PULSED FLOW CLEANING OF WHEY PROTEIN FOULING LAYERS

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ABSTRACT

This work reports the use of intermittent pulsing superimposed on a slow steady flow to enhance the rate of cleaning of a model food soil, namely a whey protein deposit, in a well characterised flow geometry.

Whey protein deposits were generated by recirculating 3.5 wt% WPC solutions through an electrically heated annular test section and were then cleaned using recirculating solutions of 0.5 wt% NaOH, simulating industrial cleaning-in-place (CIP) operations. Protein removal was monitored by local measurements of fouling resistance (at low heating power) and a total protein assay. Bulk flow velocities of 0.1 – 0.3 m/s and waviness ratios (amplitude of velocity pulse/baseline flow velocity) of 0.33 – 5.0 were studied at room temperature. Cleaning at these temperatures is a relatively slow process and allows the effect of flow regime to be followed easily.

The resulting cleaning profiles showed that protein was removed in two stages, viz.: (*i*) an initial rinsing stage, followed by (*ii*) protein swelling and gradual dissolution. Only the rinsing stage was observed in the absence of NaOH, at a noticeably lower rate. Slow flow pulsing enhanced the overall cleaning rate, which exhibited a noticeable increase when the waviness of the flow exceeded unity and backflow of the fluid occurred. The results are discussed in terms of cleaning enhancement as a function of extra flow rate and extra energy input to the process.

INTRODUCTION

The formation of fouling layers during the thermal treatment of milk and milk products is a severe problem for the dairy industry. These layers can lead to a drastic increase in resistance to heat transfer, thereby decreasing the thermal efficiency of equipment such as heat exchangers. Furthermore, the stringent requirements for quality and hygiene in dairy processes require regular and effective cleaning of production lines. Costs are thereby incurred for detergents, rinse water, disposal of spent solutions and energy. This paper considers one method for optimising the cleaning process with respect to duration and energy consumption.

The use of slow (~ 1 Hz) flow pulsations imposed on a steady flow of liquid has been shown to enhance shear stresses imposed on a surface and to mitigate fouling (Augustin and Bohnet, 2001) or enhance cleaning (Gillham *et al*., 2000). Flow pulsing is an attractive technology for systems where the liquid is too viscous to achieve turbulent flows, or where the inventory of fluid is to be minimised.

FUNDAMENTALS

Milk fouling

Milk is composed of water, fat, proteins, sugar, minerals and micro-organisms. The average percentage of proteins is 3.3%, with 2.6% casein and 0.7% whey. Burton (1968) classified milk fouling deposits as types A and B, as shown in Table 1, with higher temperatures promoting the phosphate-rich, hard type B. The fouling layers generated from whey protein solutions in this work exhibited type A characteristics (Gillham *et al*., 2000).

Table 1 Classification of milk fouling deposits (Burton, 1968)

	Process parameters	**Composition**	**Appearance**
Type A	Pasteurisation $T \leq 100°C$	Protein: 50-60% Minerals: 30-35% Fat: 4-8%	Soft and voluminous structure; white or cream-colored
Type B	Ultra-heat treatment $T = 110°C – 140°C$	Protein: 15-20% Minerals: 70% Fat: 4-8%	Brittle and porous; grey

The whey protein β-lactoglobulin plays a significant role in the fouling process. At 40°C and pH 7 it exists as a dimer; increasing temperature results in decomposition of non-covalent bonds, which reactivate mercaptan groups. The protein then is able to react with other proteins with active disulphide groups. Above 70°C, there is a large increase in activation energy as irreversible chain reaction of denatured β-lactoglobulin with similar proteins forms aggregates which subsequently deposit (Lalande *et al*., 1985).

Deposition of denatured proteins can be described as two successive processes: the adhesion of precipitated protein on a free surface, in this case the heat exchanger wall, and the growth of the layer by reactions between protein polymers. A higher rate of denaturation in the bulk solution can decrease deposition significantly (Dannenberg, 1986), so preheaters are often installed.

Cleaning of whey protein deposits

Cleaning of production lines in the food industry is frequently performed by cleaning-in-place (CIP) operations, involving:

1. Rinsing to loosen the deposit
2. Cleaning phase
3. Rinsing to remove residues and detergents.

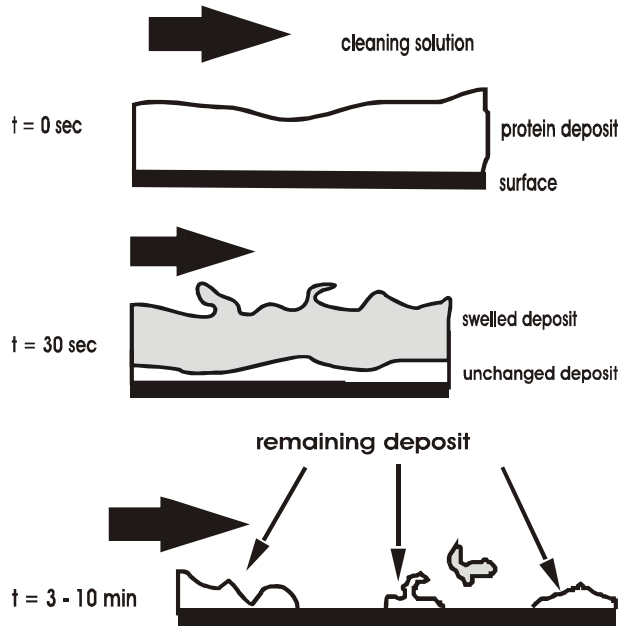

Fig. 1 Schematic of stages during cleaning

Table 2 Cleaning mechanisms

Chemical reaction of the detergent → Molecular modifications	**Physical reaction of the fluid flow → Forces**
• Reaction/modification: swelling of the layer • Detachment: Aggregates dissolve in the detergent • Ageing: Structural change of the layer with time	• Mass transport: Transport of detergent to the layer's surface; conveying of reaction products from the surface • Diffusion: Detergent in the layer; dissolved proteins in the bulk • Shear stress: Abrasion and conveying of dissolved particles

Cleaning of protein fouling deposits is a complex process with interactions between surface, deposit and detergent. Stainless steel is normally used for the surfaces in contact with the product and CIP solutions are often based on dilute sodium hydroxide solutions.

Gillham et al. (2000) described the cleaning phase in two steps as shown in Fig. 1:

(a) The detergent diffuses in the protein layer and the deposit swells in a reaction zone. These gel-like structures detach and are removed as aggregates by the shearing action of the fluid or by dissolution.

(b) Towards the end of the process, the remaining swollen protein material is removed by the shear action of the flow.

The shear strength of the deposit and the adhesion force between layer and the surface determine the cleaning behaviour of the complete system.

According to Grasshoff (1998) the driving forces are the chemistry of the detergent/cleaning agent and the physical effect of the fluid flow. Sodium hydroxide modifies the structure of the deposit by breaking peptide bonds and makes the deposits more solubilisable. The fluid flow supplies thermal and mechanical energy. The mechanisms are listed in Table 2. The dominant mechanism (and therefore source of energy) varies with the nature of the deposit. In this case there is a transition between chemical processes initially to mechanical processes later.

Fig. 2 shows a characteristic cleaning curve in terms of cleaning rate (protein removal flux). In phase I chemical reaction and swelling take place. After a constant maximum value of *P*, (phase II), the cleaning rate decreases slowly in phase III, which represents the removal of material remaining on the surface.

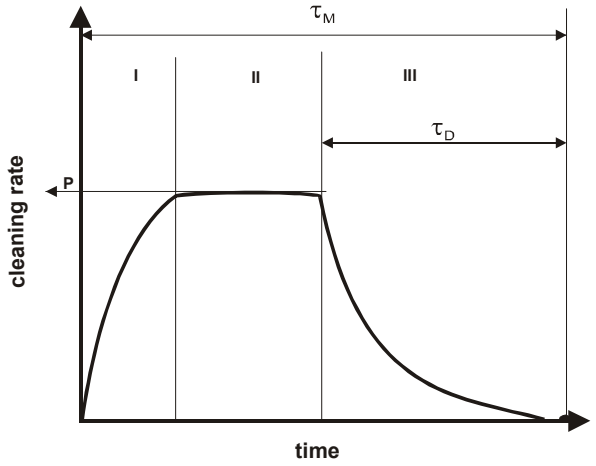

Fig. 2 Characteristic cleaning curve

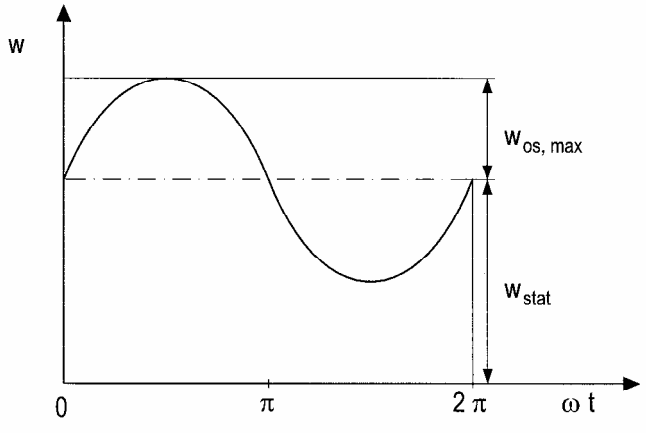

Fig. 3 Steady flow fluid and oscillating components of pulsating flow

τ_M is the time required for complete cleaning. The length of the decay phase τ_D is defined as the time taken for the cleaning rate to decay from the maximum to a value of zero. The specific protein concentration can be calculated from the area underneath the cleaning curve.

Pulsating flow

Flow velocity has a substantial influence on fouling behaviour (Krause, 1993). Higher velocities tend to break up the fouling layer due to an increase in shear stresses acting on the surface. A pulsating flow, as hypothesized, would create momentary, large accelerations of the liquid flow around the fouling layer, thus resulting in an increase in removal of the layer. The directional change of the liquid flow would affect both the deposition rate, due to greater rates of mass transport, and removal of the fouling layer (Bohnet, 1987). In the cleaning process the mass transport of the cleaning agent to the deposit layer surface is enhanced.

A pulsating flow consists of a mean steady flow fluid velocity w_{stat} and a superimposed oscillating component w_{os} (Fig. 3). The mean velocity for an oscillation interval t_{os} is defined as

$$\overline{w} = \frac{1}{t_{os}} \int_0^{t_{os}} w(t)\,dt \qquad \text{with}$$

$$w(t) = w_{stat} + w_{os} = w_{stat} + w_{os,max} \cdot \sin(\omega t) \qquad (1)$$

The dimensionless waviness W describes the intensity of the pulsation as follows

$$W = \frac{w_{os,max}}{\overline{w}} \qquad (2)$$

According to theory (Dettmann, 1991) a waviness of value $W > 1$ leads to a temporary flow reversal in the proximity of the wall as shown in Fig. 4. Higher waviness can lead to the separation of the viscous sublayer and to the formation of eddies. Furthermore, due to the variable ratio of inertial and frictional forces the 'annular effect' is characteristic for pulsating flow. Here the maximum velocity does not necessarily occur in the centre of the pipe, but can also occur near the wall, giving large shear rates and high wall shear stresses.

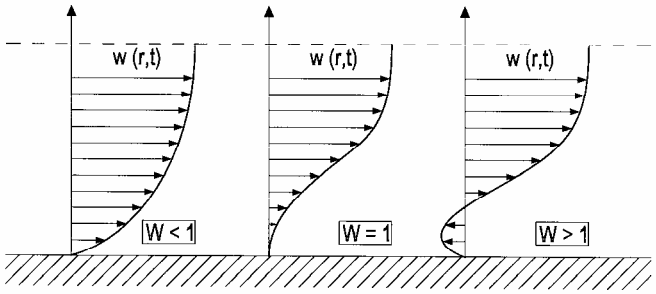

Fig. 4 Velocity profiles of pulsating flow for different waviness

EXPERIMENTS

Experimental set up

A mobile test unit including an annular tube heat exchanger was used in order to validate optimal cleaning techniques. Fig. 5 shows the flow sheet of the fouling test rig. A detailed description of its modules can be found in Förster *et al*. (1999).

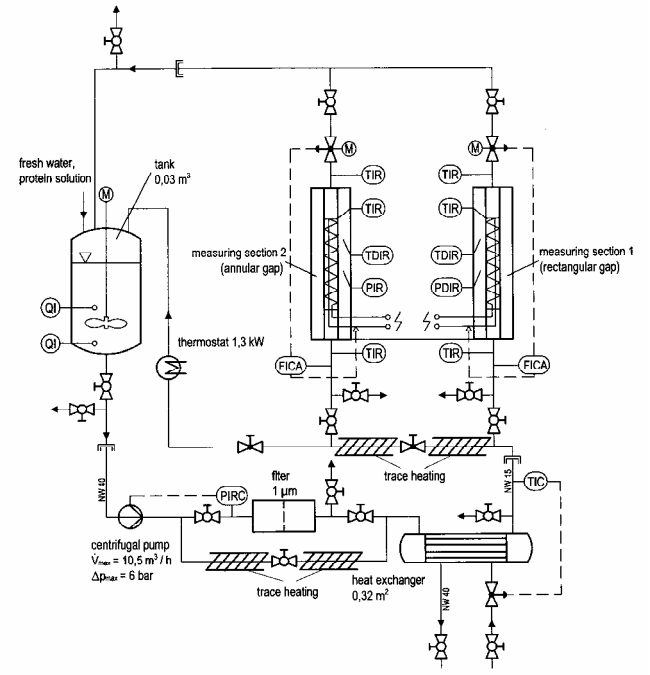

Fig. 5 Test rig

The modular design of the test unit allows a simple disassembly of the three modules:

- Module 1 contains a storage tank for the protein or cleaning solution with a stirrer to maintain a homogeneous solution. The thermostat was used as a preheater for the fouling runs only.
- Module 2 consists of a centrifugal pump and a filter which was bypassed in these experiments. The heat exchanger guarantees a constant inlet temperature at module 3. All pipes were fitted with trace heating .
- Module 3 includes two test sections, control valves and flow meters. The first test section consists of an annular tube heat exchanger, whereas the plate heat exchanger in the second test section is used for reference measurements. The inner section of the annulus shown in Fig. 6 is made from stainless steel and can be electrically heated with a maximum heat flux of 110 kW/m^2. Thermocouples are mounted inside the heating element at evenly distributed positions and measure the temperature of the exterior wall. With the bulk temperature known, this allows determination of the fouling resistance. The outer annulus has an i.d. of 25 mm and is made from silicate glass which ensures good insulation whilst also enabling visual observation of the fouling and cleaning processes.

A pulsator can superimpose an oscillating flow component on the steady flow fluid fluid flow through either measuring section. The pulsator is a reciprocating pump with a shut inlet. A wide range of piston strokes can be utilized to adjust the amplitude of the velocity pulse: an impulse generator and an integrated counter can vary both the stroke amplitude and the number of strokes per unit time.

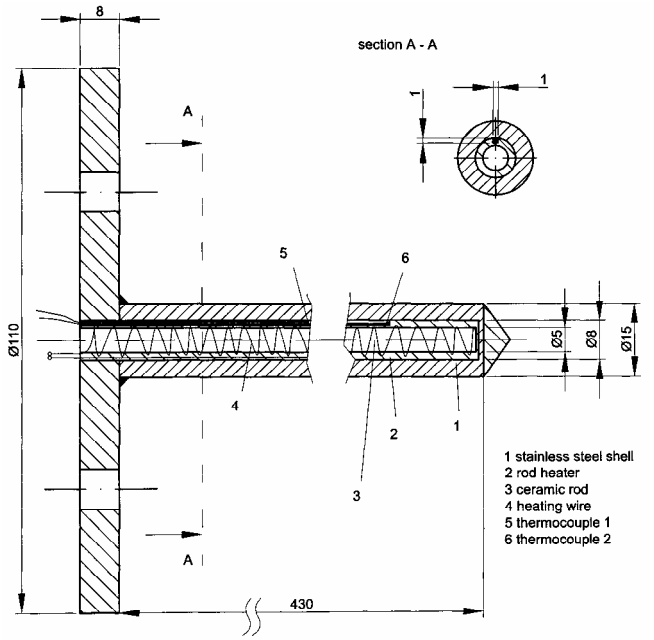

Fig. 6 Heating element located in measuring section 2 (all dimensions in mm)

Fouling runs

In order to conduct cleaning experiments, reproducible protein deposits with an even distribution over the surface and homogenous composition have to be generated in fouling runs.

Table 3 Operating conditions in fouling runs

Protein solution	20 L; 3.5 wt-%
Bulk fluid temperature	≤ 70°C
pH-value	6.0
Heating element temperature	90-110°C
Thermostat temperature	85°C
Trace heating temperature	≈ 95°C
Heat flux	16 kW/m^2
Fluid velocity	0.1 m/s
Time of experimentation	max. 80 min

A 3.5 wt% solution of a whey protein concentrate (35 wt% protein) in deionised water was mixed in the tank at room temperature with a stirrer until the protein was totally dissolved. The pH-value was set at pH 6 with 0.1 molar hydrochloric acid. The solution was then pumped through the preheated test rig back to the tank, the heating elements were switched on and the measuring procedure started. The operating conditions are listed in Table 3.

The series of fouling resistance vs. time plots in Fig. 7 shows the reproducibility obtained over a number of randomly selected fouling runs. The deviation lies in a narrow range. Fouling starts right away and once the whole heat transfer surface is covered with a thin layer, the slope of the fouling curve decreases to a constant value. The experiments were stopped after reaching a fouling resistance of approximately $8 \cdot 10^{-4}$ m^{2}K/W, which corresponds to a thickness of the layer of circa 1 mm (Fig. 8).

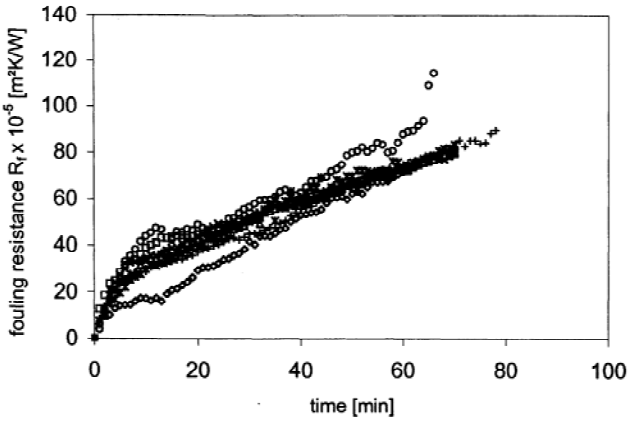

Fig. 7 Typical fouling resistance profiles during fouling

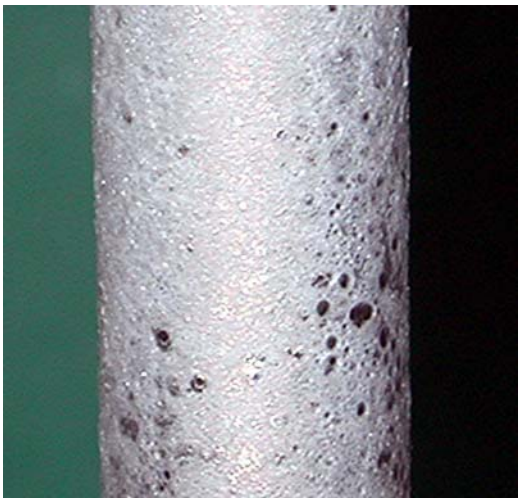

Fig. 8 Whey protein fouling layer

Table 4 Composition of the fouling layer

	Fouling layer (measured)	**Whey protein concentrate (manufacturer's data)**
Organic	82±3%	Protein 35±2% Lactose 45±2 % Σorg ≈ 84±4% Fat max. 4% Water max. 5%
Ash	Max. 21%	Max. 8%

The composition of the fouling layer was analysed in separate experiments. A sample was taken from the layer, suspended in deionised water in a crucible and dried for 48 h at 105°C in a drying oven. The dried crucibles were than combusted for 2 h at 550°C: the loss on ignition gives the organic content of the sample. This is compared with the product specifications given by the manufacturer in Table 4 and shows that the fouling layer composition is comparable with the whey protein solution.

After each fouling run the heating element with the protein layer was removed from the flow loop and stored in deionised water to avoid dehydration. The test rig was then cleaned and rinsed with deionised water several times.

Cleaning runs

For cleaning runs the fouled heating element was reinstalled and the test rig filled with deionised water into which sodium hydroxide was dissolved.

Table 5 Cleaning run operating conditions

Cleaning solution	20 L; 0.5 wt-% NaOH
Fluid temperature	≈ 18°C
pH-value	12
Heat flux	≈ 4 kW/m^2
Steady flow fluid velocity	0.1 – 0.3 m/s
Oscillating fluid velocity	0.1 – 0.6 m/s
Time of experimentation	40 min

The heat of dissolution evolved was dissipated by circulating the solution through a plate heat exchanger connected to a cooling circuit. The measuring section was by-passed until a constant fluid temperature was reached. A low heat flux was then set on the heating element in order to be able to monitor the fouling resistance during the cleaning run. The flow was then admitted to the measuring section, the oscillation applied and the measuring procedure started. The operating conditions are listed in Table 5. After each experiment the solution was neutralised before disposal and the apparatus was cleaned and rinsed.

Over the first 10 min a liquid sample was taken at 1 min intervals from the return line to the tank, and thereafter every 2 min. The protein content in the cleaning solution and thus the detached mass was quantified with the modified Bradford assay described by Gillham et al. (2000), which is a photometric method employing Coomassie Blue dye as an indicator. From the protein deposit mass m the discrete cleaning rate r_D and the main cleaning rate r_M could be calculated:

$$r_D = \frac{\Delta m}{A \cdot \Delta t} \tag{3}$$

$$r_M = \frac{m}{A \cdot \tau_M} \tag{4}$$

The fouling resistance R_f was calculated from the difference between the fouled and the clean heat transfer coefficient.

$$R_f = \frac{x_f}{\lambda_f} = \frac{1}{k_f} - \frac{1}{k_0} \tag{5}$$

For a cleaning run it was assumed that a complete cleaning was achieved, and the ´clean´ heat transfer coefficient k_0 was calculated using the wall temperature observed at the end of the run.

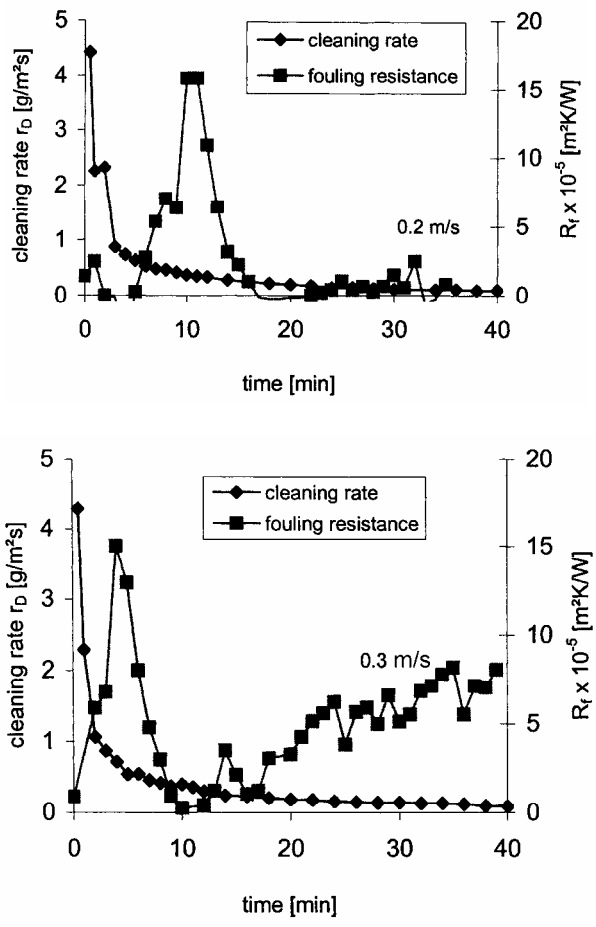

Fig. 9 Fouling resistance and cleaning rate profiles for different steady flow fluid velocities without pulsation

Examples of cleaning rate and fouling resistance profiles are displayed in Fig. 9 for different steady flow fluid velocities without pulsation. Phases I and II of the characteristic cleaning curve in Fig. 2 are missing. Phase III, characterised by the exponential decrease of the cleaning rate, can be clearly seen. The absence of the increase of the cleaning rate in phase I, Fig. 2, is explained by parallel rinsing process, removing loosely held material. Xin *et al.* (2002) reported similar phenomena for a whey protein gel, with a maximum next to the point of origin.

Initially the fouling resistance increases due to the swelling of the deposit, and then decreases as the layer is removed. The subsequent increase observed after 20 min with the 0.3 m/s test arose from fluctuations of the power

supply at low heat flux. At the higher flow velocity a maximum value of the fouling resistance is reached earlier as a result of faster cleaning because of higher turbulence. It is noteworthy that the absolute value is identical. The different positions of the peaks, comparing the curves for the cleaning rate and the fouling resistance, arises from a difference in measuring scale of swelling and removal. The fouling resistance reaches its final value earlier than the chemically determined cleaning rate because the former is obtained from a local measurement of wall temperature, whereas the assay yields the overall cleaning rate, *i.e.* an integral value.

Table 6 summarises the data obtained from 14 different cleaning runs with pulsating flow. The last two runs in the Table were carried out without the addition of sodium hydroxide. Two parameters are evidently influencing the cleaning process: the waviness and the maximum velocity. They cannot be manipulated independently in this apparatus. Table 6 is arranged in ascending order of the waviness W. The cleaning rate r_M is also increasing, but with deviations caused by higher maximum velocities.

Table 6 Summary of cleaning data, in order of W increasing

W	w_{stat}	w_{os}	w_{max}	τ_M [min]	r_M [g/m²s]	*Run*
0,3	0,3	0,1	0,4	24	0,1958	*12*
0,7	0,3	0,2	0,5	22	0,2155	*13*
1	0,1	0,1	0,2	22	0,1431	*2*
1	0,2	0,2	0,4	22	0,2399	*6*
1	0,3	0,3	0,6	26	0,2398	*14.1*
1	0,3	0,3	0,6	18	0,2365	*14.2*
1	0,3	0,3	0,6	14	0,1904	*14.3*
1	0,3	0,3	0,6	14	0,1745	*14.4*
1,5	0,2	0,3	0,5	16	0,3770	*7*
2	0,2	0,4	0,6	14	0,2665	*8*
3	0,1	0,3	0,4	24	0,1950	*3*
3	0,2	0,6	0,8	14	0,4596	*9*
4	0,2	0,8	1	10	0,4125	*10*
5	0,1	0,5	0,6	12	0,4143	*4*
0	0.3	0	0.3	>40	0.04	*15*
2.7	0.3	0.8	1.1	>40	0.04	*16*

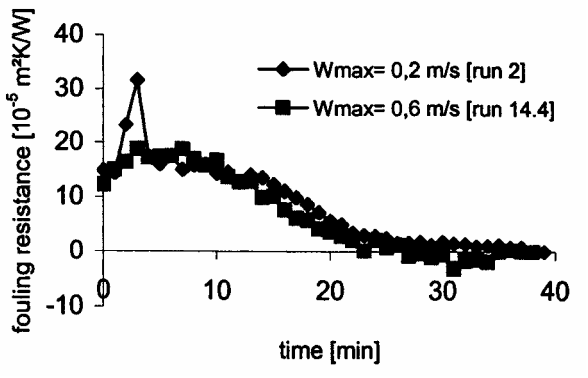

Fig. 10 Fouling resistance for different maximum velocities

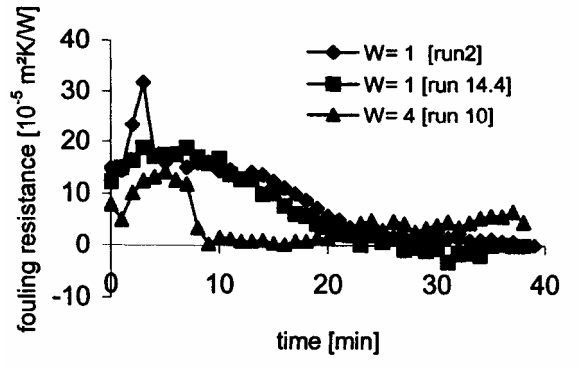

Fig. 11 Fouling resistance for different waviness

The effect of the maximum velocity can be seen in Fig. 10. The higher velocity leads to a lower peak in the fouling resistance, which indicates that there is less swelling of the deposit because of the superimposed removal. Furthermore, the final value of zero is reached earlier. Comparing these two runs with a run of higher waviness, as shown in Fig. 11, shows another positive effect on the cleaning behaviour. The maximum peak is lowered again and the final value of ´total cleanness´ is achieved much earlier.

The cleaning time and rate are also controlled by the interplay of waviness and maximum velocity, as displayed in Figs. 12 and 13. Both show the expected behaviour trends: the cleaning time is decreasing and the cleaning rate increasing with rising maximum velocity and waviness, respectively.

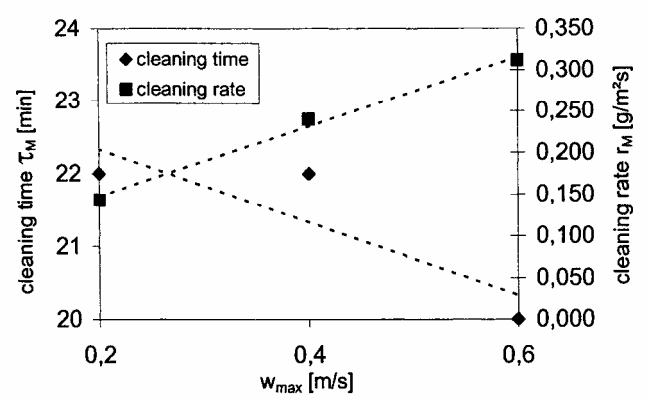

Fig. 12 Cleaning time and rate depending on maximum velocity for constant waviness $W = 1$

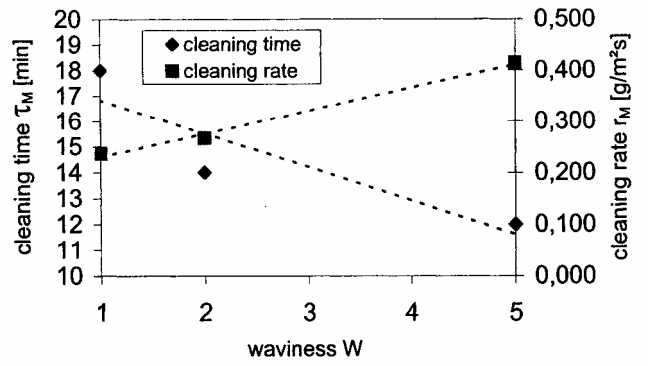

Fig. 13 Cleaning time and rate depending on waviness for constant maximum velocity $w_{max} = 0.6$ m/s

The relationship between cleaning time and waviness for all experiments is plotted in Fig. 14. Although the data points represent runs with different maximum velocities, a decreasing tendency of the cleaning time with increasing waviness is obvious. The required time declines from approximately 25 min to 10 min. Also evident is the sudden drop at a waviness $W = 1$, which reproduces the physical effect of the beginning temporary flow reversal in the proximity of the wall.

Finally two experiments (runs 15 and 16 in Table 6) were carried out without the addition of sodium hydroxide, one with high and one without pulsating flow. In both cases, visual and analytical observation indicated almost no detachment of the deposit. The shear force introduced by the fluid flow is therefore insufficient to remove the protein layer by itself.

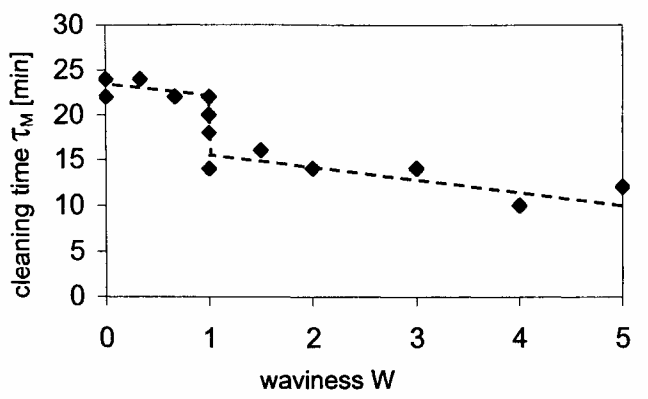

Fig. 14 Cleaning time versus waviness

CONCLUSIONS

The experimental results illustrate the potential for improving the cleaning process of whey protein fouling layers by the application of pulsating flow. The cleaning time could be shortened by two and a half times: this result, at low temperature, compares favourably with the result reported by Gillham *et al*. (2000).

Waviness and maximum velocity proved to be the crucial parameters for the effectiveness of the process. A higher maximum velocity leads to less swelling of the deposit because of the superimposed removal, and a clean surface is obtained earlier. Higher waviness shows another positive effect on the cleaning behaviour: the maximum peak is lowered again and the final value of ´total cleanness´ is achieved much earlier.

Reviewing all the results indicates that the waviness is effecting the cleaning process more strongly than the maximum velocity. Operating conditions with $W > 1$ are recommended, because of the high turbulence in the proximity of the wall due to the effect of temporary flow reversal. This demonstrates the potential of the pulsation technique.

Experiments without any detergent showed that the chemical effect of the detergent is predominantly the precondition for the positive physical effect of the pulsating flow. The understanding of the physical and chemical interactions of pulsating flow and detergent, *i.e.* its impact on shear strength and adhesion, is a crucial premise for the development of a comprehensive model describing the cleaning process.

ACKNOWLEDGEMENTS

This work was performed as part of a collaborative project funded by the British Council [Project ARC 1197] and the German Academic Exchange Service (DAAD).

NOMENCLATURE

A	heating surface area, m^2
k	heat transfer coefficient, $Wm^{-2}K^{-1}$
m	deposited mass, kg
P	maximum cleaning rate, $kgm^{-2}s^{-1}$
R_f	fouling resistance, m^2KW^{-1}
r_D	discrete cleaning rate, $kgm^{-2}s^{-1}$
r_M	main cleaning rate, $kgm^{-2}s^{-1}$
T	temperature, °C
t	time, s
W	waviness, -
w_{max}	maximum velocity, $m\,s^{-1}$
w_{os}	oscillating velocity, $m\,s^{-1}$
w_{stat}	steady flow fluid velocity, $m\,s^{-1}$
x_f	thickness of the fouling layer, m
λ_f	thermal conductivity of the fouling layer, $Wm^{-1}K^{-1}$
τ_M	cleaning time, h
τ_D	cleaning time of phase III, h

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