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DESCRIPTION OF THE CLEANING MECHANISM OF A MODEL FOOD SOIL USING AN OPTICAL DETECTION METHOD AND THE FDG TECHNIQUE

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ABSTRACT

The effects of the most relevant industrial operating parameters to remove a baked egg yolk layer were investigated using an optical detection method and a flow channel with a sudden expansion.

In this work the local cleaning times in several channel regions with different flow regimes were measured to determine the factors controlling removal of egg yolk. An empirical model has been developed and used to analyse the results in terms of the work required. Depending on the flow behavior, the influence of the investigated process parameters (NaOH concentration, temperature, flow rate) changed significantly. Investigations have shown that temperature is the most relevant parameter but in particular sodium hydroxide in typical concentration of 1.5 wt% shows an adverse effect. This fact is also confirmed by Fluid Dynamic Gauging measurements. In addition, the combination of process parameters affected the type of removal (adhesive or cohesive failure).

INTRODUCTION

Currently in many branches of the manufacturing industry cleaning processes are highly automated, with cleaning-in-place (CIP) technologies. These technologies should help to increase productivity and shall in particular ensure product safety. However the process of designing an appropriate cleaning protocol for a given situation is still semi-empirical or based on personal knowledge (Fryer 2009). Moreover the effort to secure compliance with legal requirements and to minimize the risks of insufficient cleaning and their consequences (e.g. recall action, lack of consumer confidence) leads to a tendency, systematic „over cleaning“ (too long, too hot, too intensively). At present, a large number of cleaning tests at industrial scale are needed to identify an acceptable combination of the process parameters. This is more or less a trial and error approach and time and cost intensive. Optimization of cleaning processes can only be attained by understanding the interaction, how deposits' behavior depends on their material properties, their interactions with the surface and specific cleaning solutions, respectively temperature, chemistry and mechanics. To obtain further knowledge Föste et al. (2014) presented “An unified approach to classify removal processes of different soils using dimensionless numbers“. The aim is the prediction of the cleaning success through the characterization of industrial deposits by laboratory analysis. To achieve this, experimental methods and dimensionless numbers need to be developed. As an integral part of that, the relevant effects of process parameters on the removal of soils must be represented in an experimental set-up and expressed quantitatively. This intermediate part will be described in this work based on a baked egg yolk layer. For this work yolk was chosen due to the importance for the food industry. In pasteurization processes persistent deposits in the heat exchanger are built up, which reduce the efficiency and endanger the product quality. The complex combination of fat, protein and other ingredients is a challenging task for cleaning.

For a better understanding of cleaning processes the optical detection method allows the investigation of the type of removal. Studies by Liu et al. (2006a, 2006b) and Christian & Fryer (2004) discussed the influence of the type of removal and classified two distinct groups of removal mechanisms:

- removal of soil in large chunks with adhesive failure occurring between soil and the surface, and
- removal of soil in small chunks from the surface.

The type of failure is decisively controlled by the dominant force interaction. Liu et al. (2002) and Bobe et al. (2007) assign van-der-Waals forces, electrostatic forces and contact area effects as principal factors for the adhesive force. Furthermore cohesive forces between elements of the soil depend on the nature of the material. In the following the investigation shows that the process parameters affect the primary cleaning.

MATERIALS AND METHODS

Soiling procedure

Egg yolk samples were used as the test soil for experimentation. The model food soil samples were prepared by stirring 40 g egg yolk powder into 50 ml of reverse osmosis (RO) water at 1600 rpm for 45 min. The pasteurized and spray dried powder (Emultherm, OVOBEST Eiprodukte GmbH & Co, Germany) contains around 31% of proteins and 57% of fats. The pastes were applied to stainless steel coupons (AISI 304 2B (IIIc), cold rolled) using a temporary polyvinylchloride mask (PVC, 175 µm thick). The surplus egg yolk pate is removed with a scraper, described by Schöler et al. (2009). After the PVC mask is removed, a defined egg yolk layer with a thickness of approximately 175 µm in the wet state is remaining as a defined soiling layer. Following, the samples were placed in a preheated oven at 80°C for 9 min. Thereby proteins denature and other structural changes result. Afterwards the baked egg yolk samples stored at 23°C, 50% relative humidity for 24 h resulting in an initial mean surface mass of $m_s = 96.5 \pm 8\%$ g/m² (Fig. 1).

Fig. 1 Photograph of an egg yolk layer used in this work.

Cleaning test rig

A detailed description of the cleaning test rig is given in Schöler et al. (2009, 2012). The system closely replicates the geometry and behavior of an industrial CIP system, allowing experimental results to be extrapolated to the industrial scale with confidence. The cleaning solution in varying temperature and sodium hydroxide concentration is circulated from holding tanks through the test section. The test region consists of a rectangular channel with a sudden expansion, as depicted in Fig. 2. The stainless steel coupons (inlet coupon with 115 x 28 x 1 mm³, outlet coupon with 145 x 43 x 1 mm³) were inserted into the channel and fixed by guide slots. The fluid flow in the channel is characterized by several regions (inlet, dead area and outlet). From the channel dimensions and the volumetric flow rate Q, the Reynolds number in the three regions of the flow channel can be calculated by

$$\mathrm{Re} = \frac{\rho \cdot Q}{\eta \cdot d_h} \tag{1}$$

where ρ is the density of the fluid , η the dynamic viscosity and d_h is the hydraulic diameter of the square cross sectional area. In order to conduct the measurement of the cleaning process in situ, one side of the flow channel includes a window made of borosilicate glass (Schott TGS GmbH).

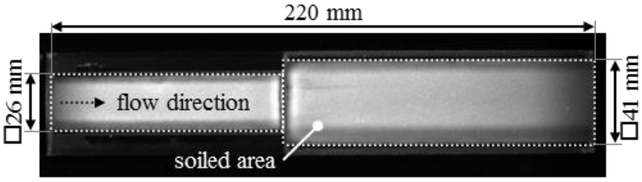

Fig. 2 Rectangular flow channel with sudden expansion, within the regions of interest (dotted white lines). Flow direction from left to right.

The cleaning process was optically monitored with the local phosphorescence detection (LPD) method, which was first introduced by Schöler et al. (2009). To measure the fluorescence properties of the egg yolk itself, the LPD-method was modified. Two UVA lamps were used to illuminate the egg yolk and the cleaning process was photographed every 0.3 s with a CCD camera (Matrix Vision, mvBlueCougar-X124G), whereby the cleaning progress (as the decrease of the fluorescence intensity) can be temporally and spatially resolved. An opaque housing around the test region ensures that only the egg yolk fluorescence is detected.

The operating parameters were varied by using the design of experiment principles within the combined influence of the following conditions:

- concentration C = 0 wt% … 1.82 wt% NaOH
- solution temperature T = 24ºC … 60°C
- volumetric flow rate Q = 9.6 L/min … 86 L/min

- $1.2 \times 10^4 \leq \mathrm{Re}_{\mathrm{inlet}} \leq 1.2 \times 10^5$
- $7.4 \times 10^3 \leq \mathrm{Re}_{\mathrm{outlet}} \leq 7.4 \times 10^4$
- 17 different experiments with 47 valid runs

Data analysis

The determination of the residual soil during cleaning is based on an optical method which determines the fluorescence intensity of the egg yolk. The grey scale images of all experiments are automatically analysed by a MATLAB® script. An image is cropped to a region of interest to avoid side effects. To reduce image noise the grey value over a range of 0.7 x 0.8 mm² is averaged. To obtain the quantitative removed mass of soil, the correlation between fluorescence intensity and soil mass is necessary. Therefore, different thicknesses of the egg yolk layers were prepared with the scraping module, the initial soil mass of every sample is determined and the fluorescence intensity is measured with the camera system. Fig. 3 shows a linear dependence between the fluorescence intensity and the soil mass up to a soil mass of 100 g/m². For analysing the effects of different cleaning parameters the cleaning time is set to t_{95}. The cleaning time t_{95} is the time which is necessary to remove 95% of the soil mass. Based on Mauermann et al. (2010) and Köhler et al. (2011), a normalized cleaning curve can be calculated with the normalized soil mass m_s/m_{s0} as a function of the cleaning time. Fig. 4 shows the local cleaning time for the flow channel with a sudden expansion.

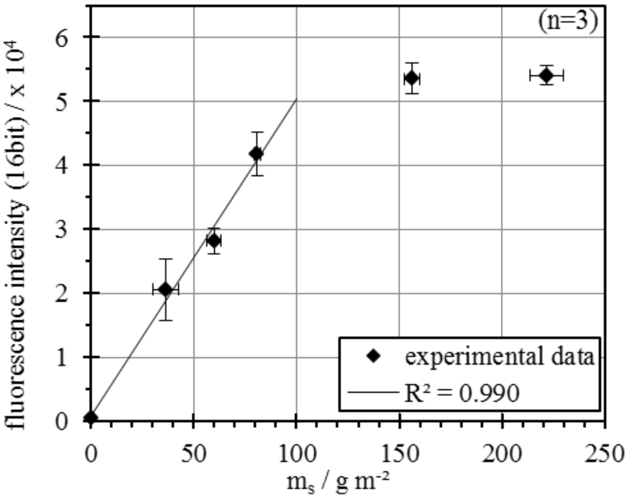

Fig. 3 Correlation between fluorescence intensity and soil mass of egg yolk, for constant exposure time.

Depending on the region in the flow channel different cleaning times can be seen, which suggests different influences of cleaning parameters. In order to derive the effects of process parameters on the removal of egg yolk, the analysis focuses on the three main regions of the fluid flow in the channel. For this purpose, the cleaning times are averaged in the inlet, the dead area behind the sudden expansion and the channel outlet, as shown in Fig. 4.

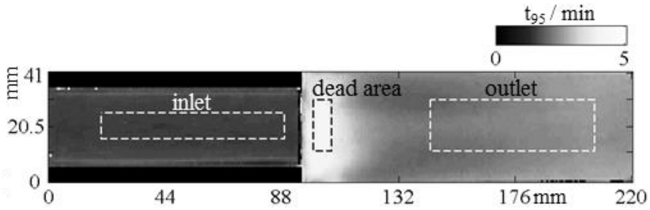

Fig. 4 Local cleaning time t_{95} over the flow channel and the main regions marked as dashed lines.

Fluid dynamic gauging

The Fluid Dynamic Gauging (FDG), developed and initially described by Tuladhar et al. (2000), enables the in-situ and contactless determination of the soil layer thickness and their changes over time.. The experimental setup used in this work was described by Föste et al. (2014). A nozzle represents the central element and is placed in a box-shaped reservoir close to the ground where the examined soil layer can be located. To ensure a constant fluid level and therefore a constant hydrostatic pressure a weir is integrated in the reservoir. The outlet of the weir is connected to a tempered (by a cryostat) double jacket vessel which ensures constant process temperatures. From the double jacket vessel, the fluid gets pumped back into the reservoir. The pressure gradient between nozzle (inlet) and outlet causes the driving force for the outflow through the nozzle. If the nozzle is placed in a small distance to the soil layer, the mass flow rate is sensitive to the distance and leads therefore to the determination of the soil layer thickness without any contact. A detailed description about the measuring principle is given in Tuladhar et al. (2000).

RESULTS & DISCUSSION

Based on the experimental data the statistical analysis was used to develop an empiric model, to analyse the influence of the temperature, the NaOH concentration and the flow rate on the removal of baked egg yolk layers. Analyses of variance (ANOVA) were used for each channel region separately to verify the suitability of the models and the significance of the different factors (coefficients) and possible interactions of several factors. The factors were normalized and influences with no statistical significance (p value > 0.05) were excluded. The final models include linear and quadratic effects of the three process parameters (Q, T, C) as well as the interaction between the temperature and the concentration of sodium hydroxide. Table 1 summarises the coefficients to complete the mathematical models, represented by Eq. (2):

$$t_{95}\big/\text{min} = \left(c_0 + c_Q\, Q\Big/\tfrac{L}{\text{min}} + c_T\, T\big/{}^{\circ}C + c_C\, C\big/wt\% + c_{TC}\, T\big/{}^{\circ}C\; C\big/wt\% + c_{C^2}\left(C\big/wt\%\right)^2\right)^2 \qquad (2)$$

where *Q* stands for the volumetric flow rate (L/min), *T* for the temperature (°C) and *C* for the NaOH concentration (wt%). The statistical analysis of the results indicated that Eq. 2 represents satisfactorily the experimental data, with coefficients of determination of 0.893, 0.913 and 0.923 within the tested ranges.

Table 1 Statistical relevant coefficients of the cleaning model for the different flow channel region

coefficient	inlet (SE)	dead area (SE)	outlet (SE)
c_0	3.270	4.452	3.508
c_Q	-0.024 (0.003)	-0.024 (0.004)	-0.022 (0.003)
c_T	-0.020 (0.010)	-0.035 (0.013)	-0.026 (0.013)
c_C	8.312 (0.922)	12.50 (1.186)	13.68 (1.146)
c_{TC}	-0.085 (0.017)	-0.143 (0.022)	-0.160 (0.021)
c_{C^2}	-2.404 (0.295)	-3.345 (0.379)	-3.569 (0.367)
R^2	0.893	0.913	0.923

Fig. 5 indicates that the regression models fits reasonably accurate over a large range of the operation parameters.

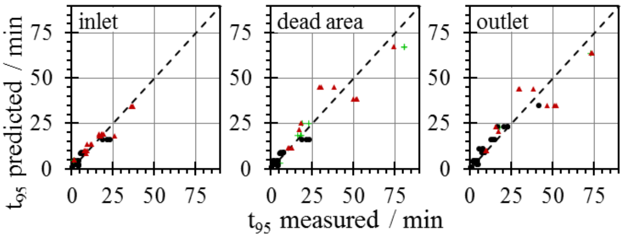

Fig. 5 Comparison of predicted (Eq. (2)) and measured cleaning time for the different flow channel region. Black dots display a continuous removal, red triangles display an adhesive removal and green crosses display a mixture of both types of removal.

For cleaning times over 25 min it must be expected with increased deviations of the predictions and the experimental results. The variations can result from the soiling and baking process. Furthermore, the results showed that measurements with large scattering underlie an adhesive removal. Further investigations in the range of middle to long cleaning time with focus on the type of removal are planned in order to improve the model.

Effects of process parameter of cleaning time

From an industrial perspective, plants and components with area of low flow velocity or stagnant flow are difficult to clean. These areas limit the cleaning time for the whole system and the efficiency of the cleaning process. Therefore, the following section illustrates the influence of the parameters for the dead area of the channel. Fig. 6 presents the contour plot for the cleaning time t_{95} in the dead area as function of the temperature and the sodium hydroxide concentration of the cleaning solution for a constant flow rate at 50 L/min.

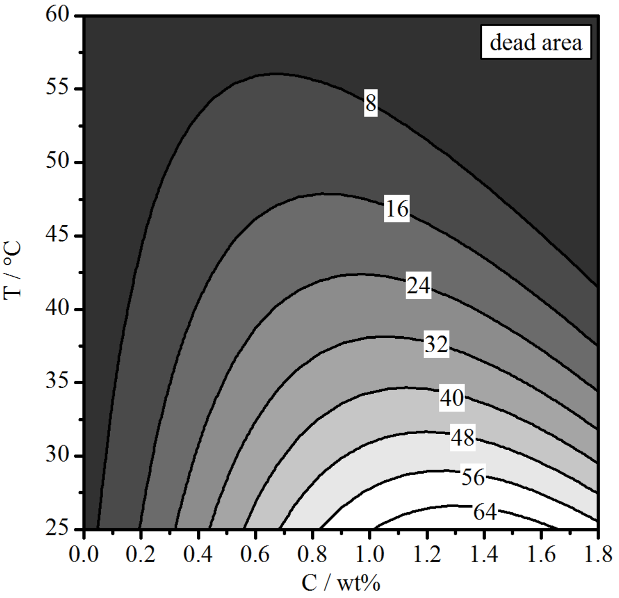

Fig. 6 Contour plot for the calculated cleaning time t_{95} (min) in the dead area at a flow rate of 50 L/min in the flow channel. C and T represent the NaOH concentration and the temperature of the cleaning solution.

The cleaning time ranges from few minutes to more than one hour. Due to the interaction between the temperature and the concentration (c_{TC} coefficient) and the quadratic effect of the concentration (c_{C^2} coefficient) the cleaning time shows a strongly non-linear dependency. The worst cleaning result prevails at low temperatures and concentrations around ~1.4 wt% NaOH. This means that an egg yolk layer can be removed at room temperature the fastest with tap water. Increasing the temperature tends to result in improved removal of egg yolk layers. If other requirements demand the use of sodium hydroxide, it should be used at temperatures above 45°C, whereby the influence of the concentration becomes negligible. This behavior is similar to the results in the inlet and outlet area of the channel but with different absolute cleaning times.

This conclusion is consistent with the FDG measurements. Fig. 7 compares the swelling and removal behavior of egg yolk layers immersed in RO water and a NaOH solution with 1.5 wt% at 30°C.

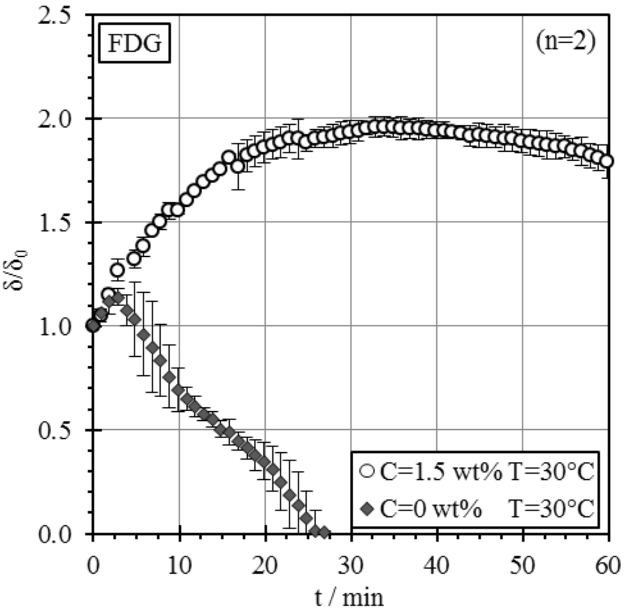

Fig. 7 The effect of sodium hydroxide concentration on the swelling (dissolution) of an egg yolk layer shown by FDG measurements of the normalized layer thickness depending on time.

In the NaOH solution the layer swells steadily to twice its initial thickness, followed by a moderate removal after ~25 min. When no alkali is present swelling is interrupted after 3 min, and after ~6 min a thickness below the initial one was observed. After 27 min the layer was completely removed at the gauged locations. The interaction between sodium hydroxide and egg yolk were also discussed by Gordon et al. 2012. They described that unbaked egg yolk layers swelled significantly in alkali but did not dissolve or erode when subject to flow stresses. A possible explanation for this unexpected behavior could be a chemical reaction between the sodium hydroxide ions and the lipids which are with an amount of 57% the main contents of the egg yolk. The most common reaction which is taking place if these two components are present is the saponification reaction illustrated in Eq. (3).

$$R-COOR+NaOH\rightarrow R-COONa+ROH \qquad (3)$$

The egg yolk containing lipids (R-COOR) and sodium hydroxide are reacting to glycerin (R-OH) and sodium soap (R-COONa). To substantiate this assumption additional examinations need to be carried out, nevertheless it could be show that the Fluid Dynamic Gauging is able to reflect the effects of the cleaning examinations in industrial scale and therefore enables an additional examination of the acting mechanisms in laboratory scale to gain a deeper understanding.

The influence of the volumetric flow rate *Q* is clarified in Fig. 8. The lines represent the predicted cleaning time for different combinations of concentration and temperature and for comparison all experimental data are plotted. The deviation of the model at long cleaning time discussed in Fig. 5, is also reflected in the wide confidence band at high NaOH concentrations. However, the principal influence of the volume flow can be assessed.

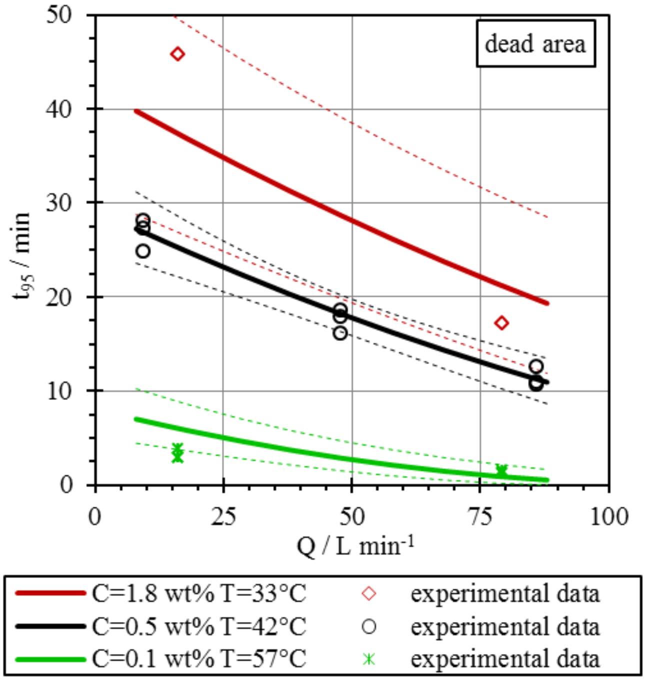

Fig. 8 Cleaning time in the dead area as function of the flow rate for three combinations of temperature and concentration. Lines are results predicted by the model with 95% confidence interval (dashed lines).

Thus, the effect of the flow rate on the cleaning time is strongly dependent on the choice of the solution’s configuration. For an unfavourable parameterization (temperatures under 45°C in combination with high NaOH concentrations) an increasing flow rate decreases the cleaning time. But in such case, even at very high flow rates, significantly longer cleaning times have to be expected compared to combinations of temperatures exceeding 50°C and tap water or low NaOH concentrations. Fig. 8 indicates the essential role of the temperature and chemical action rather than mechanical action in removal an egg yolk layer, even in the dead area with lower flow velocities.

Depending on flow behavior in the channel, the influence of the investigated process parameters (flow rate, NaOH concentration and temperature) has changed. Sinner (1960) suggests the interaction of four cleaning parameters – mechanical action, chemical action, temperature and time – to describe cleaning processes. Based on this Sinner’s circle, if one parameter is reduced, other factors must be increased to compensate (Lelieveld 2005). To assess the effect of the investigated process parameters, the percentage influence was determined. I^{i}_{effect} is defined as the fluctuation of the cleaning time Δt^{i}_{95} of the parameter concerned, divided by the total amount of all fluctuation of the cleaning time.

$$I^{i}_{effect} = \frac{\Delta t^{i}_{95}}{\Delta t^{Q}_{95} + \Delta t^{T}_{95} + \Delta t^{C}_{95}} \tag{4}$$

The fluctuation of the cleaning time corresponds to the difference of the predicted maximum and minimum cleaning time for the respective process parameter while the remaining parameters are located in middle of the parameter range.

$$\Delta t^{i}_{95} = t^{i}_{95.\max} - t^{i}_{95.\min} \tag{5}$$

Fig. 9 shows the comparison of the percentage influence of the process parameters for each channel area. Depending on flow behavior in the channel, the influence of the investigated process parameters changed. The sudden expansion reduces the flow velocity and thereby declines the mechanical action in the dead area and the outlet. Due to the reduced mechanical action downstream the sudden expansion the flow rate becomes less influence compared to the inlet. The decreased mechanical action affects the influence of the remaining factors. In the present case, the influence of the temperature increased because the chemical action remains nearly constant. From an industrial point of view, if there is an existing plant with many critical areas, it is advisable to adjust the temperature for an effective cleaning result.

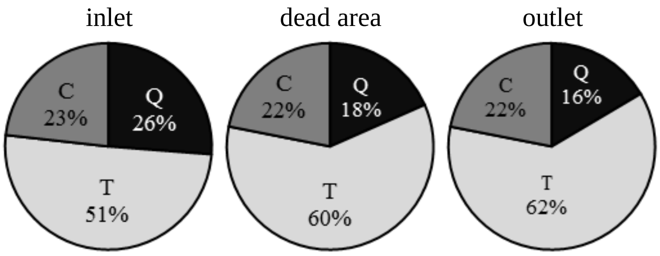

Fig. 9 Comparison of the influence of the process parameters to remove an egg yolk layer depending on the channel region.

Effects of process parameter on the type of removal

In addition to assessing the influence of the process parameters on the cleaning time, the measurements of the present work allows the investigation of the effects of the process parameters to change the type of removal.

In Fig. 10 and Fig. 11 the adhesive removal is opposed to the continuous removal of an egg yolk layer, investigated in the inlet area of the flow channel. Consistent with the authors above, after a reaction time of ~12 min with unchanged layer thickness, the egg yolk layer was completely removed in large chunks (see Fig. 10). In contrast, the continuous removal is characterized by a steady decrease of the layer thickness (see Fig. 11). But it is uncertain whether it is caused by dissolution effects or by removing small particles of the layer.

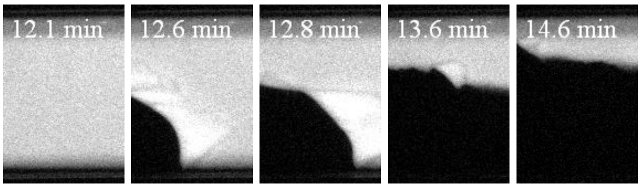

Fig. 10 Adhesive failure: removal of an egg yolk layer in large chunks at 30°C, 1.0 wt% NaOH, 48 L/min. Flow direction from left to right.

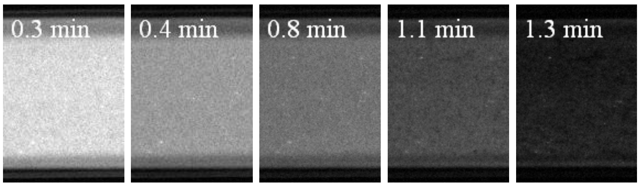

Fig. 11 Cohesive failure: continuous removal of an egg yolk layer at 33°C, 0.2 wt% NaOH, 80 L/min. Flow direction from left to right.

For concentrations in the range of 0.5 to 1.5 wt% NaOH and low temperatures adhesive removal can be expected, whereas continuous removal dominates above 50°C for all concentrations and all flow rates. However, concentrations below 0.1 wt% NaOH exhibit continuous removal also at room temperature.

Apart from these classifications, in this study also adhesive processes in the channel inlet and continuous removal in the outlet could be found (see Fig. 12). Attention should be drawn here especially to the influence of surface treatment.

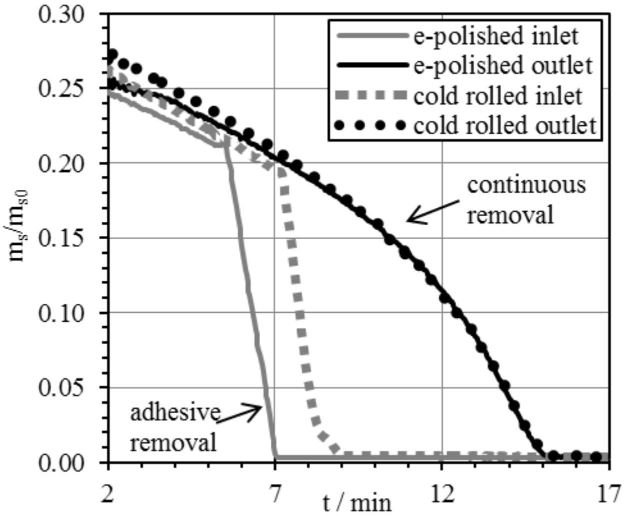

Fig. 12 Normalized mass of remaining soil for different surface treatments depending on cleaning time and channel region. Constant cleaning parameters Q = 48 L/min, T = 42°C, C = 0.5 wt% NaOH.

For constant cleaning conditions, the normalized mass of soil is shown for a cold rolled and an electro-polished stainless steel surface. Whereas the continuous removal in the outlet leads to no change in cleaning process, adhesive removal in the inlet results in significant differences in cleaning time. This result comes from the fact that in case of a continuous removal the forces between soil and the surface are only relevant for the final removal of the soil layer. In agreement with other authors (Saikhwan 2006, Bobe 2007, Mauermann 2009), therefore, the surface properties (roughness, surface energy) can influence the cleaning process. But the based mechanism is crucial, which depends on the soil properties and there interaction with the cleaning solution. First results demonstrate that the influence of the surface properties must be assessed in relation to the type of removal. Fig. 13 shows the comparison of the cleaning time for a cold rolled and an electro-polished stainless steel surface for each channel area. In the inlet area the electro-polished surface was associated with significant reduction of the cleaning time (no overlapping of the error bars). In contrast, the dead area and the outlet showed no clear differences in cleaning times. Whereas in the dead area and the outlet the removal of the egg layer based on a cohesive failure or a mixture of both types of removal.

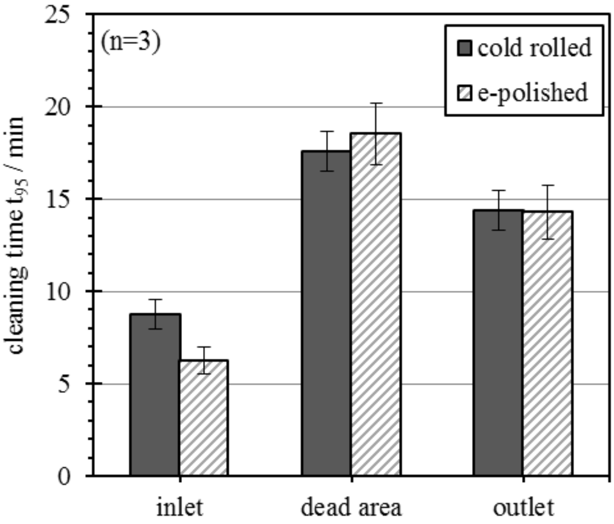

Fig. 13 Cleaning time for different surface treatments depending on the channel region at constant cleaning conditions (Q = 48 L/min, T = 42°C, C = 0.5 wt% NaOH). Bars represent standard deviation.

CONCLUSIONS

The optical detection method according to Schöler et al. (2009) was further developed and allows the temporally and spatially investigation of cleaning processes of various soils. The influence of cleaning process parameters and their interactions on the cleaning time are demonstrated for a baked egg yolk layer as a model food soil in conjunction with a statistical analysis. The flow channel with a sudden expansion allows the consideration of shifting influences of the process parameters depending on the flow behavior. This enables recommendations for cleaning in critical areas at industrial plants. Against expectations, the presence of sodium hydroxide ions leads to an increase of the cleaning time for the investigated baked egg yolk in comparison to pure water. This result is in agreement with FDG measurements, which were chosen here as a lab scale experiment to predict chemical influences on cleaning at industrial scale. Based on current knowledge, the saponification reaction is assumed as a possible explanation. Further rheological investigations are planned to understand the nature of the baked egg yolk at different temperatures and NaOH concentrations. The micromanipulation technique (Liu 2006b) will be used to receive more information on the adhesive and cohesive forces within soil layers.

In addition the optical detection method allows the investigation of the type of removal and of the influence of process parameters on the cleaning time. The results show that the process parameters affect the primary type of removal significantly. Furthermore, results based on two exemplary surfaces show that the influence on cleaning cannot be assessed in general. At the current stage it is assumed that the surface properties have an influence on the cleaning process only for adhesive failure. In conjunction with the fact, that the type of removal can be changed by variation of process parameters, it comes out that all variables of the Sinner's circle (condition of the soil, the design of the plant resp. surface properties and the condition of the cleaning fluid) have to be considered to understand the cleaning process in detail. The approach of this work offers the potential to predict the cleaning effects of changing process parameters and provides an incentive to evaluate the influence of surface treatments.

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NOMENCLATURE

C	NaOH concentration of the solution, wt%
d_h	hydraulic diameter, m
I^i_{effect}	influence of an effect, %
m_{s0}	initial surface soil mass, kg m^{-2}
m_s/m_{s0}	normalized mass of soil on the coupons, -
n	numbers of experiments, -
Q	volumetric flow rate, m^3 s^{-1}
Q^2	estimate of the predictive capacity of the model, -
R^2	coefficient of determination, -
Re_{inlet}	Reynold number in inlet channel, -
Re_{outlet}	Reynold number in outlet channel, -
SE	standard error, -
t	time, s
t_{95}	cleaning time, s
Δt^i_{95}	fluctuation of predicted cleaning time, s
T	solution temperature, °C
δ	thickness of soil layer, m
δ_0	initial thickness of soil layer, m
η	dynamic viscosity , kg m^{-1} s^{-1}
ρ	liquid density, kg m^{-3}

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