim of present study was to develop an innovative Cleaning-in-place (CIP) system, having control system with instrumentation, for dairy process equipments. Its performance was evaluated for three-stage thin film scraped surface heat exchanger (TS-TFSSHE). CIP system consists of multi-partition tank, control system with instrumentation and fluid flow system. Experiments were conducted with three independent variables (scraper speed: 300, 225 and 150 RPM; solution temperature: 80, 70 and 60°C; solution concentration: 2%, 1.375% and 0.75%) using response surface methodology. CIP performance responses were chemical rinsing duration, total CIP time, total plate count and coliform count. Optimized solution was obtained as 0.85% sodium-hydroxide concentration, 72.7°C CIP solution temperature and 150 RPM scraper speed. Validation of optimized solution showed that predicted response values were comparable with mean experimental values and found non-significant ($p>0.05$). This CIP system is movable (wheel mounted) and may be used for cleaning of other dairy process equipments at other places as well.

Keywords: Cleaning; CIP; Process equipment; Scraped surface heat exchanger

Introduction

Cleaning-In-Place (CIP) is unit operation for cleaning or removal of fouling deposits and product traces from surfaces of equipment and pipelines which helps in production of quality products. It is defined as "cleaning of complete items of plant any pipeline circuits without dismantling or opening of equipment and with small or no manual involvement on part of operator" (Romney, 1990). In dairy and food processing industries, re-use and multi-use of cleaning-in-place (CIP) system is practiced without taking equipment apart (Gésan-Guiziou et al. 2002) for maintaining high level of hygiene._Industries such as dairy, beverage, brewing, processed foods etc. require high level of hygiene and rely on CIP.

The fouling of heat exchangers is a major concern in dairy industry, with a negative impact on operational costs and product quality (Andritsos *et al.,*2002). Fouling reduces heat transfer and capacity of the heat exchanger and consequently the process fluids may be affected adversely. In general, for plate heat exchangers, increase in temperature resulted in substantial reduction in cleaning time and increase in flow rates had very significant effect on cleaning rates (Timperley and Smeulders, 1988). For CIP, chemicals should be selected on the basis of their ability to remove organic and inorganic fouling layers (Chisti and Moo-young, 1994). Alkaline detergent should be circulated to remove organic soil or fouling layers e.g. proteins and fats. It helps in lifting of the soil from the surface and holds it in suspension or dissolved in the alkaline detergent solution (Memisi et al. 2015).Temperature and flow velocity of CIP solution had an evident effect on effectiveness of CIP (Tuladhar et al. 2002). Kumari and Sarkar (2014) reported the use of 1.5% concentration of sodium hydroxide solution at 65°C for 30 minutes followed by use of 1% concentration of nitric acid with water rinsing step (at start, in between and end) for optimum removal of biofilm formation. Burfoot et al. (2017) suggested that the use of air bubbles in water could provide small improvements in cleaning surfaces or potentially similar contamination removal using less water._Various researchers have studied on different aspects of CIP of process equipments (Bava_et al. 2011; Chisti and Moo-young, 1994; Gésan-Guiziou et al. 2002; Khalid et al. 2015; Mattila et al._1990;_Kumari_and_Sarkar, 2014;_Sundberg et al. 2011;Timperley and Smeulders, 1988)._Van Asselt et al. (2002) reported the use of electrochemical sensors for monitoring of system for improving cleaning efficiency of CIP processes. Khalid et al. (2015) developed a test rig for cleaning studies and evaluated laboratory-scale experiments.

---

[1]Dairy Engineering Division
[2] Dairy Microbiology Division
ICAR-National Dairy Research Institute, Karnal

P Barnwal (✉)
Dairy Engineering Division,
ICAR-National Dairy Research Institute, Karnal-132 001, Haryana, INDIA
Email:pbarnwal@rediffmail.com

In industries, scraped surface heat exchangers (SSHEs) are widely used for the processing of dairy and food products with high viscosity (Rao and Hartel, 2006). For continuous manufacture of Indigenous milk products of India, a thin film scraped surface heat exchanger could be very effectively used (Abichandani and Sarma, 1991). Three-stage thin film SSHE (TS-TFSSHE) is a versatile dairy process equipment in which high temperature, pressure, flow rate of product and high heat transfer rates co-exist during processing operations (Dodeja and Deep, 2012). These harsh conditions are conducive to form tenacious layer of milk solids on the surface of equipment. Proper cleaning of product surfaces of the heat exchanger is essential for production of the quality product. Hence three stage SSHE was selected as model equipment for testing of proposed CIP system.

Considering the hygienic importance of CIP and human interactions involved in traditional or conventional cleaning process (time consuming and risk prone), the objective of this study was to develop an innovative CIP system for dairy process equipments and it was evaluated for cleaning of three-stage scraped surface heat exchanger.

Materials and Methods

Development of CIP system

This study was conducted at Dairy Engineering Division, ICAR-NDRI, Karnal, Haryana, India. The existing three stage thin film scraped surface heat exchanger (TS-TFSSHE) has three heat exchangers, each identical in length. The scraper assembly consisted of solid SS shaft of 2.5×10^{-2} m diameter. The first and second stages of TS-TFSSHE had four variable clearance blades each of 1.332 m length, 0.005 m thickness and 0.04 m width and are hinged between cross supports in each scrapper at 1.405 m distance from front end and 0.356 m from rear end. Control system consists of variable frequency drives, process controller, control switches/buttons, MCBs, digital displays for process parameters. Its instrumental parts consist of pressure gauge, magnetic flow meter, I/P converter, pressure transmitters, pneumatic valves, air pressure indicators. Some ancillary equipments associated with TS-TFSSHE consisted of balance tank, feed pump and valves for steam supply (Dodeja and Deep, 2012).

To design proposed CIP system, experiments were conducted to determine the hold-up volume for TS-TFSSHE at fixed parameters i.e. steam pressure, flow rate and scrapper speed for water as a process fluid. It was found as 10 litres (approx.) for operating parameter range (scraper speed 200, 175, 40 rpm; flow rates 350 to 528 kg/h and steam pressures 3.5, 2.5, 1kgf/cm^2 for first, second and third stage respectively). The following equations were used for determination of the holdup volume and volumetric flow rate during steady state flow condition:

$$\text{Hold up volume} = \frac{\pi d^2 \times (H_i - H_f)}{4} \qquad (1)$$

$$\text{Volumetric flow rate} = \frac{\text{volume}}{(T_f - T_i)} \qquad (2)$$

Where d is diameter of balance tank; H_i is initial level of balance tank; H_f is final level of balance tank; T_i is initial time in minutes at H_i level of balance tank; T_f is final time in minutes at H_f level of balance tank.

The developed CIP system consists of CIP multi-partition tank and pipelines, control panel and its components, direct steam Injection and inline filter and instrumentation.

CIP tank and pipelines

Important considerations for CIP multi-partition tank (capacity: 700 L; five partitions) were savings of resources such as space, water, cleaning time, heat energy etc. The volume of multi partition CIP tank (200 L) was intended for circulation of CIP solution four times (4 times hold up volume: 50 L approx.) in its CIP procedure. The tank along with mobile stand (wheels) was fabricated which consists of five partitions on the basis of hold-up volume. The five partitions were made for conc. Acid (50 L), conc. sodium hydroxide (50 L), dilute Acid (200 L), dilute sodium hydroxide (200 L) and water (200 L). These concentrated solutions were intended to be stored for making up the required concentration of CIP solutions as and when needed. During manufacturing of product using TS-TFSSHE, some milk particles falls out from vapour ducts on to nearby area including CIP tank which will make recovered and fresh solutions in tank unsuitable for re-use. To prevent it, a stainless steel (SS 304) cover (1.5 mm thick sheet) for the CIP tank was fabricated. The forward line was fabricated using 38 mm pipe (SS 304) along with two different sensors i.e. temperature (Pt-100) and pH sensors. The CIP return line was made to recover the CIP solution when it was passed through the electrical conductivity sensor (Figure 1). The valves were operated manually to recover the solution after the pump for recovery was started.

Control panel and its components

The control panel was designed to house main circuit breaker, direct on line motor starters for all four pumps (conc. acid, conc. sodium hydroxide, CIP forward, and CIP return pumps), Arduino Uno microcontroller for pH sensing system, electrical conductivity monitor, temperature indicator and level indicators for all partitions. It was positioned above the forward and return CIP pipelines for easy operation.

Direct steam Injection and inline filter

Direct steam injection (DSI), having non-return valve (NRV), was used to effectively heat the fluid either water or CIP solution. The pipeline after NRV was welded inclined in order to save inner surface of the opposite side of pipeline from high pressure steam.

The in-line filter (stainless-steel cylindrical shape) is connected in the recovery line of CIP next to the recovery pump.

Instrumentation for CIP system

The level of prepared solution was examined visualising by light emitting diode (LED). There are three different levels i.e. lower, medium, higher are indicated by LED lights (located at control panel). One resistance temperature detector (Pt-100) sensor device was used to measure the temperature of cleaning solution. The pH sensor was calibrated by the use of different buffer solutions, having the pH 4, 7, 9.2 by Arduino programming. The outgoing solutions pH was monitored by the calibrated pH sensor. Electrical conductivity sensor was fitted in the CIP return line for conductivity monitoring of recovering material. If the conductivity is matched with the inline flow, the recovery valve was open manually. Conductivity measurement is suitable for this application as only one solution was flowing at a time.

Materials required for experimental trials

Fresh buffalo milk was procured from Experimental Dairy (a Unit for teaching, and training of UG/PG students), at ICAR-NDRI, Karnal. *Khoa* manufacturing using TS-TFSSHE was selected for soiling because the process equipment (TS-TFSSHE) was primarily developed for continuous manufacture of *khoa* (Dodeja and Deep, 2012). In TS-TFSSHE, most of mineral deposits were not expected to be deposited on heat transfer cylindrical surface due to scrappers use. For CIP, sodium hydroxide is the most widely and regularly used CIP chemical in dairy industries. The *khoa* manufacturing process was used for simulating actual process plant condition soiling after production where milk fat and proteins are the major factors contributing to soiling deposit on process equipment/heat transfer surface. Sodium hydroxide flakes (having 80% assay) were diluted in potable water and a solution of 0.750 to 2.000 % strength was prepared. SS vessels and trays were used for *khoa* collection.

Experimentation

Initiation of Experiment: For each trial 40 litre buffalo milk (20-25°C) was procured from Experimental Dairy, ICAR-NDRI, Karnal, Haryana. Initially, TS-TFSSHE was rinsed with the potable water. After this, *khoa* was prepared (for soiling of equipment) from milk concentration by using TS-TFSSHE and it was collected in tray.

Product recovery and gross debris removal: After complete milk was utilised for *khoa* production, the TS-TFSSHE was flushed with water through balance tank to recover milk solids and also removal of gross debris.

Initial water flushing: For conducting CIP trials, the main balance tank was filled with hot water (60/70/80°C). The feed pump was then switched on manually and flow rate was set at 8.4-8.5 lit/min and setting the CIP return line to drain. The scraper speed was kept fixed and same (150/225/300 rpm) for all three different stages, respectively as soon as the water started to come in the shell.

Chemical rinsing: After water started coming clear (seen visually) then sodium hydroxide solution at selected concentration (0.750/1.375/2.000%) was forwarded from CIP system to TS-TFSSHE manually. When Electrical conductivity (EC) reading matched with forward EC, time taken (chemical rinsing) was recorded.

Final water flushing: Then water was circulated through the TS-TFSSHE until EC reading in the return line matched with forward water EC reading. During CIP, the steam supply to TS-TFSSHE was not closed.

Performance testing: A swab was wiped on the marked surface area after CIP was completed.

In swab test, a cotton swab moistened with quarter strength ringer's solution is applied over the known area of equipment and in this process, micro-organism adhered to the surface of the equipment are transferred to the swab. Generally, two areas admeasuring 7cm×7cm each are randomly selected at the outlet/product discharge end (third or final stage) on the test equipment. Being concentrated product with least mechanical agitation in third stage, the chances of most tenacious soil formation will be more as compared to second and first stages. Sterilized petri dishes, test tubes, sterilized cotton swab, nutrient agar (NA), violet red bile agar (VRBA), phosphate buffer solution were used for swab method. The colony forming unit (cfu/cm^2) was computed from following Equation

$$:\text{Colony forming unit}\left(\frac{\text{cfu}}{\text{cm}^2}\right) = \text{Average}\left(\frac{\text{colony count on petriplate} \times 10^{(\text{dilution used})}}{\text{area from which swab was wiped}, \text{cm}^2}\right) \ldots\ldots\ldots (3)$$

Mainly swab test is conducted as performance determination parameter as it would indicate the efficacy of cleaning in terms of microbiological analysis after CIP was done. Target for cleanliness using developed CIP system was to assure NIL counts from microbial analysis (swab test) and minimising time taken for circulation of CIP solutions along with visual cleanliness of all product contact surfaces.

The terms used for performance analysis of the developed CIP system are:

(i) **Chemical rinsing duration Y_1:** Time taken by the system during which sodium hydroxide solution was in circulation

(ii) **Total CIP time Y_2:** Total time taken by the CIP system to complete CIP process

(iii) **Total plate count, Y_3:** Total colony forming units as calculated by using Equation (3) on data obtained from nutrient agar plates and

(iv) **Coliform count, Y_4:** Total colony forming units as calculated by using Equation (3) on data obtained from VRBA plates.

Experimental design and analysis

For experimental trials, three independent variables (Table 1) were chosen and responses (dependent variables) were chemical rinsing duration (Y_1), total CIP time (Y_2), total plate count (Y_3), coliform count (Y_4) (Table1). Response Surface Methodology (RSM) was used to optimize performance of the system. It was assumed that independent variables (X_1,X_2, X_3) affect the performance responses.

After experimental trials, the values of performance responses may be defined in terms quadratic model by following equation (Yadav et al. 2012):

$$Y_k = \beta_{k0} + \sum_i^3 \beta_{ki} X_i + \sum_{i=1}^3 \beta_{kii} X_i^2 + \sum_{i=1}^2 \sum_{j=i+1}^3 \beta_{kij} X_i X_j + e_k \quad \ldots (4)$$

Where Y_k is the performance response (Y_1 = Sodium hydroxide time, Y_2 = Total CIP time, Y_3 = Total plate count and Y_4 = Coliform Count);β_{k0} is the regression coefficient (value of fitted response at centre point of the design); β_{ki}, β_{kii}, β_{kij} are linear,quadratic, interaction regression coefficient;X_i, X_j are independent variables (i = 1, 2, 3, respectively where X_1 = concentration of sodium hydroxide solution, %;X_2 = temperature of solution, °C; and X_3 = scraper speed, rpm) and e_k is unexplained error.

A face centred central composite design (FCC) with three variables was used and experimental design is presented in Table 1. There may be effect of unexpected variability in performance responses due to extraneous factors, so experiments were performed in random order. Based on result of preliminary trials, the experimental range of independent variables was identified for experimental design. In present study, the optimization was carried out using Design-Expert v.8.0.7.1 software (Stat Ease Inc., Minneapolis) to obtain the optimized conditions on suitable criteria (Table 3).

Experimental Validation of optimized solution

Responses were numerically optimized using design expert software which provides optimum conditions (sodium hydroxide concentration, X_1; temperature of CIP solution, X_2 and scraper speed, X_3) under criteria listed in Table 3 within range of experimental data. The experiments were conducted in triplicates at optimum conditions. To test the significance difference between experimental and predicted value at optimum parameters, student's t-test ($p<0.05$) was used. The Equation (5) was used for computing p values (Lamauro et al.1985):

$$P = \frac{100}{n} \sum_{i=1}^{n} \left| \frac{Q_{i_{exp}} - Q_{i_{pre}}}{Q_{i_{exp}}} \right| \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

Where, P is p value, $Q_{i_{exp}}$ is experimental value of response and $Q_{i_{pre}}$ is predicted value of response.

Results and Discussion

Figure 1 shows the developed CIP system (isometric drawing and assembled unit) for cleaning of three stage thin film scraped surface heat exchanger. It consists of mainly multi-partition tank along with mobile stand (on wheels), CIP fluid flow system, and Instrumentation and control system.

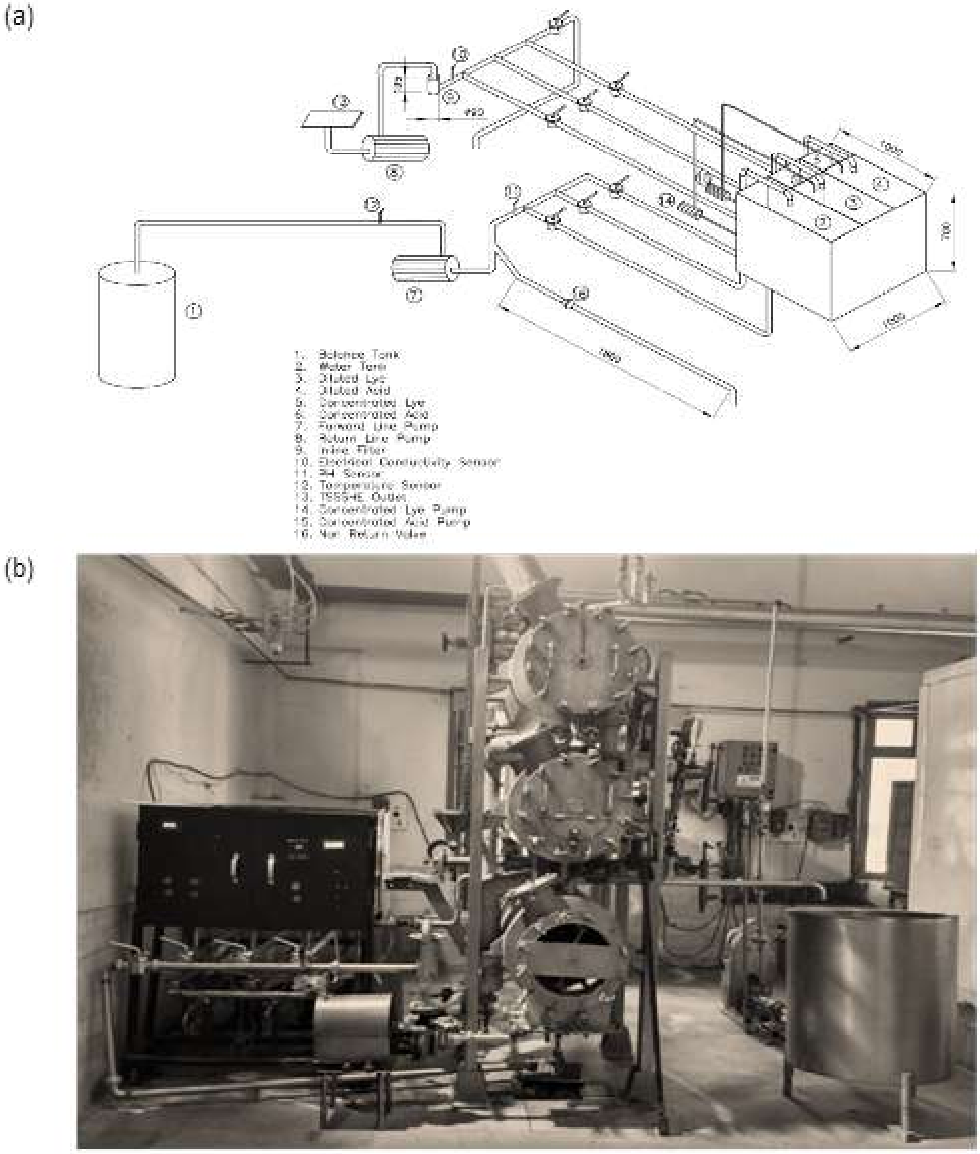
Performance Evaluation of CIP system

Table 2 shows ANOVA using quadratic (second-order) response surface model which describe influence of independent variables on the performance responses. The model was significant ($p \leq 0.05$) and the lack of fit was non-significant ($p>0.05$). The negative sign of a regression coefficient, at linear level, showed reduction in response value with an increase in level of independent variable whereas at interactive level, level of one variable decreased while that of other variable increased to give similar response values (Table 2). The 3-D response surface plots were created (Figures 2-3) to visualize the combined effect of two variables on a specific response.

Influence of process parameters on chemical rinsing duration

The maximum and minimum chemical rinsing duration were 740 s and 333 s, respectively (Table 1). With increase in temperature from 60° C to 80° C at constant concentration and speed, chemical rinsing duration decreased more rapidly ($p \leq 0.01$), which may be due to decrease in viscosity of hot solution (along with soil material) and the more cleaning effect at higher temperature and hence some increase in its flow velocity. However, as the speed increased, there was a continuous increase ($p \leq 0.05$) in the chemical rinsing duration at a specific concentration and temperature, which may be due to obstruction by scraper against flow of solution leading to increased residence time of the solution in the equipment (Table 2). Non-significant effect was observed for chemical rinsing duration with quadratic levels of concentration, temperature and scraper speed (Table 2). However, with increase in time and temperature of exposure by CIP solution, a linear increase in removal of bacterial load was observed (Kumari and Sarkar, 2014).The response surface and contour plots for chemical rinsing duration scores in relation to concentration-temperature, speed-concentration and speed-temperature have been presented in Figure 2. The coefficient of determination

Fig.1 Developed CIP system. (a) schematic sketch, (b) Assembled unit



(R^2=0.86) was in reasonable agreement with adjusted R^2-value of 0.74.The interaction effect of concentration and temperature showed a rapid increasing effect of chemical rinsing duration ($p \leq 0.01$). The decreasing effect ($p \leq 0.05$) of chemical rinsing duration was observed with interaction of concentration and scraper speed as well as temperature and scraper speed (Figure 2, Table 2).

Influence of process parameters on Total CIP time

The maximum total CIP time observed was 2004 s, while the minimum was 1549 s (Table 1). With increase in temperature at constant concentration and speed, total CIP time decreased more rapidly ($p \leq 0.01$), which may be due to decrease in viscosity of hot solution and hence some increase in its flow velocity (Table 2). However, no significant effect was observed for total CIP time with quadratic levels of concentration, temperature and scraper speed (Table 2). The response surface and contour plots for sodium hydroxide time scores in relation to concentration-temperature, speed-concentration and speed-temperature have been presented in Figure 2. The coefficient of determination (R^2= 0.86) was in reasonable agreement with adjusted R^2 of 0.73. The interaction effect of concentration and temperature showed an increasing effect of total CIP time ($p \leq 0.05$). The rapid decreasing effect ($p \leq 0.01$) of total CIP time was observed with interaction of concentration and scraper speed as well as temperature and scraper speed (Figure 2, Table 2). Khalid et al. (2015) reported that process of fouling deposit removal increased with increasing chemical concentration, temperature and velocity.

Influence of process parameters on Total plate count (TPC)

The maximum TPC observed was 1.15 cfu/cm^2, while the minimum was 0 cfu/cm^2 (Table 1). At linear level, CIP solution temperature was having highly significant negative effect ($p \leq 0.01$) for TPC which may be due to increase in microbes removal with increasing temperature and hence effective cleaning action whereas concentration and scraper speed were non-significant (Table 2). Kumari and Sarkar (2014) also reported a linear increase in removal of bacterial load with time and temperature of exposure by CIP solution. At quadratic level, temperature showed highly positive significant effect ($p \leq 0.01$) on TPC (Table 2). However, the regression coefficient value of temperature was very less. The response surface and contour plots for sodium hydroxide time

Table 1: Experimental design and data for the response surface analysis (FCC)

S.No.	Factors (X)									Responses (Y)			
	X_1: Sodium Hydroxide Concentration (%)			X_2: CIP Solution Temperature (°C)			X_3: Scrapper Speed (rpm)			Time (s)		Microbiological Testing (cfu/cm^2)	
										Y_1: Chemical Rinsing Duration	Y_2: Total CIP	Y_3: Total Plate Count (TPC)	Y_4: Coliform Count
Coded Value	-1	0	+1	-1	0	+1	-1	0	+1				
Actual Value	0.750	1.375	2.000	60	70	80	150	225	300				
1		0.750			60			300		740	2004	0.900	3.500
2		1.375			70			225		414	1739	0.000	0.000
3		1.375			70			225		414	1738	0.000	1.800
4		0.750			80			300		401	1695	0.700	2.261
5		2.000			60			150		357	1663	1.150	0.145
6		2.000			80			300		356	1549	0.214	0.000
7		1.375			70			225		432	1782	0.020	0.020
8		2.000			70			225		422	1747	0.031	0.000
9		2.000			60			300		399	1729	0.600	0.000
10		0.750			80			150		333	1617	0.500	0.200
11		1.375			80			225		348	1685	0.200	0.100
12		1.375			70			225		415	1772	0.000	0.000
13		1.375			70			300		365	1748	0.032	0.900
14		1.375			70			225		363	1687	0.020	0.000
15		1.375			60			225		438	1762	0.850	0.000
16		1.375			70			150		349	1667	0.000	0.000
17		0.750			60			150		423	1735	0.900	0.000
18		0.750			70			225		365	1685	0.100	1.030
19		2.000			80			150		440	1741	0.041	1.600
20		1.375			70			225		379	1686	0.800	0.020

Table 2 Analysis of variance for Y_1: chemical rinsing duration, Y_2: total CIP time, Y_3: total plate count (TPC)and Y_4: coliform count using quadratic response surface model

Source	df	Y_1: Chemical Rinsing Duration ($R^2 = 0.86$, $R^2_{adjusted} = 0.74$)				Y_2: Total CIP time ($R^2 = 0.86$, $R^2_{adjusted} = 0.73$)				Y_3: Total Plate Count (TPC) ($R^2 = 0.80$, $R^2_{adjusted} = 0.61$)				Y_4: Coliform Count ($R^2 = 0.85$, $R^2_{adjusted} = 0.71$)			
		SS	MS	F	p	SS	MS	F	p	SS	MS	F	p	SS	MS	F	p
Model^a	9	1.19×10^5	13189	6.85**	0.003	1.23×10^5	13623	6.65**	0.003	2.4	0.27	4.31*	0.016	15.59	1.73	6.15**	0.005
X_1	1	8294.4	8294.4	4.31^{NS}	0.065	9424.9	9424.9	4.60^{NS}	0.056	0.11	0.11	1.83^{NS}	0.206	2.75	2.75	9.78*	0.011
X_2	1	22944	22944	11.91**	0.006	36724	36724	17.93**	0.002	0.75	0.75	12.19**	0.006	0.027	0.03	0.09^{NS}	0.765
X_3	1	12888	12888	6.69*	0.027	9120.4	9120.4	4.45^{NS}	0.061	2.09×10^{-3}	2.09×10^{-3}	0.03^{NS}	0.856	2.22	2.22	7.90*	0.019
X_{12}	1	27495	27495	14.27**	0.004	13203	13203	6.45*	0.029	0.1	0.1	1.62^{NS}	0.232	0.78	0.78	2.76^{NS}	0.128
X_{13}	1	22791	22791	11.83*	0.006	27966	27966	13.65**	0.004	0.04	0.042	0.67^{NS}	0.431	6.67	6.67	23.70**	0.001
X_{23}	1	17578	17578	9.12*	0.013	25200	25200	12.30**	0.006	0.11	0.11	1.73^{NS}	0.218	1.05	1.05	3.72*	0.083
X_{11}	1	1663	1663	0.86^{NS}	0.375	51.28	51.28	0.03^{NS}	0.878	3.21×10^{-3}	3.21×10^{-3}	0.05^{NS}	0.824	0.47	0.47	1.66^{NS}	0.226
X_{22}	1	1596	1596	0.83^{NS}	0.384	27.84	27.84	0.01^{NS}	0.910	0.67	0.67	10.85**	0.008	7.57×10^{-3}	7.57×10^{-3}	0.03^{NS}	0.873
X_{33}	1	390.02	390.02	0.20^{NS}	0.662	451.84	451.84	0.22^{NS}	0.649	6.30×10^{-4}	6.30×10^{-4}	0.01^{NS}	0.922	0.33	0.33	1.18^{NS}	0.303
Residual	10	19267	1926.7			20484	2048.4			0.62	0.062			2.82	0.28		
Lack of Fit^b	5	15864	3172.8	4.66^{NS}	0.058	12182	2436.5	1.47^{NS}	0.342	0.10	0.019	0.18^{NS}	0.958	0.14	0.03	0.05^{NS}	0.997
Pure Error	5	3402.8	680.57			8302	1660.4			0.52	0.1			2.68	0.54		
Correlation Total	19	1.38×10^5	13189			1.43×10^5				3.01	0.27			18.41	1.73		

sum of squares; df, degree of freedom; MS, mean sum of squares; F, ratio of variances; p, probability

Subscript for X: 1 for Sodium hydroxide Concentration (X_1); 2 for Temperature (X_2); 3 for Scraper Speed (X_3)

^{a}: Significant; ^{b}: Non-significant

*NS: Not Significant, * Significant at 5 % level ($P \leq 0.05$), ** Significant at 1% level ($P \leq 0.01$)*

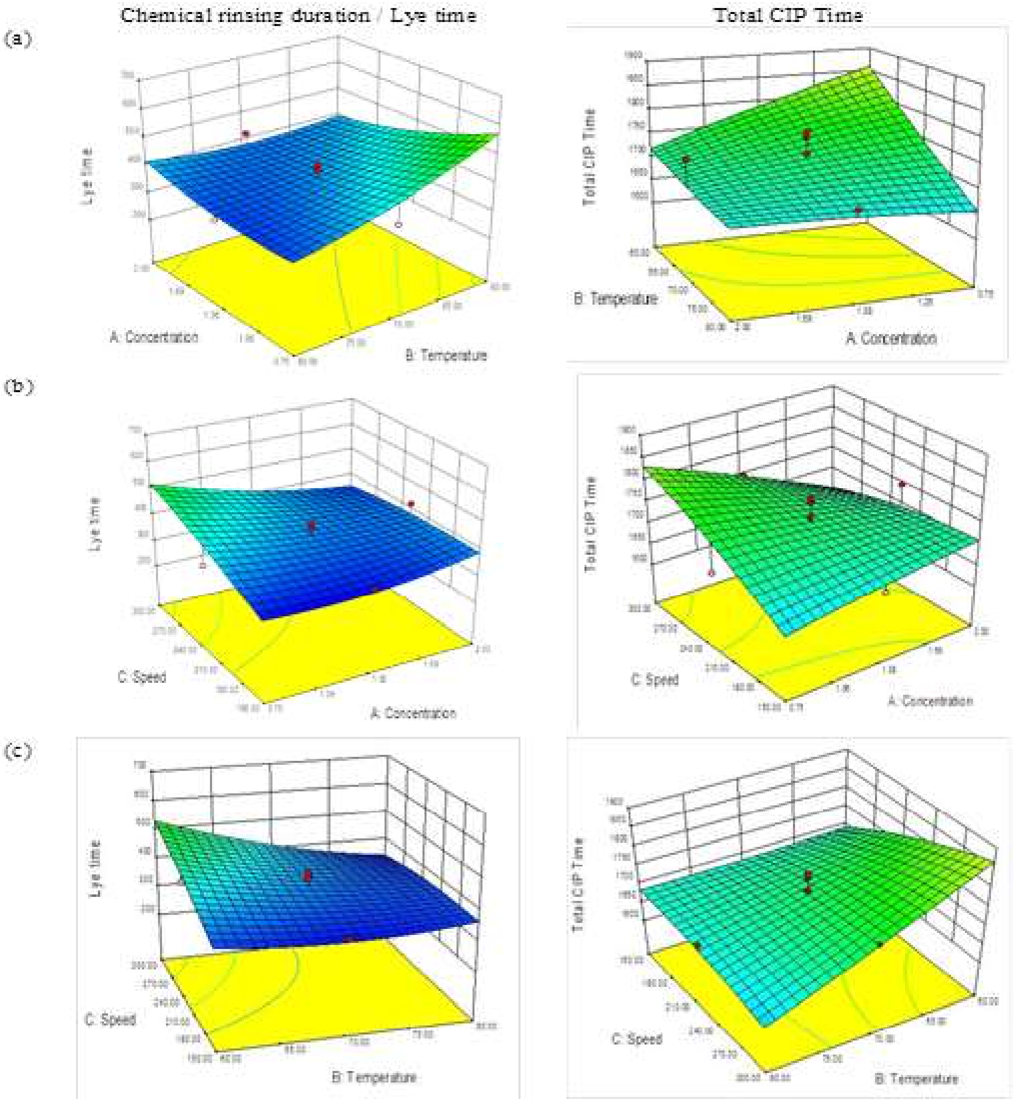


Fig.2 Response surface and contour plots of chemical rinsing duration / (lye) time and total CIP time as influenced by level of. (a) concentration and temperature at 225 rpm scraper speed, (b) scraper speed and concentration at 70°C temperature, (c) scraper speed and temperature at 1.375 % sodium hydroxide concentration

Table3 Constraints,criteria for optimization, solution along with predicted and actual response values

Constraints	Goal	Lower limit	Upper limit	Importance	Solution	ARV	%P	T Value
X_1	in range	0.750	2.000	3	0.850	-	-	-
X_2	in range	60.00	80.00	3	72.70	-	-	-
X_3	in range	150	300	3	150	-	-	-
Y_1	Minimize	333	740	3	326.328	341.33±4.04	4.38	-2.87
Y_2	Minimize	1549	2004	3	1648.7	1648.5±4.5	0.18	0.07
Y_3	Target 0	0	1.15	5	0.1039	0	-	-
Y_4	Target 0	0	3.5	5	0.1012	0	-	-

Factors (X): X_1, Sodium hydroxide Concentration (%); X_2, Temperature (°C); X_3, Scraper Speed (rpm)
Responses (Y): Y_1, Chemical rinsing duration (s); Y_2, Total CIP time (s); Y_3, TPC (cfu/cm^2); Y_4, Coliform Count (cfu/cm^2)
ARV Actual response values (Mean ± SD)
The predicted values and actual reported values for any response differed non-significantly ($p<0.05$)

scores in relation to concentration-temperature, speed-concentration and speed-temperature have been shown in Figure 3. The coefficient of determination ($R^2 = 0.80$) was in reasonable agreement with adjusted R^2 of 0.61. At interaction level, all process variables were non-significant (Figure 3, Table 2).

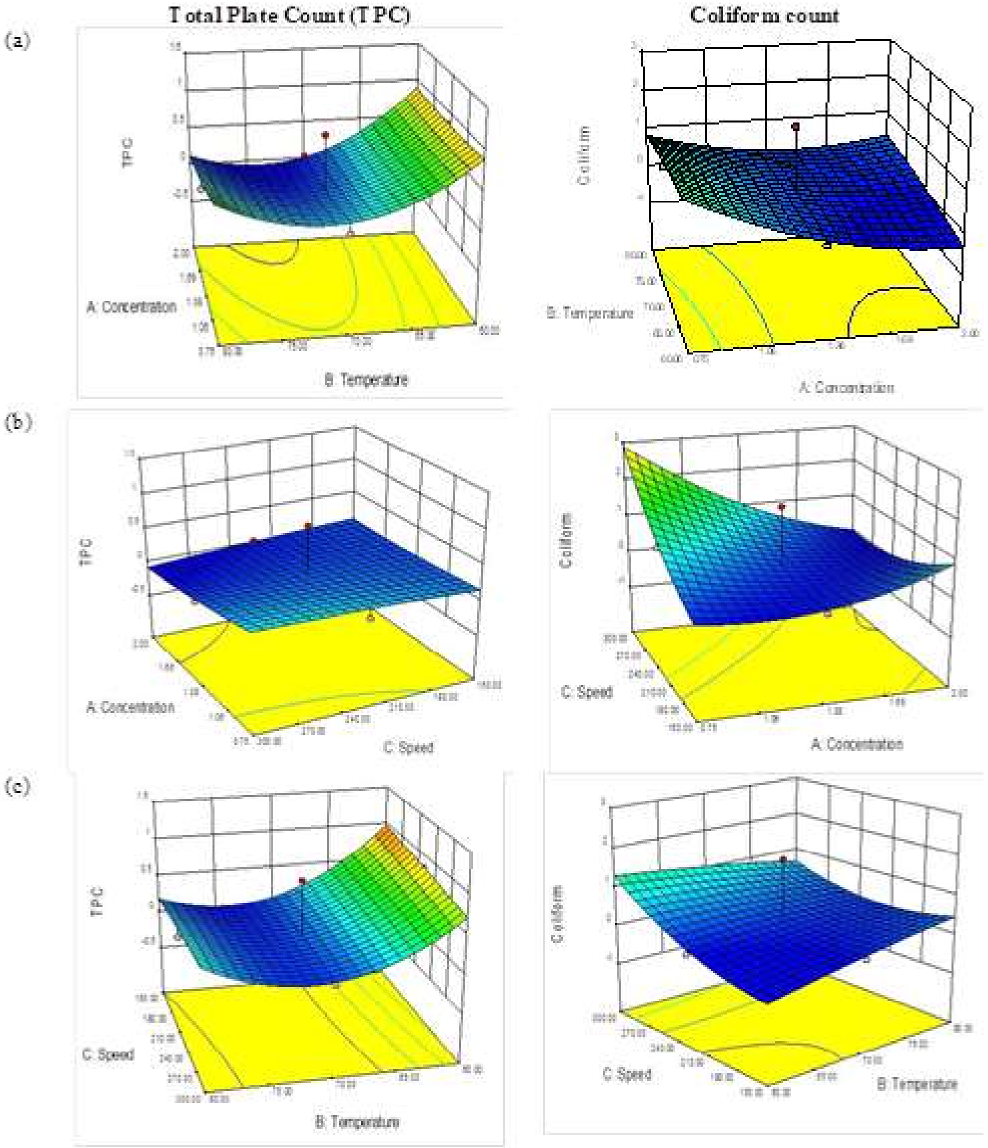


Fig.3 Response surface and contour plots of total plate count (TPC) and coliform count as influenced by level of (a) concentration and temperature at 225 rpm scraper speed, (b) scraper speed and concentration at 70°C temperature, (c) scraper speed and temperature at 1.375 % sodium hydroxide concentration

Influence of process parameters on Coliform counts

The maximum coliform count observed was 3.5 cfu/cm^2, while the minimum was 0 cfu/cm^2 (Table 1). With increase in concentration at constant temperature and speed, coliform count decreased significantly ($p \leq 0.05$), which may be due to increased mortality of microbes as concentration increased. Under flow conditions, Lelievre_et al. (2002a,b) observed that 0.5% concentration of sodium hydroxide was adequate for removal of majority of bacterial spores. With increase in scraper speed at constant temperature and concentration, coliform count increased significantly ($p \leq 0.05$), which may be due to separation of microbes from hot surface at higher scraper speed. However, CIP solution temperature as well as quadratic levels of concentration, temperature and scraper speed were having non-significant effect (Table 2). The response surface and contour plots for coliform count scores in relation to concentration-temperature, speed-concentration and speed-temperature have been shown in Figure 3.The coefficient of determination (R^2= 0.85) was in reasonable agreement with adjusted R^2 of 0.71. There was a significant decreasing effect on coliform count due to interaction effect of concentration and scraper speed ($p<0.01$) as well as temperature and scraper speed ($p<0.05$) (Figure 3, Table2).

Optimization of process parameters

Table 3 represents the constraint criteria for optimization, solution along with predicted and actual values of responses. Using constraint criteria, the optimum process conditions were obtained as 0.85% sodium hydroxide concentration (X_1), 72.70°C temperature of CIP solution (X_2) and 150 RPM scraper speed (X_3). The experimental data was compared with predicted data by conducting statistical analysis (student's t-test) and it was observed that the experimental values were not significantly different from the predicted values with respect to all responses at 5% level of significance. The %P (calculated using Equation(5)) in order to compare the precision of fit of the model and it was less than 5, for all responses suggesting that the experimental data were in good agreement with the predicted values. Thus, the response surface optimization model was adequate. The TPC and coliform counts value during validation was found as zero which is necessary for proper CIP.

In conventional cleaning process, human interactions are time consuming and risk prone etc. The fouling of heat exchangers occurs during production of dairy products with negative impact on operational costs and product quality. CIP system reduces the downtime of production due to less cleaning time for process equipment. During CIP, cleaning steps were considered to be efficiently completed when electrical conductivity (EC) reading matched with forward EC (chemical and water rinsing). Developed CIP system will save the resources e.g. labour requirement and time etc. as compared to manual cleaning. The response i.e. chemical rinsing duration and total CIP time may vary with the nature of soil (product made), size and type of process equipment.

Conclusion

An innovative CIP system was developed and its performance (time and microbial counts) was evaluated for proper cleaning of TS-TFSSHE. Optimized process parameters were 0.85% concentration of sodium-hydroxide, 72.7°C CIP solution temperature and 150 rpm scraper speed. Optimized predicted values were non-significant with the mean experimental values ($p>0.05$). TPC and coliform count were also not detected at optimized solution. This system is mobile CIP system (wheel mounted), so it may be used for other processing equipments at other places as well.

Acknowledgments

This research was conducted in Dairy Engineering Division and supported by ICAR- National Dairy Research Institute, Deemed University, Karnal, Haryana, India.

Conflicts of interest

None.

Ethical guidelines

Ethics approval was not required for this research.

References

Abichandani H, Sarma SC (1991) Evaporation in a horizontal thin film scraped surface heat exchanger. J Food Process Eng 14(3):173-187

Andritsos N, Yiantsios SG, Karabelas AJ (2002) Calcium phosphate scale formation from simulated milk ultrafiltrate solutions.Food Bioprod Process 80(4):223-230

Bava L, Zucali M, Sandrucci A, Brasca M, Vanoni L, Zanini L, Tamburini A (2011) Effect of cleaning procedure and hygienic condition of milking equipment on bacterial count of bulk tank milk. J Dairy Res 78(2): 211-219

Burfoot D, Limburn R, Busby R (2017) Assessing the effects of incorporating bubbles into the water used for cleaning operations relevant to the food industry. Int J Food Sci Technol 52(8): 1894-1903

Chisti Y, Moo-young M (1994) Clean-in-place systems for industrial bioreactors: design, validation and operation. J Ind Microbiol 13(4):201-207

Dodeja AK, Deep A (2012) Mechanized Manufacture of Danedar Khoa using Three Stage SSHE. Indian J Dairy Sci 65(4): 274-284

Gésan-Guiziou G, Boyaval E, Daufin G (2002) Nanofiltration for the recovery of caustic cleaning-in-place solutions: robustness towards large variations of composition. J Dairy Res 69(4): 633-643

Khalid NI, Nordin N, Abdul Aziz N, Ab Aziz N, Taip FS, Anuar MS (2015) Design of a Test Rig for Cleaning Studies and Evaluation of Laboratory-Scale Experiments Using Pink Guava Puree as a Fouling Deposit Model. J Food Process Eng 38(6): 583-593

Kumari S, Sarkar PK (2014) In vitro model study for biofilm formation by Bacillus cereus in dairy chilling tanks and optimization of clean-in-place (CIP) regimes using response surface methodology. Food Control 36(1):153-158

Lamauro CJ, Bakshi AS,Labuza TP (1985)Evaluation of food moisture sorption isotherm equations. i. Fruit, vegetable and meat products. LWT- Food Sci Technol 18:111-117

Lelievre C, Antonini G, Faille C, Benezech T (2002a) Cleaning-in-place modelling of cleaning kinetics of pipes soiled by bacillus spores assuming a process combining removal and deposition.Trans. IChemE 80 C:305–311

Lelievre C, Antonini G, Faille C, Benezech T (2002b) Modelling of cleaning kinetics of pipes soiled by Bacillus spores assuming a process combining removal and deposition. Food Bioprod Process 80(4):305-311

Mattila T, Manninen M, Kyläsiurola AL (1990) Effect of cleaning-in-place disinfectants on wild bacterial strains isolated from a milking line. J Dairy Res 57(1): 33-39

Memisi N, Moracanin SV, Milijasevic M, Babic J, Djukic D (2015) CIP cleaning processes in the dairy industry. Procedia Food Sci 5:184-186

Rao CS, Hartel RW (2006) Scraped surface heat exchangers. Critical Rev Food Sci Nutr 46(3):207-219.

Romney AJD (1990) CIP: cleaning in place. ed. 2. Society of Dairy Technology.

Sundberg M, Christiansson A, Lindahl C, Wahlund L,Birgersson C (2011)Cleaning effectiveness of chlorine-free detergents for use on dairy farms. J Dairy Res 78(1): 105-110.

Timperley DA,Smeulders CNM (1988) Cleaning of dairy HTST plate heat exchangers: optimization of the single stage procedure. Int J Dairy Technol 41(1):4-7

Tuladhar TR, Paterson WR, Wilson DI (2002) Investigation of alkaline cleaning-in-place of whey protein deposits using dynamic gauging. Food Bioprod Process 80(3):199-214

Van Asselt AJ, Van Houwelingen G, TeGiffel MC (2002) Monitoring system for improving cleaning efficiency of cleaning-in-place processes in dairy environments. Food Bioprod Process 80(4): 276-280

Yadav BS, Yadav RB, Jatain M (2012) Optimization of osmotic dehydration conditions of peach slices in sucrose solution using response surface methodology. J Food Sci Technol 49 : 547-555