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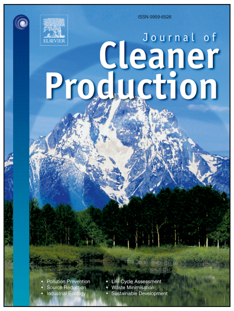

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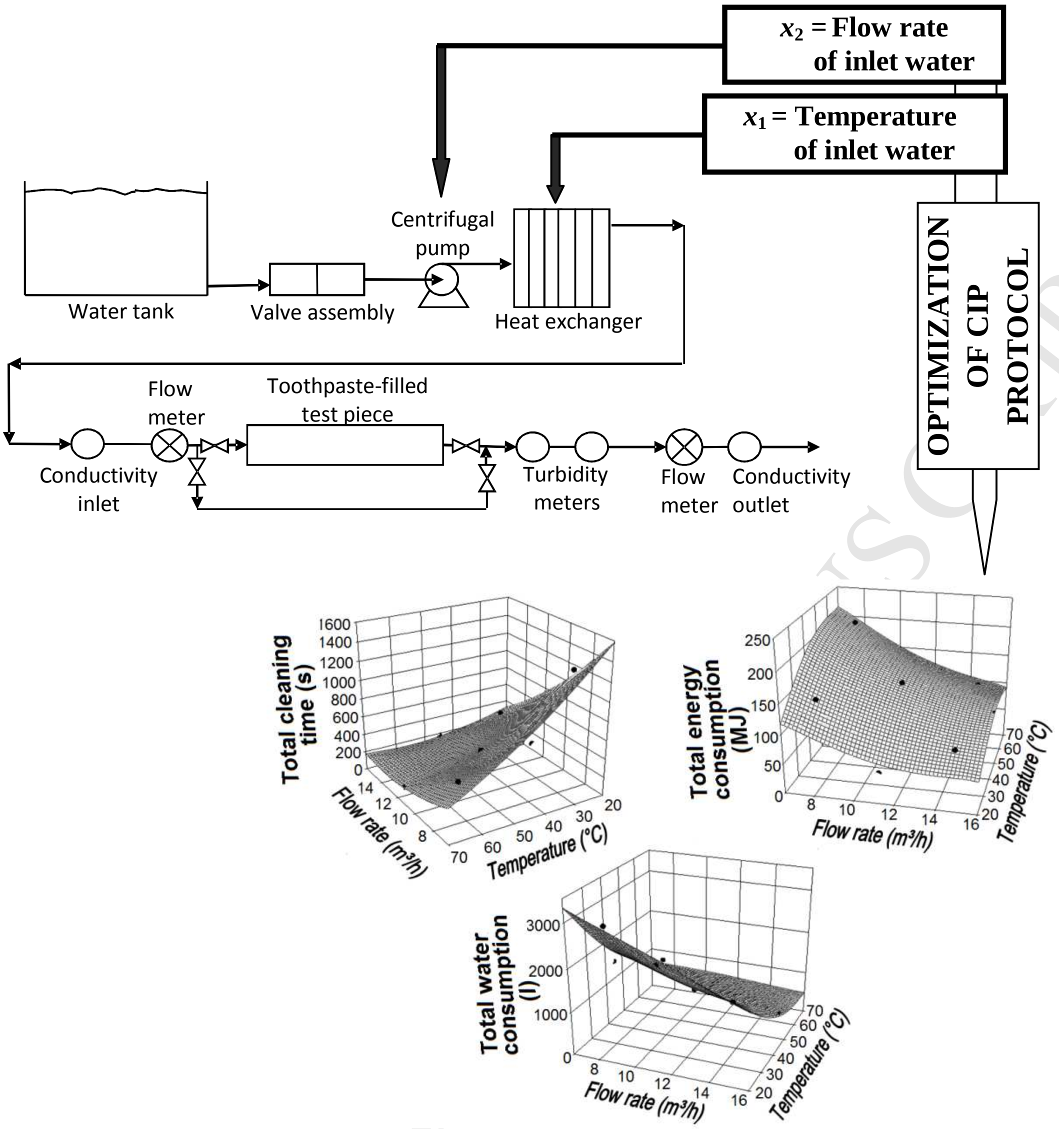

**Fig. 1.** Schematic representation of the pilot plant for optimization of cleaning-in-place protocol (CIP).

# Minimising the environmental footprint of industrial- scaled cleaning processes by optimisation of a novel clean-in-place system protocol

**Running title:** Clean-in-place optimisation in food plants

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## ABSTRACT

Cleaning of food fouling deposits in processing equipment is costly and time consuming. Fouling deposits form as a result of adhesion of species to the surface and cohesion between elements of the material. Cleaning can result from either or both adhesive and cohesive failure. In this study, the aim was to investigate the removal kinetics of an adhesive material and to design a novel cleaning in place (CIP) protocol for these kinds of materials at industrial scale to reduce environmental impact of cleaning processes. It was detected that different variables controlled the cleaning process in removal of adhesive deposit. Temperature was not found as a significant variable in the initial stage of cleaning. Velocity of cleaning water controlled the cleaning at this stage when top layers of the deposit were removed by fluid mechanical removal due to breakdown of weak cohesive interaction. In the later cleaning stage, both velocity and temperature significantly contributed to cleaning, which suggested that both hydrodynamic forces and rheological changes are needed to overcome adhesion forces between the deposit and surface. Hence, a novel “two step CIP protocol” was proposed due to existence of different mechanisms in cleaning. When compared with conventional one step CIP protocols currently used in the processing plants, the proposed CIP protocol reduced the energy consumption by 40 % without decreasing the cleaning efficiency.

Keywords: Cleaning in place, optimisation, adhesive material, pilot scale experiments, response surface methodology

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## 1. Introduction

Fouling, the unwanted build-up of deposits on a surface is a significant problem in many different industries. As a result, regular cleaning of production equipment is needed. Fouled deposits result in pressure drop and reduce the efficiency of processing equipment, increasing operating costs. Moreover, fouling may compromise product quality by cross contamination, which reveals the necessity for effective cleaning procedures. In many industries, cleaning is performed by a cleaning-in-place (CIP) procedure. This involves the circulation of hot cleaning fluids through a closed system of pipes and heat exchangers without dismantling any component from production line.

For effective cleaning, a considerable amount of water and energy is consumed at industrial scale applications, which requires process optimisation. Especially, water is an important material since it provides material flow (Koroneos et al., 2005). However, the conditions used in CIP are far from optimal. This is both because cleaning is still poorly understood (Fryer and Asteriadou, 2009) and significant brand damage may occur if contaminated product reaches the market. Cleaning has considerable economic and environmental impact (Jeurnink, and Brinkman, 1994) as it consumes substantial resources (Cole, 2011):

- high water and possible cleaning chemical usage
- energy usage to heat, pump the water and operate equipment during cleaning

Increasing fuel costs and legislative pressures towards zero emission processes make optimisation of cleaning protocols crucial. Process optimisation makes reduction in water and energy consumption possible at industrial scale, which would result in reduced economic and environmental costs such as cleaning utilization of cleaning agents (Kirby et al., 2003; Pettigrew et al., 2015). Therefore; one of the most important aims of cleaning research should be to minimise cleaning costs and the amount of effluent released during cleaning.

There are two steps to achieve this:

i) to understand and explore the mechanisms of cleaning and identify how process variables affect cleaning,

ii) to optimise the process in terms of water, energy used and time spent during cleaning.

Processing of fluid foods at an industrial scale is consisted of a complex series of sequential and simultaneous batch/continuous processes. This is why proper analysis of these processes chains in challenging step in terms of monitoring and optimising process efficiency (Pettigrew et al., 2015). In this respect, any cleaning process must overcome both the (i) cohesive forces that bind elements of deposit together, as well as (ii) adhesion forces between the deposit and surface. Many food and personal care processes involve the removal of product (such as pastes and creams) that forms layers thicker than 1 cm on the surfaces of tanks and vessels and can completely fill pipework.

In previous work (Palabiyik et al., 2014), a number of kinetic processes were observed in the cleaning of a viscoelastic material (toothpaste) from a fully filled straight pipe. Three stages were identified; (i) a short “core removal stage” of product recovery, before water breaks through the filled pipe, (ii) “a film removal stage” when there is a continuous wavy annular film of material on the wall, and (iii) “a patch removal stage” in which the material is present as patches on the wall. These stages were found in the cleaning of other yield stress materials, such as hand cream and ketchup. Core removal displaced about 50 % of the material in the tube. In the film removal stage, where cleaning disrupted the cohesive forces between deposit elements, ca. 95 wt% of the remaining deposit film was removed, largely as chunks of material. In the patch removal stage, adhesive forces between deposit elements and surface governed cleaning. Removal of deposit was slow; around half of the total cleaning time was spent in this stage to remove the remaining 5 wt% of the deposit.

Toothpaste was used as a model deposit; little work has been done on this type of fluids, as previous studies have generally focused on cleaning of deposits formed after heat treatment (Christian and Fryer, 2006; Liu et al., 2007). Also, cleaning is anticipated to depend on the material rheology for this kind of deposits (Fryer and Asteriadou, 2009). Results may well be appropriate for the cleaning of a wide range of yield stress materials in the food and personal care industries, where products are commonly of complex rheology. Existence of these different stages suggests that cleaning might be optimised by applying different cleaning conditions in each region. General practice in CIP is to circulate hot water rapidly throughout the process; however, this may not be the best practice.

It is important to carry out experiments at an appropriate scale – since, for cleaning, scale-up rules are not known (Fryer and Asteriadou, 2009). Response Surface methodology (RSM) is a suitable method to use as it can reveal general trends from the minimum number of experiments. It is a very effective tool in the statistical modelling and optimisation studies (Baş and Boyaci, 2007; Velioğlu et al., 2010). Many response surface problems involve the analysis of several responses. To perform a simultaneous consideration of multiple responses, an appropriate response surface model should be built for each response at the first step. Following this, a set of operating conditions that optimises the response should be estimated (Montgomery, 2001). In this respect, some of the variables are aimed to be maximised and some to be minimised. However, a competition occurs between these responses in many cases; namely, improving one response may lead another response to deteriorate. Several approaches have been developed to overcome this.  Constrained optimisation may be used, or different response surfaces superimposed to identify optima. Alternatively, a desirability function, which combines all the responses into one measurement, could be used. This has three advantages: (i) different scaled responses can be compared, (ii) different responses can be simply and quickly transformed to a single measurement, and (iii) it is possible to simultaneously use qualitative and quantitative responses (Harrington, 1965; Derringer and

Suich, 1980).

The main aim of this work was to find an CIP protocol with a lower environmental footprint compared to conventional CIP protocols in food and chemical processing plants. Some previous works suggest advantages of applying different CIP procedures such as pulsing cleaning chemicals (Christian and Fryer, 2006) or pulsed flows (Blel et al., 2009). The following issues are addressed;

- to determine the degree to which cleaning depends on temperature and velocity;
- to detect how this dependence changes during cleaning and;
- to perform CIP optimisation by using the multiple response optimisation (MRO) technique of response surface methodology.

## 2. Materials and methods

### *2.1. Materials and pilot plant*

Toothpaste was supplied by GSK (Brentford, UK). It is a Herschel–Bulkley fluid with an apparent yield stress of 92 Pa and is shear thinning according to (based on a model fit):

$$\sigma = 92 + 0.55(\gamma)^{0.78} \quad (1)$$

where $\sigma$ and $\gamma$ are shear stress (Pa) and shear rate ($s^{-1}$), respectively (Cole et al., 2010).

A pilot plant system at industrial scale was used to simulate a CIP set-up to monitor the cleaning procedure of toothpaste from pipe work. Industrially, cleaning fluid is generally recirculated or recycled to allow a more efficient use of resource. In this case, water was not recycled to allow quantification of the amount of water consumed during cleaning. The experiments were conducted in a pilot plant system previously used in cleaning studies at University of Birmingham (Cole et al., 2010).

A schematic of the pilot plant system is illustrated in Fig. 1. A centrifugal pump (Variflow centrifugal pump, 3 bar, 5.5 kW) being capable of transferring up to 20 $m^3$/h (3.1 m/s) water was used to pump water around the system. The test section used in this work was 0.5 m long pipe with a 0.0477 m ID and 1.6 mm wall thickness. The instrumentation used were:

- in-line inductive conductivity probes (conductivity and temperature, LMIT 08: Ecolab Ltd.), flow meters (Promag 51P, Endress-Hauser, from Ecolab Ltd.) at the inlet and outlet of the system
- two turbidity meters at outlet; Kemtrak TC007, (Kemtrak ab) and Optek TF16 (Optek-Danulat GmbH).

In this study, the Optek turbidity meter was used to monitor cleaning process over time since it was calibrated to provide greater detail at the lower end of the cleaning experiment. A reading of ‘3 ppm’ on the Optek turbidity meter was selected as the end-point of cleaning for proper comparison. In the early stages of cleaning the sensor saturated, but at the 3 ppm mark, visual examination showed the pipe to be completely clean or with only a few tiny islands of deposit, with <0.1 % of the starting weight remaining. The same cleaning procedure was applied as in previous work (Cole et al., 2010).

### *2.2. Determination of cleaning times, energy and water consumption during cleaning*

In the previous study (Palabiyik et al., 2014), a short pulse of cold and fast water was found as the best core removal condition. In the present study, water at 20 °C and 16 $m^3$/h (2.5 m/s) was used in the initial 2 s to remove the core of the material from the fully filled pipework. It was then important to identify when patch removal began. Visual observation and the online turbidity meter were compared. The glass pipe after the test section was used to follow the process, and the point where particles of removed material could no longer be seen (the end of film removal) was usually close to the point where the turbidity meter generally started to be unsaturated. For simplicity, the flow was divided into two regions; Region 1 for which the sensor saturated, and Region 2 for which it did not saturate.

Typical cleaning behaviour and cleaning regions are shown in Fig. 2. Data shows the response of the turbidity meter at 70 °C and 11.2 $m^3/h$ (1.75 m/s) water flow. The cleaning rate was initially very high, and the turbidity meter was saturated up to 125 s, the duration of Region 1. Then, the response decreased exponentially until the end of cleaning. This stage was defined as Region 2 and lasted 90 s. For each cleaning stage, water and energy consumption were calculated using:

$$V = Qt / 3600 \tag{2}$$

where $V$ ($m^3$) was volume of the water used during cleaning, $Q$ ($m^3/h$) was the volumetric flow rate and $t$ (s) was time for each region. Energy consumption was calculated by addition of hydraulic energy to drive the pump and thermal energy to heat the cleaning water:

$$E = \frac{V\rho g h}{\varepsilon} + V\rho c_p \Delta T \tag{3}$$

where $E$ was energy consumed in megajoule (MJ), $\rho$ ($kg/m^3$) the density of water, $g$ (9.81 $m/s^2$) the acceleration due to gravity, $h$ (m) was the friction head loss component of the system, $\varepsilon$ was pump efficiency, $c_p$ (4185.5 J/kgK) was heat capacity of water and $\Delta T$ (K) was temperature difference (temperature of cleaning water – datum temperature). $\varepsilon$ was found from the pump performance chart as 0.64. $h$ was calculated as 30 m by finding the maximum rate of flow rate of fluid that could be pumped in the pilot plant. Datum temperature was the average ambient temperature (17 °C), and 20 °C was selected for the minimum temperature for experiments. Pumping energy ranged between 0.3 % and 5 % of the total energy consumption in cleaning experiments.

### *2.3. Experimental design and statistical analysis*

In the response modelling, multiple linear regression analysis was used and the following second-order polynomial equation of function $x_i$ was fitted for each factor assessed at each experimental point.

$$\hat{y} - E = \beta_0 + \sum_{i=1}^{2} \beta_i x_i + \sum_{i=1}^{2} \beta_{ii} x_i^2 + \sum_{\substack{i=1 \\ i<j}}^{2} \sum_{j=i+1}^{2} \beta_{ij} x_i x_j \ , \tag{4}$$

where $\hat{y}$ was the estimated response; $\beta_0$ was the average value of the response at the centre point of the design, $\beta_1$, $\beta_2$, $\beta_{12}$, $\beta_{11}$ and $\beta_{22}$ were linear, interaction and quadratic terms, respectively and $E$ was the statistical error term.

Models were built to describe the effect of independent variables (cleaning water temperature and flow rate) on the cleaning time, energy and water consumption for both film removal (Region 1), patch removal (Region 2) and the combined total cleaning stages (the 1st + 2nd regions). A 2-factor-5-level Central Composite Rotatable Design (CCRD) with two replicates at the centre point was used. The two factors, levels and experimental design in terms of coded and uncoded (actual values) can be seen in Table 1. The CCRD is an optimal design that allows calculation of a model, with a minimum number of experiments. It consists of 2kfactorial points (coded as ±1 notation), augmented by 2k axial points (±α,0,0,…,0), (0,±α,0,…,0), (0,0,±α,…,0),…,( (0,0,0,…,±α) located at a specified distance α from the centre in each direction on each axis defined by the coded factor levels. n0 is each centre point (0,0,…0). k is the number of factors. The relationship between coded and actual values of variables was calculated using:

$$x_i = \frac{z_i - 0.5\left(z_{i,\max} + z_{i,\min}\right)}{0.5\left(z_{i,\max} - z_{i,\min}\right)} \tag{5}$$

where *z* was the actual variable, the subscripts min and max referred to the minimum (27 ºC and 7.86 $m^3/h$ (1.2 m/s), respectively) and maximum values (63 ºC and 14.54 $m^3/h$ (2.3 m/s), respectively) and *x* was the coded variable. In this study, rotatability was selected; the design is rotatable if the variance of the response is constant for all variables at a given distance from the design centre. The CCD is rotatable if:

$$\alpha = \sqrt[4]{2^k} \tag{6}$$

The best fitting models were determined using multiple linear regressions with backward elimination regression (BER) where insignificant factors and interactions were removed from the models and only variables significant at $P<0.01$, $P<0.05$ and $P<0.1$ levels were selected for the model.

### *2.4. Multiple response optimisation (MRO)*

The operating conditions, x providing the “most desirable” response values can be found by multiple response optimisation. Different desirability functions $d_i(Y_i)$ can be used depending on whether a particular response $Y_i$ is to be maximized and minimised (Derringer and Suich, 1980).

Let $L_i$, $U_i$ and $T_i$ be the lower, upper and target values, respectively, desired for response $Y_i$. If a response is to be maximized, then its individual desirability function is with the exponent s that determines how significant it is to hit the target value. For $s = 1$, the desirability function increases linearly towards $T_i$ which indicates a large adequate value for the response; for $s < 1$, the function is convex, and for $s > 1$, the function is concave (Eren and Kaymak-Ertekin, 2007):

$$d_i(\hat{y}_i) = \begin{cases} 0 & \hat{y}_i(x) < L_i \\ \left(\dfrac{\hat{y}_i(x) - L_i}{T_i - L_i}\right)^s & L_i \le \hat{y}_i(x) \le T_i \\ 1 & \hat{y}_i(x) > T_i \end{cases} \tag{7}$$

If a response is to be minimised, then its individual desirability function is with *T<sub>i</sub>*, which indicates a small adequate value for the response:

$$d_i(\hat{y}_i) = \begin{cases} 1 & \hat{y}_i(x) < T_i \\ \left(\dfrac{\hat{y}_i(x) - U_i}{T_i - U_i}\right)^s & T_i \le \hat{y}_i(x) \le U_i \\ 0 & \hat{y}_i(x) > U_i \end{cases} \tag{8}$$

Having computed for each response variable, desirability values were combined into a single desirability index, *D*. For this purpose, each response was transformed in a dimensionless function, the partial desirability function, $d_i$, which reflects the desirable ranges for each response. The desirable ranges varies from zero to one (least to most desirable). The global desirability function *D* is the weighted geometric mean of *n* individual desirability functions (all transformed responses) [Eq. (9)]. The simultaneous objective function is a geometric mean of all transformed responses (Lewis et al., 1999; Myers and Montgomery,1995):

$$\begin{aligned} D &= \left(d_1^{p_1} \times d_2^{p_2} \times d_3^{p_3} \times \ldots \times d_\mathrm{n}^{p_i}\right)^{1/\sum p_i} \\ &= \left[\prod_{i=1}^{n} d_i^{p_i}\right]^{1/\sum p_i} \end{aligned} \tag{9}$$

where $p_\mathrm{i}$ was the weighting of the $i_\mathrm{th}$ term, and was normalized in order that $\sum_{i=1}^{n} p_i = 1$. By weighting of partial desirability functions, it is possible to enable the optimisation process to take the relative importance of each response into consideration. Allowing the examination of the form of the desirability function, it is permitted to find the region where the function is close to 1 and to determine the compromise optimum conditions.

In the present study, multiple response optimisation were separately conducted for each stage, with parameters;

- *Region 1* : “film removal ”; first cleaning time - FCT; first energy consumption - FEC; first water consumption - FWC,
- *Region 2*: “patch removal stage”; second cleaning time - SCT; second energy consumption - SEC; second water consumption- SWC ) and
- *Total cleaning*: ; total cleaning time - TCT; total energy consumption -TEC; total water consumption - TWC.

In each stage the aim was to minimise cleaning time, energy and water usage. The same importance was applied to each response during the optimisation analysis. The modelling procedure and optimisation methodology by RSM is diagrammed in Fig. 3. The computational work was performed using a statistical package, Design-Expert version 7.0 (Stat-Ease Inc., Minneapolis, USA).

## 3. Results and discussion

### *3.1. Interpretation of the RSM model fit*

Table 1 shows the coded and actual levels of the experimental factors (independent variables). The experiments were run in a random order to minimise the effect of uncontrollable variables. Tables 2, 3 and 4 show the ANOVA results used to evaluate the significance of the constructed quadratic models. Model terms were used after the insignificant ones were eliminated, and other statistical parameters were obtained using backward elimination regression (BER) procedure. The fits for the models were significant ($P>0.05$), indicating that the fitted models could describe the variation of the data.

Residual analysis, $R^2$ (coefficient of determination), adj-$R^2$ (adjusted $R^2$), pred-$R^2$ (predicted $R^2$) and adequate precision (adeq-precision) values were used to check the adequacy of the models (Tables 2-4). The $R^2$ values generally ranged between 0.790 and 0.988, indicating that the models generated were adequate. An adequate precision value greater than 4 is desirable. In practice, values between 9.24 and 24.0 were found (Tables 2-4) which indicated that these models could be used to navigate the design space. Results in Tables 2-4 show;

- ($R^2$) values for time, energy and water consumption were 0.921, 0.912 and 0.936 when variables (temperature and flow rate) were fitted to data for the total cleaning process.
- However, when variables were fitted to Regions 1 and 2 separately, $R^2$ values for time, energy and water consumption increased (to 0.988, 0.906 and 0.975, respectively for Region 2).

The model thus gave a better description of cleaning when Regions 1 and 2 were considered separately. This suggested that Regions 1 and 2 had different cleaning kinetics, and that both have to be considered in an optimum CIP protocol.

### 3.2. The effect of temperature and flow rate

#### 3.2.1. Cleaning times

The effects of temperature and flow rate values on the cleaning times in Region 1 are presented in Tables 2-4. Results clearly revealed that linear effects of the temperature were significant ($P<0.01$) in all stages (Tables 2-4). Fig. 4 illustrates these effects as response surfaces. Fig. 4-a shows that at high flow rates (16 $m^3/h$-2.5 m/s), increasing the temperature has little effect on cleaning times in Region 1. In this case, breakage of cohesive bonds in the deposit controls cleaning; data suggests that beyond some flow velocity these bonds are weak enough to be broken by flow, so further increase in temperature has little effect. However, temperature had a considerable impact in the cleaning time in Region 2 in Fig. 4-b. At any flow rate, increasing temperature decreased the cleaning time. These results implied that the adhesive bonds that must be broken to remove the final layers of deposit are temperature sensitive. This is in agreement with the work of Akhtar et al. (2010) who found that toothpaste showed higher adhesive than cohesive forces. Whey protein deposits (Liu et al., 2006) and yeast (Goode, 2011) were also found to have this behaviour. For all of these deposits, cleaning occurred through removal of chunks initially, and the last stages of removal was the limiting step (Goode, 2011; Bird and Fryer, 1991).

For the effect of flow rate, cleaning times were significantly ($p<0.01$) influenced by flow velocity in all regimes (Tables 2-3). From the Fig. 4 (a, b and c), the cleaning times (FCT, SCT and TCT) can be observed to decrease with flow rate at each stage.

These results again showed different kinetics in the two regions, therefore different cleaning protocols should used in each stage for optimisation, this will be discussed in section 3.3.

To improve the accuracy of the regression model equations, their insignificant ($p>0.1$) factors and interactions were removed from the models using BER. They were generated to predict effects of the processing variables in Fig. 4 and calculated:

$$\hat{y}_{(\text{first cleaning time, }FCT)} = 1611 - 19.62(T) - 100.2(FR) + 1.297(T)(FR) \tag{10}$$

$$\hat{y}_{(\text{second cleaning time, }SCT)} = 2.404 - 35.57(T) - 191.6(FR) + 1.052(T)(FR) + 0.17(T)^2 + 4.85(FR)^2 \tag{11}$$

$$\hat{y}_{(\text{total cleaning time, }TCT)} = 3148 - 39.75(T) - 183.3(FR) + 2.349(T)(FR) \tag{12}$$

where $T$ (°C) was the temperature and $FR$ ($m^3/h$) was the flow rate.

### *3.2.2. Energy consumption*

Tables 2-4 show the effects of temperature and flow rate on energy consumption in cleaning. Significant ($p<0.01$) linear effects of temperature were observed for energy consumption in Region 1. Energy usage in this stage increased as the temperature of the cleaning water increased. As temperature did not help cleaning in this stage, as noted above, increased temperature of the cleaning water caused energy waste. However, in Region 2, an increase in the temperature did not have a clear effect on the energy consumption (SEC) (Table 3 and Fig 4-e), which indicated the complexity of the cleaning process in Region 2. Figure 4-e shows that raising temperature to 50 °C increased the energy usage, and a further increase above 50 °C reduced energy usage especially at the highest flow rate. Hence, results implied that there was a threshold temperature value above which adhesive bonds of the deposit were weakened so that they could be easily removed. Thus, energy usage was reduced by improved cleaning efficiency at high temperatures.

FEC, SEC and TEC were ($p<0.01$) influenced by flow rate (Tables 2-4). Fig. 4 (d, e and f), showed that these values decreased with flow rate at each stage, indicating that energy waste can be decreased with increasing flow rates. Again this showed the importance of flow rate in the whole cleaning process.

The second order regression model equations, after insignificant ($p>0.1$) factors were removed, were as follows:

$$\hat{y}_{(\text{first energy consumption}, \mathit{FEC})} = 69.41 + 1.406(T) - 5.187(FR) \quad (13)$$

$$\hat{y}_{(\text{second energy consumption}, \mathit{SEC})} = 129.7 + 3.183(T) - 20.95(FR) - 0.036(T)^2 + 0.727(FR)^2 \quad (14)$$

$$\hat{y}_{(\text{total energy consumption}, \mathit{TEC})} = 60.07 + 7.35(T) - 9.83(FR) - 0.067(T)^2 \quad (15)$$

where $T$ (°C) was the temperature and $FR$ ($m^3/h$) was the flow rate.

### 3.2.3. Water consumption

As can be seen from tables 2-4, linear effects of temperature were found significant ($P<0.01$) on water consumption at all stages. Fig. 4-g showed that water usage in Region 1 could be slightly reduced by increasing the temperature at the highest flow rate (16 $m^3/h$-2.5 m/s). Whereas in Region 2, Fig 4-h showed that increased temperature of cleaning water decreased the water consumption regardless of the flow rate. This result indicated that increasing temperature levels at this region would be advantageous for the environmental impact due to less amount of water released during cleaning.

FWC, SWC and TWC were significantly ($p<0.01$; 0.05) influenced by flow (Tables 2-4). From Fig. 4 (g, h and i), it was seen that the water consumption values (FWC, SWC and TWC) decreased with flow rate at each stage. The second order regression model equations after insignificant ($p>0.1$) factors and interactions were removed from the models were:

$$\hat{y}_{(\text{first water consumption, } FWC)} = 1922 - 13.81(T) - 52.96(FR) \quad (16)$$

$$\hat{y}_{(\text{second water consumption, } SWC)} = 2944 - 62.98(T) - 41.19(FR) + 0.43(T)^2 \quad (17)$$

$$\hat{y}_{(\text{total water consumption, } TWC)} = 5285 - 98.13(T) - 94.14(FR) + 0.666(T)^2 \quad (18)$$

where $T$ (°C) was the temperature and $FR$ ($m^3/h$) was the flow rate.

Similar trends between second cleaning region and total cleaning profile in figures 4-b and 4-c, 4-e and 4-f, 4-h and 4-i importantly illustrated that Region 2 was the dominating stage which generally comprised 60-70 % of the total cleaning time, and mechanisms in the removal of the last patches of deposit were the limiting processes in overall cleaning.

### 3.3. Finding an optimum CIP protocol

In this study, the multiple response optimisation (MRO) technique was separately applied for stage 1 (FCT, FEC, FWC), stage 2 (SCT, SEC, SWC) and total cleaning stage (TCT, TEC, TWC). For optimisation, desirability functions of RSM were used to obtain the resultant optimum operating conditions with the minimisation of the values for each stage (Eq. 9). The desirability values (*D*) for the minimisation were calculated to be 0.897, 0.998 and 0.910 for stage 1, stage 2 and total cleaning stages, respectively, indicating that all responses or factors were inside acceptable desirability ranges. By applying desirability function method, three solutions were obtained for each optimisation process (minimisation).

For the most desirable solutions for the minimisation of each response variable (time, energy and water consumption) at each removal stage, the following conditions should be applied:

- 20 ºC and 16 $m^3$/h (2.5 m/s) in region 1. At this circumstance, the solution had the lowest value of FCT (42.6 s), FEC (22.6 MJ) and FWC (727.4 L) values to get the optimum CIP protocol.
- 70 ºC and 16 $m^3$/h (2.5 m/s) in region 2 which induced the lowest value of SCT (39.1 s), SEC (25.2 MJ) and SWC (108.9 L) values according to response surface models.
- For the conventional CIP system (without applying different conditions throughout the cleaning process), 70 ºC and 16 $m^3$/h (2.5 m/s) should be used for the total cleaning. At this circumstance, the solution had the lowest value of TCT (64.5 s), TEC (89.2 MJ) and TWC (178.2 L) values. This result confirmed the conditions used in the conventional CIP protocol. As known, current practice in industrial CIP operations is to use hot and fast water throughout the cleaning process.

### *3.4. Validation of the optimum CIP protocol*

In this part, three CIP protocols were tested at the pilot scale pipe work to validate whether the optimum CIP protocol determined by MRO technique would provide savings in real applications. These were:

**i)** cold conventional CIP protocol - 20 °C water at 16 $m^3/h$ (2.5 m/s) was used for the overall cleaning. This kind of flow (high-velocity water at ambient temperature) is often used in the pre-rinse stage of CIP operations. Cold CIP was chosen to figure out the water saving when the optimum CIP procedure is used instead of cold CIP.

**ii)** hot conventional CIP protocol - 70 °C water at 16 $m^3/h$ (2.5 m/s) was used for the overall cleaning. Hot high-velocity water is generally applied in the industry. It was selected to enable comparison of the energy usage between the hot CIP and optimum CIP protocols.

**iii)** the novel two-step CIP protocol – water at 20 °C - 16 $m^3/h$ (2.5 m/s) was used in region 1 and water at 70 °C - 16 $m^3/h$ (2.5 m/s) was used in region 2 as determined in section 3.3. The experiment was done by starting cleaning with water flow at 20 °C - 16 $m^3/h$ (2.5 m/s). When the turbidity meter began to unsaturate, pump was stopped immediately. Then, water at 70 °C at the flow rate of 16 $m^3/h$ (2.5 m/s) was pumped to the system until the turbidity meter reached to 3 ppm.

Fig. 5 showed the measurements on the turbidity meter for the three CIP protocols. It illustrated that

- comparable cleaning times were obtained in the hot (100 s) and the optimum CIP (126 s) protocols,
- in the optimum CIP protocol, water at 20 °C was applied up to 73 s at which unsaturation started. Right after the application of water at 70 °C, turbidity reading saturated again during the time elapse between 73 and 106 s due to increase in the removal rate induced by hot water. Then, a very quick region 2 was observed after 106th s (20 s), which validated the generated response surface models by showing the temperature sensitivity of this region,

- the cold CIP protocol caused ca. 100 % (265 s) increase in cleaning time as compared to the optimum CIP protocol, mainly due to long cleaning time spent in region 2.

Fig. 6 shows the results obtained from the tested CIP protocols in terms of cleaning time (s), energy (MJ) and water (L) consumption. The hot CIP protocol was observed to result in great reductions (at least 75 %) in terms of cleaning time and water consumption, as compared to the cold CIP protocol. This showed the advantage of applying hot and high-velocity water (2.5 m/s-16 $m^3$/h) in conventional CIP procedures. However, the hot CIP protocol caused the highest energy consumption amongst the tested CIP protocols, i.e. almost quadrupled the amount of energy consumed in the cold CIP protocol.

The optimum CIP protocol notably reduced the amount of waste water and cleaning time by ca. 50 % and 53 %, respectively, compared to the cold CIP protocol. Moreover, 39 MJ less energy (ca. 40 %) was consumed in the optimum CIP protocol, compared to the hot CIP protocol. From the results, it can be deduced that in water starved areas, the hot CIP protocol should be used in cleaning operations in plants. However, sustainability is increasingly important and one of the major areas where optimisation is sought is in energy usage. Therefore, the optimum CIP protocol has a big advantage over conventional CIP protocols as the results imply that it can substantially decrease the carbon footprint and fuel costs of cleaning processes in plants where adhesive products are manufactured.

## 4. Conclusion

The increasing need to reduce water consumption and emissions in manufacturing industries demands the improvement of cleaning operations in the food industry. In this study, two different cleaning stages were identified by the turbidity meter and visual observations. Although velocity had considerable effects at both stages (stages 1 and 2), the effect of temperature was not found influential on the cleaning time and water consumptions in stage 1, especially at high flow rates. Consequently, increase in temperature of cleaning water used in stage 1 increased the energy consumption. However, in stage 2, both temperature and velocity significantly contributed to cleaning due to the strong adhesive forces of the deposit and increase in these variables reduced the energy consumption during cleaning.

After determination of the kinetics of the two cleaning stages and how cleaning of the deposit would depend on temperature and flow rate, a novel two step CIP protocol was designed using MRO technique. The optimum CIP protocol reduced the amount of waste water and cleaning time by ca. 50 % and 53 %, respectively, compared to the cold one step CIP protocol. In addition, the energy consumption was reduced by ca. 40% compared to the hot one step CIP protocol during cleaning.

As a result, this work demonstrated how to evaluate the effect of process conditions on cleaning of a specific deposit. By this, it is possible to design better CIP protocols, which can be applied to target any similar industrial process in order to substantially decrease the environmental footprint of processing plants during cleaning.

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## Figure captions

**Fig. 1.** Schematic representation of the pilot plant.

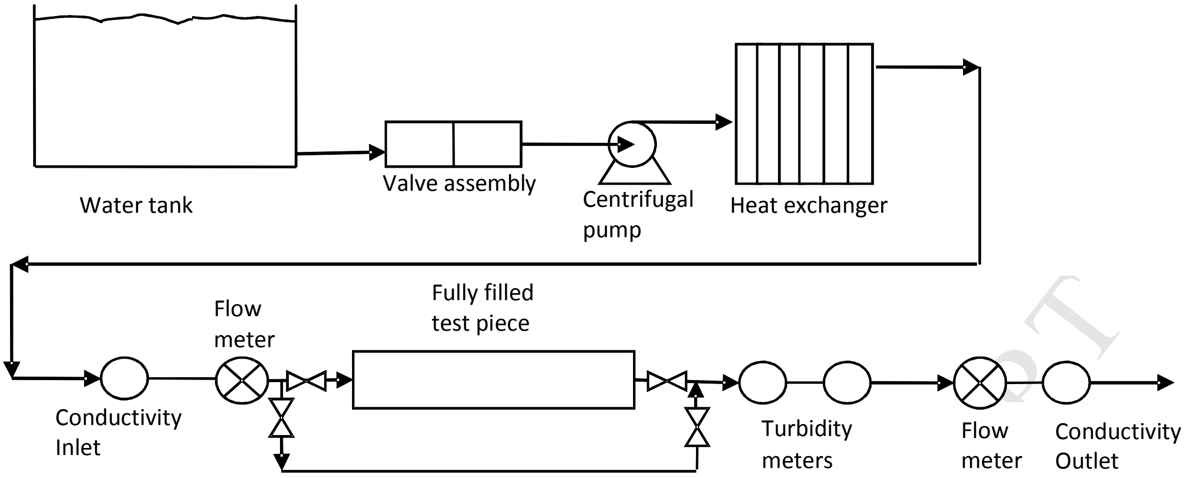
**Fig. 2.** Typical cleaning behaviour that showed decreasing dirt particle concentration in effluent water. It was measured with turbidity meter at ppm level. Turbidity reading was obtained during the cleaning of toothpaste at 70 °C and 11.2 m3/h-1.7 m/s from a pilot scale straight pipe (0.5 m and 0.0477 m ID).

**Fig.3.** Steps of modelling and optimisation by CCRD of RSM. 1. time, 1. energy and 1. water indicate cleaning time, energy and water consumptions at stage 1 which ends when turbidity meter unsaturates. 2. time, 2. energy and 2. water indicate cleaning time, energy and water consumptions at stage 2 which starts after turbidity meter become unsaturated. Total time, total energy and total water indicate cleaning time, energy and water consumptions during the total cleaning process without considering the individual cleaning stages.

**Fig.4.** Response surface plots of different cleaning stages influenced by varying temperature and flow rate values of water applied during cleaning. Effect of temperature and flow rate on (a) FCT, (b) SCT, (c) TCT, (d) FEC, (e) SEC, (f) TEC, (g) FWC, (h) SWC and (i) TWC values.

**Fig. 5.** Readings for dirt particle concentration in effluent water (ppm) obtained during three tested (cold, hot and optimum) CIP protocols (flow rate was 16 $m^3/h$ in all systems).

**Fig. 6.** Cleaning time, water and energy consumptions measured at three tested (cold, hot and optimum) CIP protocols (flow rate was 16 $m^3/h$ in all systems). In cold CIP protocol (grey), water was used at 20 °C and in hot CIP protocol (black), water was used at 70 °C during the whole cleaning (without changing conditions at stage 1 and 2). In optimum CIP procedure (white), water at 20 °C was used at stage 1 and at 70 °C at stage 2.



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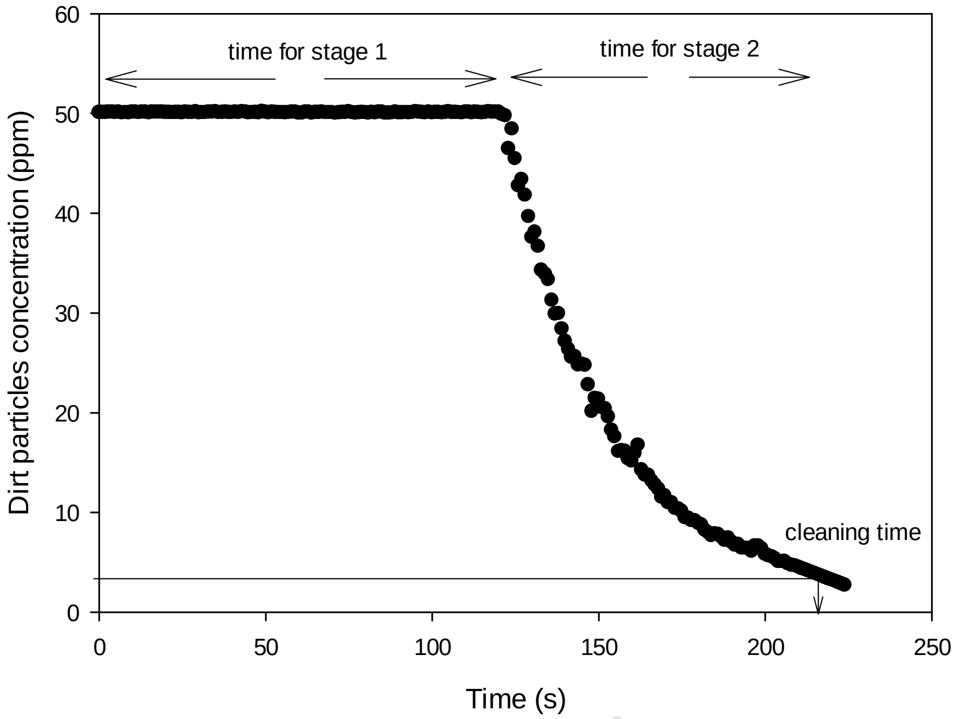

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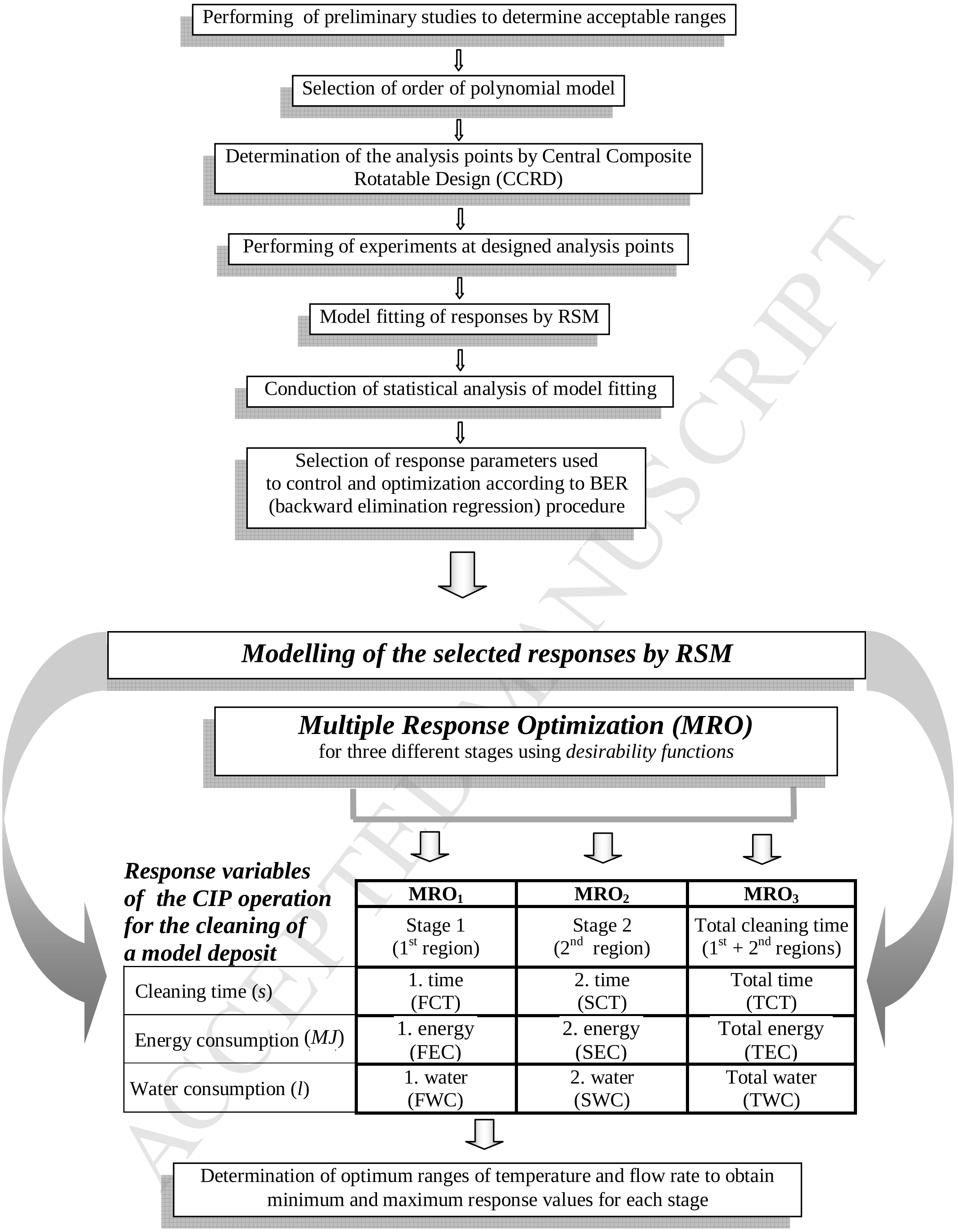

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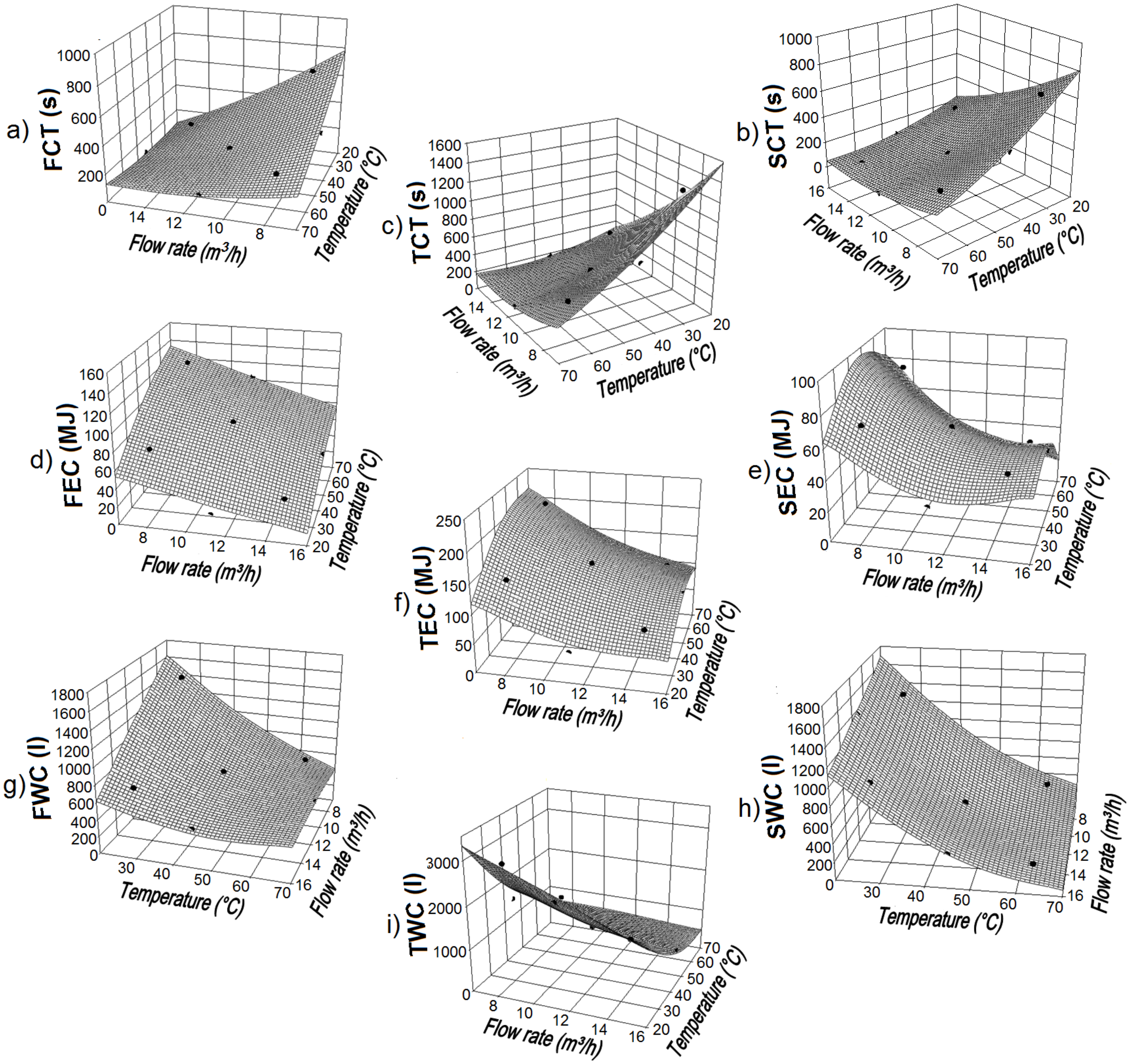

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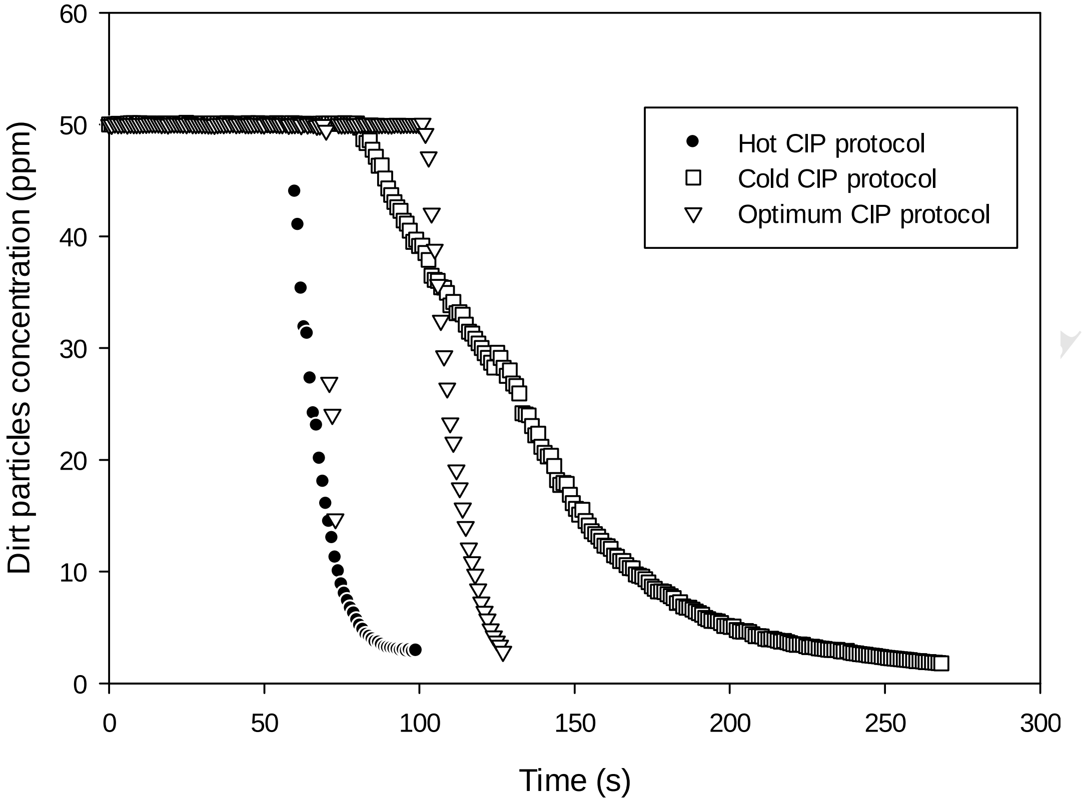

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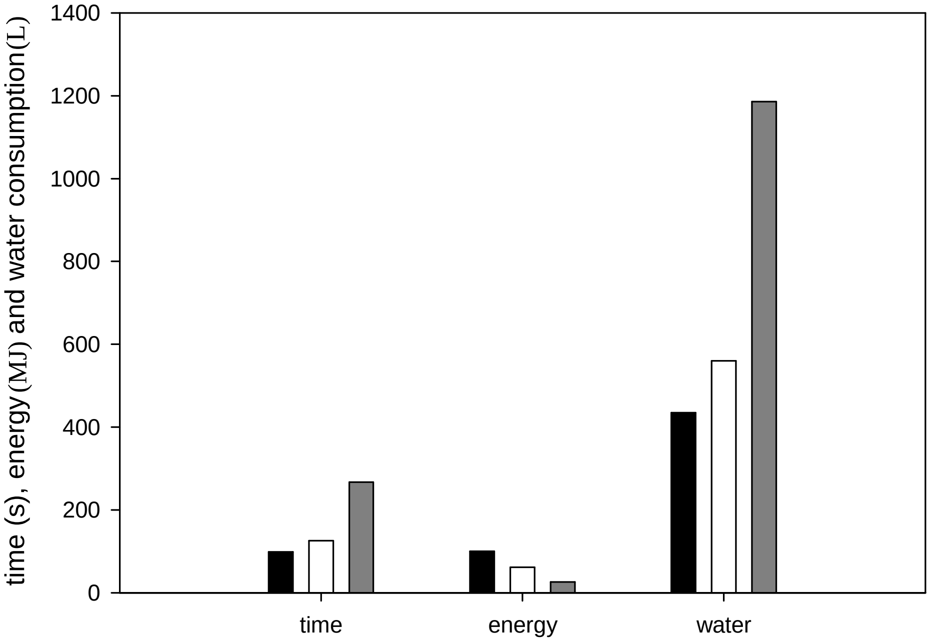

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**Table 1**

Second-order design matrix indicating the levels of coded and actual for two variables

| Runs | Coded levels of variables | | Actual level of variables[a] | |
|---|---|---|---|---|
| | Temperature **($X_1$)** | Flow rate **($X_2$)** | Temperature (°C) | Flow rate ($m^3$/h) |
| *Factorial points* | | | | |
| 1 | –1 | –1 | 27.0 | 7.86 |
| 2 | 1 | –1 | 63.0 | 7.86 |
| 3 | –1 | 1 | 27.0 | 14.54 |
| 4 | 1 | 1 | 63.0 | 14.54 |
| *Axial points* | | | | |
| 5 | $-\alpha$ (–1.414) | 0 | 19.5 | 11.20 |
| 6 | $+\alpha$ (+1.414) | 0 | 70.5 | 11.20 |
| 7 | 0 | $-\alpha$ (–1.414) | 45.0 | 6.48 |
| 8 | 0 | $+\alpha$ (+1.414) | 45.0 | 15.92 |
| *Center points* | | | | |
| 9 | 0 | 0 | 45.0 | 11.20 |
| 10 | 0 | 0 | 45.0 | 11.20 |

[a] Temperature and flow rate values are those values of the water used during cleaning.

**Table 2**

Mean values of first cleaning time (FCT), first energy consumption (FEC) and first water consumption (FWC), the significance of the regression models (*F* values) and the effects of temperature ($b_1$) and flow rate ($b_2$) on FCT, FEC and FWC measured at stage 1

| | ***1st stage*** | | | | | | *F* values and effect of independent variables | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Independent variables | | Dependent variables | | | | FCT | | FEC | | FWC | |
| Runs | Temp. (°*C*) | Flow rate ($m^3/h$) | FCT (s) | FEC (*MJ*) | FWC (*L*) | Source of variance | DF | *F* | DF | *F* | DF | *F* |
| *Factorial points* | | | | | | Model | 3 | 17.68[a] | 2 | 37.25[a] | 2 | 13.17[a] |
| | | | | | | *Linear* | | | | | | |
| 1 | 27.0 | 7.86 | 670 | 74.92 | 1463 | $b_1$ | 1 | 14.37[a] | 1 | 50.74[a] | 1 | 17.50[a] |
| 2 | 63.0 | 7.86 | 280 | 123.2 | 611.3 | $b_2$ | 1 | 33.45[a] | 1 | 23.77[a] | 1 | 8.85[b] |
| 3 | 27.0 | 14.54 | 168 | 34.90 | 681.3 | *Cross* | | | | | | |
| 4 | 63.0 | 14.54 | 90 | 73.69 | 365.8 | $b_{12}$ | 1 | 5.21[c] | - | BER[d] | - | BER[d] |
| *Axial points* | | | | | | *Quadratic* | | | | | | |
| | | | | | | $b_{11}$ | - | BER[d] | - | BER[d] | - | BER[d] |
| 5 | 19.5 | 11.20 | 332 | 22.64 | 1033 | $b_{22}$ | - | BER[d] | - | BER[d] | - | BER[d] |
| 6 | 70.5 | 11.20 | 145 | 104.3 | 451.1 | Residual | 6 | | 7 | | 7 | |
| 7 | 45.0 | 6.48 | 403 | 92.09 | 727.6 | *lack of fit* | 5 | 38.56 | 6 | 5.70 | 6 | 26.09 |
| 8 | 45.0 | 15.92 | 102 | 57.37 | 453.3 | *pure error* | 1 | | 1 | | 1 | |
| | | | | | | Total model | 9 | | 9 | | 9 | |
| *Center points* | | | | | | $R^2$ [e] | | 0.898 | | 0.914 | | 0.790 |
| | | | | | | *adj*-$R^2$ [f] | | 0.848 | | 0.890 | | 0.730 |
| 9 | 45.0 | 11.20 | 198 | 78.31 | 618.8 | *pred*-$R^2$ [g] | | 0.587 | | 0.813 | | 0.514 |
| 10 | 45.0 | 11.20 | 215 | 84.65 | 668.9 | *adeq pre* [h] | | 11.73 | | 15.49 | | 9.240 |

[a] $p \leq 0.01$.
[b] $p \leq 0.05$.
[c] $p \leq 0.1$.
[d] BER, the removed variable by "backward elimination regression" procedure.
[e] $R^2$, coefficient of determination.
[f] adjusted $R^2$.
[g] predicted $R^2$.
[h] adequate precision.

**Table 3**

Mean values of SCT, SEC and SWC, *F* values and the effects of temperature and flow rate on SCT, SEC and SWC measured at stage 2

| | $2^{nd}$ ***stage*** | | | | | | *F* values and effect of independent variables | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Independent variables | | Dependent variables | | | | SCT | | SEC | | SWC | |
| Runs | Temp. (°C) | Flow rate ($m^3/h$) | SCT (s) | SEC (*MJ*) | SWC (*L*) | Source of variance | DF | *F* | DF | *F* | DF | *F* |
| *Factorial points* | | | | | | Model | 5 | $63.01^{a}$ | 4 | $12.09^{a}$ | 3 | $77.24^{a}$ |
| | | | | | | *Linear* | | | | | | |
| 1 | 27.0 | 7.86 | 623 | 69.66 | 1360 | $b_1$ | 1 | $172.7^{a}$ | 1 | $0.20^{b}$ | 1 | $198.3^{a}$ |
| 2 | 63.0 | 7.86 | 180 | 79.18 | 393.0 | $b_2$ | 1 | $108.8^{a}$ | 1 | $26.37^{a}$ | 1 | $19.49^{a}$ |
| 3 | 27.0 | 14.54 | 232 | 48.19 | 940.9 | *Cross* | | | | | | |
| 4 | 63.0 | 14.54 | 42 | 34.39 | 170.7 | $b_{12}$ | 1 | $15.28^{c}$ | - | $\mathrm{BER}^{e}$ | - | $\mathrm{BER}^{e}$ |
| *Axial points* | | | | | | *Quadratic* | | | | | | |
| | | | | | | $b_{11}$ | 1 | $13.47^{c}$ | 1 | $8.59^{c}$ | 1 | $13.92^{a}$ |
| 5 | 19.5 | 11.20 | 438 | 29.87 | 1363 | $b_{22}$ | 1 | $12.75^{c}$ | 1 | $4.11^{d}$ | - | $\mathrm{BER}^{e}$ |
| 6 | 70.5 | 11.20 | 35 | 25.17 | 108.9 | Residual | 4 | | 5 | | 6 | |
| 7 | 45.0 | 6.48 | 384 | 87.75 | 693.3 | *lack of fit* | 3 | 111.4 | 4 | 61.69 | 5 | 100.4 |
| 8 | 45.0 | 15.92 | 83 | 46.68 | 368.9 | *pure error* | 1 | | 1 | | 1 | |
| | | | | | | Total model | 9 | | 9 | | 9 | |
| *Center points* | | | | | | $R^{2\ f}$ | | 0.988 | | 0.906 | | 0.975 |
| | | | | | | *adj*-$R^{2\ g}$ | | 0.972 | | 0.831 | | 0.962 |
| 9 | 45.0 | 11.20 | 140 | 55.37 | 437.5 | *pred*-$R^{2\ h}$ | | 0.911 | | 0.626 | | 0.926 |
| 10 | 45.0 | 11.20 | 145 | 57.09 | 451.1 | *adeq pre* [i] | | 21.70 | | 10.51 | | 23.97 |

[a] $p \leq 0.01$.

[b] The term was a hierarchical term added after BER (backward elimination regression) process.

[c] $p \leq 0.05$.

[d] $p \leq 0.1$.

[e] BER, the removed variable by “backward elimination regression” procedure.

[f] $R^2$, coefficient of determination.

[g] adjusted $R^2$.

[h] predicted $R^2$.

[i] adequate precision.

**Table 4**

Mean values of TCT, TEC and TWC, *F* values and the effects of temperature and flow rate on TCT, TEC and TWC measured at stage 2

| | ***Total cleaning stage (1st + 2nd regions)*** | | | | | | *F* values and effect of independent variables | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Independent variables | | Dependent variables | | | | TCT | | TEC | | TWC | |
| Runs | Temp. (°*C*) | Flow rate ($m^3/h$) | TCT (s) | TEC (*MJ*) | TWC (*L*) | Source of variance | DF | *F* | DF | *F* | DF | *F* |
| *Factorial points* | | | | | | Model | 3 | 23.37[a] | 3 | 20.67[a] | 3 | 29.29[a] |
| | | | | | | *Linear* | | | | | | |
| 1 | 27.0 | 7.86 | 1293 | 144.6 | 2823 | $b_1$ | 1 | 30.25[a] | 1 | 17.94[a] | 1 | 68.75[a] |
| 2 | 63.0 | 7.86 | 460 | 202.4 | 1004 | $b_2$ | 1 | 34.69[a] | 1 | 33.78[a] | 1 | 14.38[a] |
| 3 | 27.0 | 14.54 | 400 | 83.09 | 1622 | *Cross* | | | | | | |
| 4 | 63.0 | 14.54 | 132 | 108.1 | 536.4 | $b_{12}$ | 1 | 5.16[c] | - | BER[d] | - | BER[d] |
| *Axial points* | | | | | | *Quadratic* | | | | | | |
| | | | | | | $b_{11}$ | - | BER[d] | 1 | 10.30[b] | 1 | 4.74[c] |
| 5 | 19.5 | 11.20 | 770 | 52.51 | 2396 | $b_{22}$ | - | BER[d] | - | BER[d] | - | BER[d] |
| 6 | 70.5 | 11.20 | 180 | 129.5 | 560.0 | Residual | 6 | | 6 | | 6 | |
| 7 | 45.0 | 6.48 | 787 | 179.8 | 1421 | *lack of fit* | 5 | 76.54 | 5 | 9.20 | 5 | 32.28 |
| 8 | 45.0 | 15.92 | 185 | 104.1 | 822.2 | *pure error* | 1 | | 1 | | 1 | |
| | | | | | | Total model | 9 | | 9 | | 9 | |
| *Center points* | | | | | | $R^2$ [e] | | 0.921 | | 0.912 | | 0.936 |
| | | | | | | *adj-*$R^2$ [f] | | 0.882 | | 0.868 | | 0.904 |
| 9 | 45.0 | 11.20 | 360 | 141.8 | 1120 | *pred-*$R^2$ [g] | | 0.723 | | 0.717 | | 0.812 |
| 10 | 45.0 | 11.20 | 338 | 133.7 | 1056 | *adeq pre* [h] | | 12.82 | | 12.36 | | 14.77 |

[a] $p \leq 0.01$.

[b] $p \leq 0.05$.

[c] $p \leq 0.1$.

[d] BER, the removed variable by “backward elimination regression” procedure.

[e] $R^2$, coefficient of determination.

[f] adjusted $R^2$.

[g] predicted $R^2$.

[h] adequate precision.

**Highlights**

> Cleaning in place protocol was optimised in terms of cleaning inputs

> A two step cleaning in place protocol was proposed for industrial cleaning processes

> The first was application of water at ambient temperature in the 1st step

> The second was application of hot water in the 2nd step at the same velocity

> The proposed protocol remarkably decreased energy consumption and waste water amount