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FOULING AND CLEANING OF MODIFIED STAINLESS STEEL PLATE HEAT EXCHANGERS PROCESSING MILK PRODUCTS

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ABSTRACT

Fouling of heat exchangers in dairy industries is still quite a severe problem both technically and economically. Altering the surface properties of the heating surfaces would be a way of solving this issue.

Modified steel surfaces were tested in an Alfa Laval V2 plate heat exchanger throughout dairy product sterilization. The behavior was analyzed for 8 different surface treatments, such as coatings (Diamond Like Carbon [DLC], Silica, SiO_x, Ni-P-PTFE, Excalibur[®], Xylan[®]) and ion implantation (SiF⁺, MoS₂).

All fouling and cleaning experiments were carried out in standard and well-controlled operating conditions. After fouling, no significant difference could be seen between all the modified steels and the reference by statistical variance analysis. Cleaning efficiency of Ni-P-PTFE appeared significantly the best. It could be suggested that the free surface energy plays a predominant role and the roughness a minor role in the level of fouling and cleaning efficiency.

INTRODUCTION

Plate Heat Exchangers (PHEs) are very common in dairy industries due to their ease of maintenance and cleaning, their compact designs and their excellent heat transfer coefficient characteristics required for thermal sterilization/pasteurization purposes (Shah and *al.*, 1988). However, fouling of plate heat exchanger is a severe problem both technically and economically. It causes significant increases in capital and operating costs (Sandu and Lund, 1983). For example, in the dairy industry, the yearly estimated total fouling costs in the Netherlands are approximated to exceed 40 million US\$ (Visser and Jeurink, 1997).

Frequent cleaning of the plant is needed for both microbiological reasons and to restore PHE heat transfer characteristics, i.e. to remove the additional heat resistance of the fouling layer and to reduce the pressure drop in the process plant. Complex Cleaning-In-Place (CIP) techniques have been developed empirically (Romney, 1990 ; Bird & Fryer, 1991). Two types of CIP treatments are mainly found in milk processing (Timperley and Smeulders, 1987):

1- Two-stage cleaning, using alkali, commonly sodium hydroxide and an acid wash of nitric or phosphoric acid.

2- Single-stage cleaning, using formulated detergents containing wetting and other surface agents as well as chelating compounds.

Selecting the correct cleaning strategy requires an understanding of milk fouling, which has been studied for a number of years. The composition of the milk deposit is now well known (Fryer *et al.*, 1996). The key roles are played by proteins, especially β -Lactoglobulin. Lalande *et al.* (1985) showed that the heat denaturation of this β -Lactoglobulin protein governs the milk deposit formation on the heat transfer area when the temperature is below 90 °C. This has been confirmed by many investigators (Jeurink, 1996 ; De Jong, 1997 ; Visser *et al.*, 1997).

A number of authors have modeled milk fouling in PHEs based on a simple representation of the process hydrodynamics. Delplace, Leuliet and Tissier (1994) performed experiments in a PHE with complex flow arrangements consisting of 13 plates. The overall heat transfer coefficient and pressure drop were measured as a function of time. The temperature profiles for each channel were determined based on numerical simulation. These profiles explained the uneven deposit formation in each channel. Delplace and Leuliet (1995) studied the milk fouling of several PHE flow arrangements by measuring both the heat transfer coefficient and dry mass of deposit. A predictive model of deposit mass in each channel was proposed based on temperature profiles and protein denaturation.

Investigation of anti-fouling techniques has received much attention in the past and a variety of chemical and mechanical methods have been suggested to reduce the formation of a deposit on heat transfer surfaces. However, mechanical methods, such as pipe inserts or circulating sponge balls, are costly and usually limited to the inside of pipe-like geometries. For PHEs, only chemical mitigation is possible. Unfortunately, using chemical additives may cause undesired product contamination or have adverse effects on the environment. The use of magnetic, electric, radiation or catalytic treatments (Donaldson and Grimes, 1988) have not yet given consistent results.

Altering the surface properties of the heating surfaces would be a way of finding a solution. Recently several new techniques such as direct ion implantation, magnetron sputtering, dynamic mixing, plasma enhanced vapor deposition and auto-catalytic Ni-P-PTFE coating (Zhao and

al., 2002) have been considered as being of interest in fouling reduction. However, very few studies have been successful in applying such novel surface treatments. Müller-Steinhagen and Zhao (1997) investigated the influence of SiF_4^+ ion implanted stainless steel, which significantly reduces CaSO_4 scale formation during pool boiling.

The aims of this work are to increase the productivity by lengthening the processing time and substantially reducing the time needed for cleaning the heat exchangers, thereby minimizing resource consumption and the use of chemicals.

Different surface treatments including Diamond Like Carbon [DLC], Silica, SiO_x , Ni-P-PTFE, Excalibur[®], Xylan[®] and ion implantation (SiF_4^+ , MoS_2) were tested.

Fouling and cleaning references on non-modified steel plates of a V2 plate heat exchangers were established to allow comparisons with the fouling and cleaning behavior of the modified steels.

The decrease in the overall heat transfer coefficient and the increase in the pressure drop were used for comparison, being a direct consequence of the fouling of the heated surfaces. The quantity of denatured β -lactoglobulin after the heat treatment was calculated.

MATERIALS

1- Pilot plant

Fouling experiments were carried out on the pilot plant presented in Fig. 1. It consists of 2 stirred tanks, 2 test heat exchangers (PHE1 and PHE2) connected to 2 heating units and a cooler (PHE3).

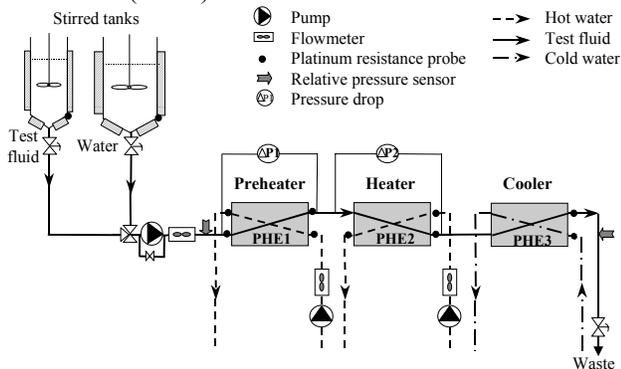


Fig. 1 Schematic drawings of the test rig

The pump used for the milk circuit was an hygienic volumetric pump (Moineau H series (PCM, France), $Q_{\max}=1.52 \cdot 10^{-4} \text{ m}^3/\text{s}$, $P_{\max}=9 \text{ Bar}$). For hot water a centrifugal pump was deployed (Sterling Fluid systems SIHI, France, CEHA 1203, $Q_{\max}=3 \cdot 10^{-4} \text{ m}^3/\text{s}$, $P_{\max}=5 \text{ Bar}$). All exchangers came from Alfa Laval Vicarb. PHE1 and PHE2 are of V2 type. The plates are 0.256 m in length and 0.078 m in width. Their hydraulic diameter is 0.005 m.

Flow arrangements in the heating parts were chosen to avoid any problem of flow maldistribution (one channel per pass) along with quite a wide temperature range (7 channels). The pre-heating (PHE1) and the heating (PHE2) sections represented in Fig. 2 were composed of 10 and 15 V2 plates, respectively. The effective exchange areas are 0.160 m^2 and 0.260 m^2 respectively. In the pre-heater and the heater, counter current mono-channel per pass was the flow arrangement chosen.

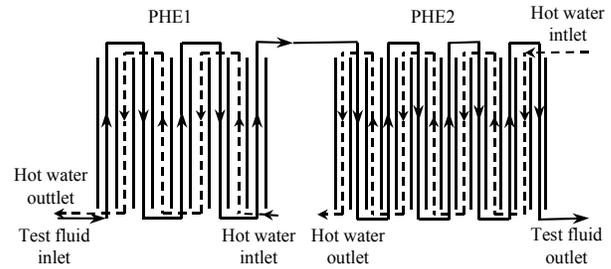


Fig. 2 Plate heat exchanger flow arrangement

Various sensors were installed such as flow meters, differential pressure sensors and platinum resistance probes in order to characterize the thermal and hydraulic performances including pressure drop and overall heat transfer coefficient.

Four platinum resistance probes were necessary to measure the inlet [i] and outlet [o] temperatures of the test fluid [f], as well the temperatures of the hot water [w]. The thermal balance was calculated as described in Eq. (1, 2, 3) and so the overall heat transfer coefficient (α_g) is known.

$$q = \dot{m}_w \cdot C_{p_w} \cdot (\theta_{iw} - \theta_{ow}) = \dot{m}_f \cdot C_{p_f} \cdot (\theta_{of} - \theta_{if}) \quad (1)$$

$$q = F_t \cdot \alpha_g \cdot S_{tot} \cdot \Delta\theta_{ln} \quad (2)$$

$$\text{with } \Delta\theta_{ln} = \frac{(\theta_{ow} - \theta_{if}) - (\theta_{iw} - \theta_{of})}{\ln\left(\frac{\theta_{ow} - \theta_{if}}{\theta_{iw} - \theta_{of}}\right)} \quad (3)$$

F_t was arbitrarily chosen as equal to 1, all of the trials being carried out in the same operating conditions.

During the fouling process, the pressure drop (ΔP) increased, ($\Delta\theta_{ln}$) increases and also α_g decreased with time. The parameters (ΔP , α_g) taken together, would allow us to estimate the fouling formation on the heated surfaces. During the cleaning process, the fouling layer swelling and its removal were followed by measuring the pressure drop (ΔP).

Three channels (channels 4, 5, 6) were modified (each with both sides of modified stainless steel surfaces) in the PHE2 heating zone. For the SiF_4^+ and DLC, only one channel could be investigated.

2- New surface modification techniques

The goal of efforts was to permanently modify the surface characteristics of stainless steel, in order to reduce the adhesion and the deposition of fouling components in the heat processing of dairy fluid. Height different surface treatments were tested: ion implantations (SiF^+ , MoS_2) and coatings (Diamond Like Carbon [DLC], SiO_x , Ni-P-PTFE, Silica, Xylan[®] and Excalibur[®]). The cost of the implantation surface (320 € m⁻²) is less expensive than the coating cost (360 to 4000 € m⁻²).

There are two kinds of ion implantation techniques: direct ion implantation and turbulent ion implantation.

The ions implanted in the first case were SiF^+ , which were accelerated to an energy of 200 keV before bombarding the substrate. The dose was equal to 5×10^{16} ions per cm². The ions settled in a depth of approximately 0.2 μm, where the location of the ions perpendicular to the surface corresponds to a statistical distribution.

In the case of turbulent ion implantation, MoS_2 was chosen. MoS_2 is well known as an anti-adhesion promoter in the mechanical engineering field. Ions penetrate the matter up to the depth of about 100 μm, where the highest concentration is at the outer surface and drops further into the substrate material.

The DLC (Diamond Like Carbon) coating investigated is a multi layer coating system. The different inter layers are deposited by plasma enhanced chemical vapour deposition (PECVD). The composition varied gradually, so that there are no abrupt changes between the layers. The overall thickness of the DLC coating is around 2 μm.

The applied SiO_x coating was also produced by PECVD. SiO_x is similar to quartz or glass. Therefore its advantage is a very inert surface. This kind of coating, as well as the DLC coating, belongs to the category of thin film coatings. The SiO_x layer used is nearly 1 μm thick.

Ni-P-PTFE (Nickel-Phosphorous-Poly-Tetra Fluoro Ethylene) is a plating produced by an autocatalytic coating process, whereby PTFE particles are incorporated in a Ni-P matrix. The adhesion of the Ni-P-PTFE coating is significantly improved by gradually increasing the PTFE content from the substrate to the top surface. The thickness of the Ni-P-PTFE plating can vary between 3 and 25 μm.

Silicat coating is a gel composed of Ti_2O_3 and silica. The thickness of the silicat can vary between 0.1 and 0.2 μm.

Xylan[®] (Whitford) is a composite of PTFE particles embedded in an external polymer matrix. This coating is applied with conventional spray and dip-spin techniques. The main characteristics of this surface are the excellent corrosion resistance, the wide temperature operating range, low friction and non-stick properties. The minimum thickness of Xylan[®] that can be used is about 17 μm.

Excalibur[®], another kind of PTFE coating, is built up by three different inter layers. First a thermal sprayed stainless steel sub layer to enhance the adhesion to the substrate is used, followed by two kinds of PTFE varnish. The overall thickness of Excalibur[®] is around 70 μm.

The roughness of the surface modified plates represented by the average roughness R_a and the root mean square roughness R_q can be seen in Fig. 3. Excalibur[®] and Xylan[®] coatings are rougher than the all surfaces.

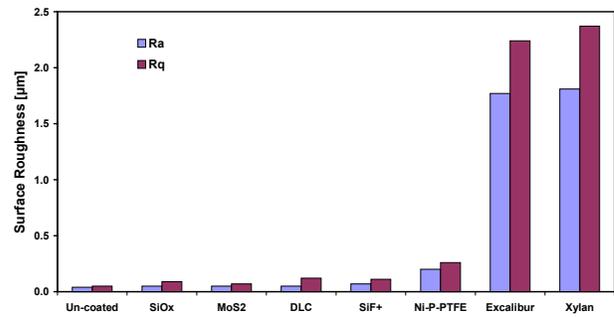


Fig. 3 Roughness values of the various surface modifications

The solid surface free energy was estimated from contact angle measurements (diiodomethane, formamide and water) by least-squares fitting of the data to Owens & Wendt (1969). In Fig. 4 the dispersive and polar components of the differing surfaces can be seen. Ni-P-PTFE, Excalibur[®] and Xylan[®], which contained Teflon particles, are hydrophobic.

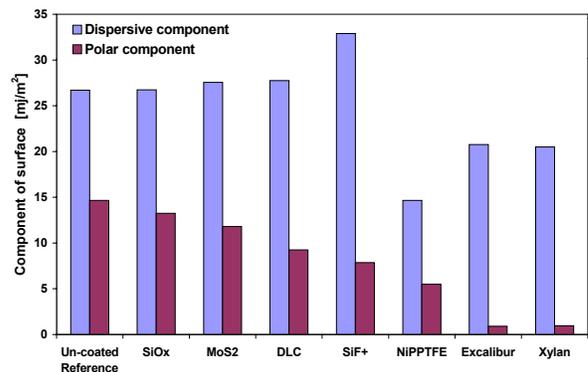


Fig. 4 Dispersive and polar components

METHODS

1- Preliminary pilot experiments: Fouling and cleaning behavior of un-coated steel

To study fouling on steel surfaces to a significant extent, in a reasonable processing time, the use of a dairy mix was found to be important. This Fouling Model Fluid [FMF] mimics a dairy dessert cream composed of whey proteins, whole milk, sugar and xanthan gum. The fouling layer is

only due to the milk and therefore whey proteins. Mineral fouling appeared to be negligible in comparison to protein fouling in our experimental conditions (data not shown). The xanthan gum, a dairy product thickener, was chosen to modify and to adjust the viscosity of the fluid. The apparent viscosity was measured on samples taken before and after the heater, using a viscosimeter (Rheomat 30, Contraves). The shear thinning behavior of the fluid can be described using the power law model ($n=0.39$; $k=0.45 \text{ Pa}\cdot\text{s}^n$).

Because of the similar composition to milk, milk thermophysical properties (thermal conductivity, heat capacity) were used for calculations.

This FMF product (pH=7) was stored in a stirred tank at 15 °C in order to prevent bacteria proliferation prior any experiments. Subsequently the fluid was heated from 15 to 60 °C with circulating hot water (63 °C) [PHE1]. Then the fouling fluid was heated to 102 °C in the PHE2 heater. After cooling and chilling [PHE3] the milk product was diverted to waste. The milk product mean velocity was fixed at a constant value of $1.42 \cdot 10^{-1} \text{ m/s}$ ($\text{Re}=120$ transitional flow regime) and the hot water mean velocity at $7.12 \cdot 10^{-1} \text{ m/s}$ ($\text{Re}=7470$ turbulent flow regime). The mean residence times (\bar{t}_i) (see Eq. (4)), which were calculated using the classical relationship, amounted in the PHE2 to 12.5 s.

$$\bar{t}_i = \frac{V_{7c}}{Q_f} \quad (4)$$

After the fouling procedure, the PHE2 was rinsing and dismantled. Then the PHE2 was cleaned with NaOH 0.5 % at 60 °C for 15 min. The soda mean velocity was $4.27 \cdot 10^{-1} \text{ ms}^{-1}$ ($\text{Re}=3460$) and the hot water (60 °C) mean velocity $7.12 \cdot 10^{-1} \text{ ms}^{-1}$ ($\text{Re}=4580$). The mean residence time was around 4 s. Residual soiling could be observed on the plates after cleaning due to the mild conditions chosen. Any variations could therefore be of help in discriminating between the various steel alterations tested. As the fouling layer was mainly composed of proteins, only soda cleaning was used. After the cleaning procedure, the PHE2 was rinsing and dismantled.

2- In situ methods

Fouling and cleaning references on un-coated steel were established to allow a comparison with the fouling and cleaning behavior of the steel before modification.

The fouling kinetics of the PHE2 were followed by the decrease in the overall heat transfer coefficient (α_g) and the increase in pressure drop (ΔP) with time. The efficiency of cleaning could be defined as the difference between the pressure drop before and after cleaning. Both the fouling and cleaning references obtained on standard steel plates were found to be reproducible.

All of the trials were performed in duplicate. The thermal and hydraulic performances (α_g , ΔP) between modified steel surfaces and reference steel were compared.

To allow comparison, normalized overall heat transfer coefficient $\alpha_g(t)^*$ Eq. (5, 7) and normalized pressure drop $\Delta P(t)^*$ Eq. (6, 8) and $\Delta P(t)^{**}$ Eq. (9, 10) values were calculated as follows:

For the fouling process :

$$\alpha_g(t)^* = \frac{\alpha_g(t)}{\alpha_{g0C}} \quad (5) \quad \text{and} \quad \Delta P(t)^* = \frac{\Delta P(t)}{\Delta P_{0C}} \quad (6)$$

$$\text{with } \alpha_{g0C} = \alpha_g(t)_{PHEclean} \quad (7) \quad \Delta P_{0C} = \Delta P(t)_{PHEclean} \quad (8)$$

$$\text{For the cleaning process: } \Delta P(t)^{**} = \frac{\Delta P(t)}{\Delta P_{0F}} \quad (9)$$

$$\text{with } \Delta P_{0F} = \Delta P(t)_{PHEfouled} \quad (10)$$

In equations 5 and 6, when the fouling phenomenon begins, $\alpha_g(t)^* = 1$ and $\Delta P(t)^* = 1$. When the cleaning phenomenon begins, $\Delta P(t)^{**} = 1$ for the each alteration of the steel.

3- Fouling ability of the FMF

The concentration of native β -lactoglobulin was assessed for the test fluid before and after the heating process. The denaturation of this protein when heated is known to govern the fouling phenomenon in dairy product pasteurization and sterilization (Delplace and *al.*, 1997). Any change in the mix before heating deeply influences the fouling of the PHE plates. Therefore the radial immunodiffusion method previously describes by Lyster (1970) was then used, with a standard error of 3 %, to measure the concentration of our initial and final fouling solution. At the inlet, the FMF solution contained 26.5 g l^{-1} of native β -lactoglobulin and at the outlet of PHE2, the concentration varied between 3.7 and 9 g l^{-1} .

4- Evaluation of the deposit mass

After each fouling and cleaning steps the PHE2 was dismantled. All the plates were weighed and photographed.

To be able to neglect the aging effect of the FMF, a weight ratio (R) was calculated with the total amount of deposit mass in the channels 4, 5 and 6 (modified surfaces) divided by the deposit mass in the un-coated channels 1, 2, 3 after fouling and after cleaning Eq. (11).

$$R = \frac{\sum m_{mc}}{\sum m_{nmc}} \quad (10)$$

with $(\sum m_{mc})$: Total mass deposit on modified channels (channels 4, 5 & 6)
 $(\sum m_{nmc})$: Total mass deposit on non-modified channels (channels 1, 2 & 3)

In the case of SiF⁺ and DLC only one channel was modified due to the expensive plate cost (4000 € m⁻²). Hence it was necessary to define the weight ratio (*R*) as follows:

$$R = \frac{\sum m_{mc}}{\left(\frac{1}{3}\right) \cdot \sum m_{nmc}} \quad (11)$$

RESULTS

1- Fouling

Linear evolution for both the pressure drop and the overall heat transfer coefficient could be observed during 90 min heating. The milk fouling layer repartition inside the PHE2 appeared to be homogeneous. On the reference steel, the overall heat transfer coefficient decreased to 17 % and the pressure drop increased to 32 % (Fig. 5).

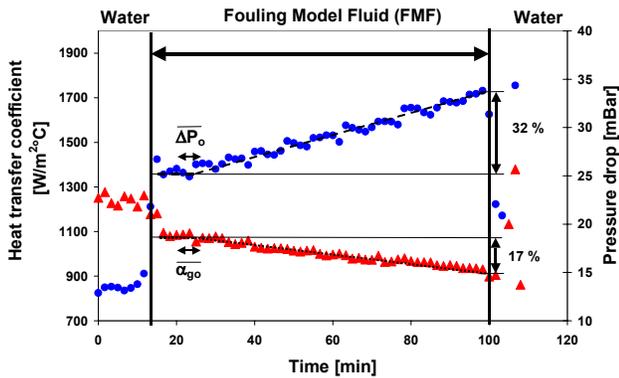


Fig. 5 Overall heat transfer coefficient (▲) and pressure drop (●) evolution with time for the PHE2 (un-coated plates) during heating

The evolution of the pressure drop and the overall heat transfer coefficient were linear for all steel modifications investigated, because the fouling period under our experimental conditions was only 90 min. A direct comparison of the slopes for both parameters could be used to compare standard steel and modified steels.

To compare the data measured, they were normalized them just to compare the evolution starting with the same value. The results for all surface modifications are summarised in Table 1. Two repetitions per steels were realized. At first, a short period (22-58 min) could be

observed where α_g remained constant. However, this induction period could not be observed on pressure drop curves.

The slope of $\alpha_g^*(t)$ appeared to be quite similar, between -0.001 and -0.002, within each trial. Little variability in ΔP^* slopes was observed regarding values covering a short range of variations (0.0033 and 0.0058).

Table. 1 Induction period and slopes of the overall heat transfer coefficient and the pressure drop during fouling

| Coating | Induction period (α_g^*) [min] | | Slope (α_g^*) | | Slope (ΔP^*) | |
|------------------------|---|-----------------------|------------------------|-----------------------|------------------------|-----------------------|
| | 1 st repet | 2 nd repet | 1 st repet | 2 nd repet | 1 st repet | 2 nd repet |
| Un-coated Reference | 22 | 32 | -0.0019 | -0.0012 | 0.0042 | 0.0037 |
| Ni-P-PTFE | 25 | 36 | -0.0020 | -0.0018 | 0.0044 | 0.0042 |
| SiOx | 23 | 33 | -0.0015 | -0.0013 | 0.0041 | 0.0058 |
| Silica | 58 | / | -0.0014 | / | 0.0045 | / |
| MoS ₂ | 28 | 33 | -0.0015 | -0.0016 | 0.0040 | 0.0036 |
| DLC | 42 | 26 | -0.0018 | -0.001 | 0.0033 | 0.0046 |
| SiF ⁺ | 42 | 26 | -0.0018 | -0.001 | 0.0033 | 0.0046 |
| Xylan [®] | 30 | 40 | -0.0014 | -0.0020 | 0.0038 | 0.0042 |
| Excalibur [®] | 34 | 26 | -0.0013 | -0.0021 | 0.0058 | 0.0041 |

Finally, no significant differences in α_g^* and ΔP^* slopes with time can be observed with the statistical variance analysis. The $\alpha_g(t)$ and $\Delta P(t)$ parameters could not accurately differentiate the coatings has investigated either regarding fouling minimization or cleaning efficiency.

But some discrepancies between the first and the second trials with the same plates could be noticed. Evolutions in the slopes of α_g^* and ΔP^* were found to be quite similar to the Ni-P-PTFE and MoS₂ whereas slight variations were observed on the other altered steel surfaces (SiO_x, Silicat, DLC, SiF⁺, Xylan[®], Excalibur[®]). As an example the normalized pressure drop and overall heat transfer drop with time on the Excalibur[®] coating (first and second trials) is shown in Fig.6.

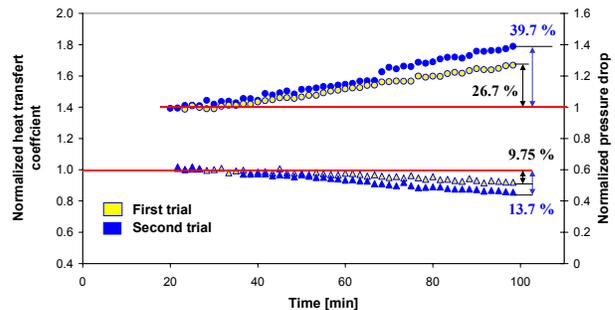


Fig. 6 α_g^* (▲) and ΔP^* (●) evolution with time on Excalibur[®] coatings during heating

The increase in pressure drop is as high as 26.7 % on the first run and as high as 39.7 % on the second. The decrease in the overall heat transfer coefficient is 9.75 % on the first and 13.7 % on the second run.

2- Cleaning

During cleaning (soda 0.5 %, 60 °C, 15 min) the pressure drop first increased due to the swelling of the soil. After peaking, the pressure drop decreases again, as observed in Fig. 7. The reason for this is on-going detachment. The difference between the end and the start reference pressure is around -0.56 Bar and represents the efficiency of cleaning.

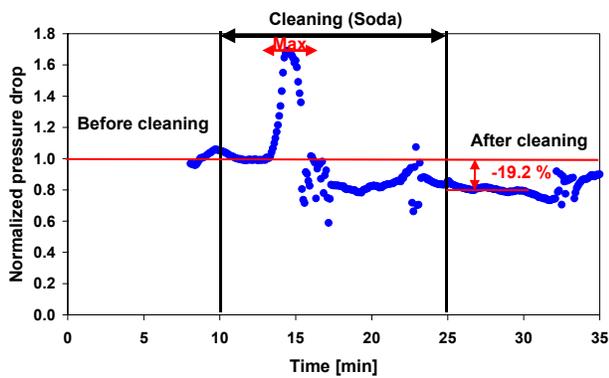


Fig. 7 Reference pressure drop evolution with time for PHE2 (un-coated plates) during cleaning

To compare the various modified steels, pressure drop data were normalized. Three parameters were chosen to compare the performances: (i) the time necessary to obtain 10% decrease in the pressure drop ; (ii) the swelling time and (iii) the maximum pressure drop corresponded to the maximum swelling of the soil. (Table 2).

Table. 2 The ΔP^{**} variations during cleaning

| Coating | ΔP^{**}_{max} | | Swelling time [min] | | Time [min] ΔP^{**} decrease 10 % | |
|------------------------|-----------------------|-----------------------|-----------------------|-----------------------|--|-----------------------|
| | 1 st repet | 2 nd repet | 1 st repet | 2 nd repet | 1 st repet | 2 nd repet |
| Un-coated Reference | 1.7 | 1.4 | 2.7 | 2 | 16.9 | 15.7 |
| Ni-P-PTFE | 1.7 | 1.5 | 4.8 | 4.2 | 19.8 | 20.2 |
| SiO _x | 2.3 | 1.6 | 2.6 | 2.7 | 16.8 | 14.9 |
| Silica | 1.8 | / | 2.8 | / | 16.5 | / |
| MoS ₂ | 1.7 | 1.8 | 2.9 | 3.1 | 16.3 | 18.7 |
| DLC | 1.8 | 1.8 | 3.1 | 3 | 16.8 | 17.6 |
| SiF ⁺ | 1.8 | 1.8 | 3.1 | 3 | 16.8 | 17.6 |
| Xylan [®] | 1.5 | 1.5 | 2.7 | 3.2 | 16.8 | / |
| Excalibur [®] | 1.9 | 2.2 | 3 | 1.8 | 16.3 | 14.8 |

At the end of cleaning, the decrease in the normalized pressure drop is almost similar for all the test plates. The

time necessary to obtain a decrease in normalized pressure drop of 10 %, varied between 15 and 20 min (Table 2). However, several differences were observed at the start of cleaning. The height and the width of the soil swelling varied according to the trial and the steel modification. For example the swelling time for the Ni-P-PTFE plating is important than for the other coatings (Fig. 8). The maximum pressure drop of SiO_x and Excalibur[®] is higher.

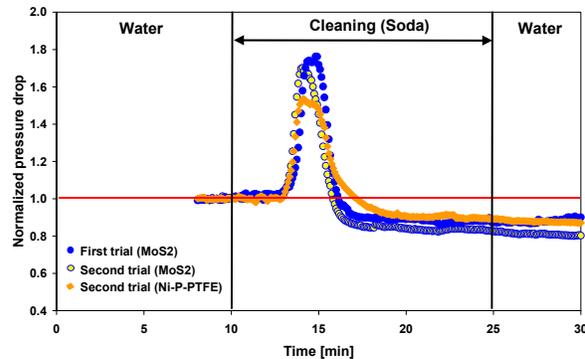


Fig. 8 Normalized pressure drop evolution with time for MoS₂ (●) and Ni-P-PTFE (◆) coatings during cleaning.

Thus, exactly as for fouling purpose, no clear difference between steel modifications could be seen for the fouling reduction or cleaning efficiency with α_g and ΔP parameters.

3- Mass deposition

No fouling phenomenon was observed on the pre-heater plates (PHE1), because not any protein denaturation actually begins at 60 °C. In the heater (PHE2), where the temperature is higher than 60 °C, the absolute mass deposit increased from channel 1 to channel 6. See Fig. 9 for the case of un-coated plates for all modified surfaces tested. After the cleaning, the mass deposit per channel was found to be independent on the channel and at about 2g.

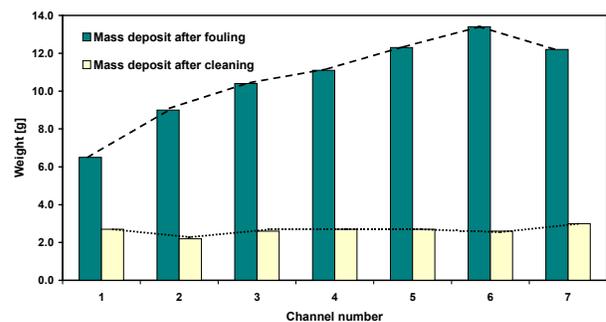


Fig. 9 Weight of deposit in V2 PHE2 (un-coated plates) channels after fouling and both fouling and cleaning

Results of deposit weight ratio after fouling and cleaning processes for steel alterations are presented in Fig. 10.

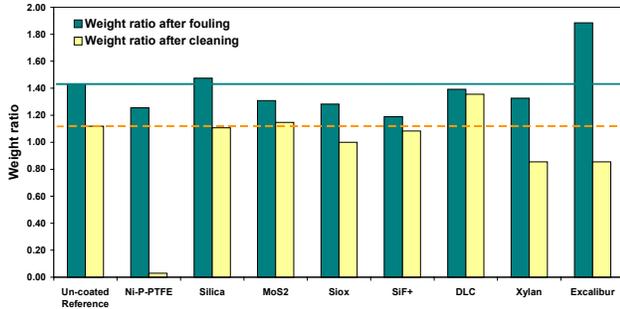


Fig. 10 Weight ratio of the different modified steel tested after fouling and cleaning.

After fouling the weight ratio for the reference steel is greater than 1 ($R=1.43$). The fouling deposits in channels 4, 5, and 6 are larger than in channels 1, 2 and 3. Indeed, denaturation of β -lactoglobulin starts when temperature is higher than 90 °C (Danneberg, 1986), a temperature which was reached in channel 4.

All of the alterations tested appeared to be better than the reference stainless steel, except for the Excalibur[®] coating. The SiF⁺ implantation seemed to be the most promising surface to minimize the fouling as was as Ni-P-PTFE, MoS₂ and Xylan[®] coatings. However, this improvement did not exceed 18 % in our experimental conditions. However compared with the reference steel, no clear difference could be seen regarding improvement in the fouling behavior confirmed by variance statistic analysis (Fig. 10).

The fouling deposit is white, sponge like and quite homogeneously spread. The fouling layer appeared to be quite, except for the SiO_x coating where some pack of remaining soils were visible.

After cleaning (Fig. 10) the weight ratio of reference steel is superior to 1 ($R=1.10$), whereas the other ratios were inferior except for the DLC coating ($R=1.22$).

A mean comparison test was realised. The Ni-P-PTFE coating was significantly the best whereas the DLC coating was significantly the worst.

No visible soil were observed on Ni-P-PTFE coating, a very few soil on silica and Excalibur[®] coatings. A skin like deposit could be seen on Xylan[®] and SiO_x coatings due to a higher consistency of the remaining soil.

Excalibur[®] coating seemed to exhibit quite a good level of ease of cleaning (55 %) and a clear increase in the fouling ability (-32 %). However Ni-P-PTFE appeared to be

really promising for both aspects regarding fouling and cleaning (Table 2).

A high level of fouling was observed on Excalibur[®] coating which exhibited a high average roughness value ($R_a=1.77$; $R_q=2.24$) (Fig. 3). However for same average roughness value ($R_a=1.81$; $R_q=2.37$) the Xylan[®] appeared to be better than the stainless steel reference ($R_a=0.04$; $R_q=0.05$) for fouling ability. A causal relationship between the roughness and the fouling level or the cleanability could not be clearly demonstrated here.

In addition both materials Xylan[®] ($\gamma_L^p=0.95$; $\gamma_L^d=20.5$) and Excalibur[®] ($\gamma_L^p=0.9$; $\gamma_L^d=20.75$) exhibited about the equivalent Owens & Wendt polar component values (Fig. 4) and equivalent cleaning weight ratio (Fig.10). However the Ni-P-PTFE coating with quite a low polar component ($\gamma_L^p=5.5$; $\gamma_L^d=14.65$) appeared to be significantly better for ease of cleaning (Fig. 10).

The variance analysis (SAS version 8, USA) confirmed the preponderant influence of Owens & Wendt components (γ_L^p , γ_L^d) and the minor influence of surface roughness (R_a , R_q) on the cleanability.

According to Boulangé-Peterman (1996) and Jullien *et al.* (2003), any contact between stainless steel and food or detergents will alter the surface energy. At the outset, the coated surfaces were new. For the second run, these steel surfaces had been already in contact with milk protein and particularly with the detergent. This may cause the significant difference in fouling and cleaning between the first and the second trial. Furthermore, we observed that the SiO_x and the silica coatings were removed after two trials. Also, the DLC coating seemed to be damaged.

CONCLUSIONS

Various surface treatments such as coatings (silica, SiO_x, Ni-P-PTFE, Excalibur[®], Xylan[®]) and as ion implantations (MoS₂, SiF⁺) were tested. Standard milk fouling and cleaning references in PHEs were established with fouling model fluid (FMF). During the fouling process, the decrease in the overall heat transfer coefficient and the increase in the pressure drop were used to appreciate the fouling formation on heated surfaces, which were weighed after both fouling and cleaning runs.

After fouling, no significant difference could be seen between all the modified steels and the reference by statistical variance analysis. Cleaning efficiency of Ni-P-PTFE appeared significantly the best. Finally, the fouling and soiling were shown to depend on the alteration of the steels. It could be suggested that the free surface energy plays a predominant role and the roughness a minor role in the level of fouling and cleaning efficiency. Yet damaged surfaces and the conditioning film also influenced the surface properties.

NOMENCLATURE

| | |
|--|--|
| C_p Heat capacity, j/kg K | R_q Mean square roughness, μm |
| F_t Corrective factor of $\Delta\theta_{\text{in}}$ | R Weight ratio |
| k Flow consistency index, $\text{Pa}\cdot\text{s}^n$ | R_a Average roughness, μm |
| m Deposit mass, kg | Re Reynolds number (-) |
| \dot{m} Mass flow rate, kg/s | R_q Mean square roughness, μm |
| n Flow behavior index (-) | S Heat exchanger area, m^2 |
| Q Flow rate, m^3/s | V Volume, m^3 |
| q Heat flux, W | |

Symbol

| | |
|---|---|
| θ Temperature, $^{\circ}\text{C}$ | ΔP Pressure, Bar |
| $\Delta\theta_{\text{in}}$ Logarithmic mean temperature difference between two fluids, $^{\circ}\text{C}$ | α_g Overall heat transfer coefficient, $\text{W}/\text{m}^2\text{K}$ |

Subscript

| | |
|----------------------------|---------------------|
| c Channels | o Outlet |
| C Clean PHE | p Milk product |
| CIP Cleaning In Place | t Time |
| f test fluid | ti Residence time |
| F Fouled PHE | tot Total |
| i Inlet | w Water |
| max Maximum | |
| mc Modified channel | $*$ Normalized |
| nmc Non-modified channel | 0 Initial |

REFERENCES

- Bird, M.R., and Fryer, P.J., 1991, An experimental study of the cleaning of surfaces fouled by whey proteins, *Trans IchemE*, Vol. 69, C, pp. 13-21.
- Boulangé-Petermann, L., 1996, Processes of bioadhesion on stainless steel surfaces and cleanability: a review with special reference to the food industry, *Biofouling*, Vol. 10, pp. 275-300.
- Dannenber, F., 1986, Zur reaktionskinetik der molkenproteindenaturierung und deren technologischer bedeutung, *PhD Thesis*, TU Munich.
- De Jong, P., 1997, Impact and control of fouling in milk processing, *Trends in Food Science and Technology*, Vol. 8, pp. 401-405.
- Delplace, F., Leuliet, J.C., and Levieux, D., 1997, A reaction engineering approach to the analysis of fouling by whey proteins of six channels-per-pass plate heat exchanger, *J. Food Engineering*, Vol. 34, pp. 91-108.
- Delplace, F. and Leuliet, J.C., 1995, Modeling fouling of a plate heat exchanger with different flow arrangement by whey protein solutions, *Trans. IchemE, C*, Vol. 73, pp. 112-119.
- Delplace, F., Leuliet, J.C. and Tissier, J.P., 1994, Fouling experiments of a plate heat exchangers by whey proteins solutions, *Trans. IchemE, C*, Vol. 72, pp. 163-169

Donaldson, J., and Grimes, S., 1988, Lifting the scale from our pipes, *New Scientist*, Vol. 18, pp. 43-46.

Fryer, P.J., Robins, P.T., Green, C., Schreier, P.J.R., Pritchard, A.M., Hasting, A.P.M., Royston, D.G. and Ritchardson, J.F., 1996, A statistical model for fouling of a plate heat exchanger by whey protein solution at UHT conditions, *Trans. IchemE, C*, Vol. 74, pp. 189-199.

Jeurnink, T.J.M., 1996, Milk fouling in heat exchangers, *PhD Thesis*, Wageningen Agricultural University, Wageningen, The Netherlands.

Jullien, C., Benezech, T., Carpentier, B., Lebret, V., and Faille, C., 2003, Identification of surface characteristics relevant to the hygienic status of stainless steel for the food industry, *J. Food Engineering*, Vol. 56, 1, pp. 77-87.

Lalande, M., Tissier, J.P., and Corrieu, G., 1985, Fouling of heat transfer surfaces related to β -lactoglobulin denaturation during heat processing of milk, *Biotechnology Progress*, Vol. 1, pp. 131-139.

Lyster, R.L.J., 1970, The denaturation of α -lactalbumin and β -lactoglobulin in heated milk, *J. Dairy Res.*, Vol. 37, pp. 233-243.

Müller-Steinhagen, H., and Zhao, Q., 1997, Investigation of low fouling surface alloys made by ion implantation technology, *Chemical Engineering Science*, Vol. 52, 19, pp. 3321-3332.

Owens, D.K., Wendt, R.C., 1969, Estimation of the surface free energy of polymers, *J. Appl. Polymer Sci.*, Vol. 13, pp. 1741-1747.

Romney, A.J.D., 1990, CIP: Cleaning in Place, *Society of Dairy Technology*, London.

Sandu, C., and Lund, D., 1983, Fouling of heat exchangers: optimum design and operation. Book: *Fouling of heat exchanger surfaces*, Engineering Foundation, New York, pp. 681-716.

Shah, R.K., Subbarao, E.C., and Mashelkar, R.A., 1988, *Heat transfer equipment design*, Washington DC: Hemisphere Publishing Corporation.

Timperley, D.A., and Smeulders, C.N.M., 1987, Cleaning of dairy high-temperature-short-time plate heat exchangers, *J. Soc. Dairy Technol.*, Vol. 40, pp. 4-7.

Visser, H., Jeurnink, T.J.M., Shraml, J.E., Fryer, P., and Delplace, F., 1997, Fouling of heat treatment equipment. *Bulletin of the IDF*, Vol. 328, pp. 7-31.

Visser, H., and Jeurnink, T.J.M., 1997, General aspect of fouling and cleaning, *Bulletin of the IDF*, Vol. 328, pp. 5-6.

Zhao Q., Liu, Y., and Müller-Steinhagen, H., 2002, Graded Ni-P-PFE coatings and their potential applications, *Surface and Coatings Technology*, Vol. 155, pp. 279-284.

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