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INVESTIGATION OF BLOCKING PHENOMENA IN CLEANING OF MICRO HEAT EXCHANGERS

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ABSTRACT

Micro heat exchangers enhance heat and mass transfer compared to conventional heat exchangers which promotes an increased energy efficient application in many industries. Despite these advantages, micro structured components in general are prone to fouling and therefore to blocking phenomena which limits their industrial application. Since fouling is not always avoidable, cleaning strategies are essential and have to consider phenomena which are specific for micro structured components. Since swelling always occurs at the beginning of the cleaning of protein-based deposits, leading to an increase in the deposit volume and thus to a reduction in cross-section, blockage and a corresponding process interruption may still occur even during cleaning. In order to be able to develop an automated cleaning process for micro heat exchangers, swelling and associated blocking phenomena need to be identified first to control swelling during cleaning. For experimental investigations, a dyed model soil system is cleaned first in a diffusion-based setup to investigate the swelling and dissolution without flow impact and afterwards in a one-dimensional micro structured flow channel through the circulation of a sodium hydroxide solution. First results indicate options to reduce swelling and thus to prevent blockage during cleaning.

INTRODUCTION

Micro components are defined as having one characteristic dimension smaller than 1 mm, resulting in small hydraulic diameters and correspondingly large surface-to-volume ratios. Hence, micro heat exchangers enhance heat and mass transfer compared to conventional heat exchangers which promotes an increased energy efficient application in pharmaceutical, chemical and food industries [1]. Despite this advantage, micro structured components in general are prone to fouling and blocking phenomena due to their small dimensions which limits their industrial application [2,3,4]. Fouling is a well-known concern at the macroscale but is more severe at the microscale because of the small cross flow sections and the much higher risk of blockage, leading to process disturbances. Nevertheless, little attention is paid to the study of fouling at microscale [2,5]. Schoenitz et al. noted that most research of fouling in micro scale focuses on particulate fouling, due to the beneficial effect of a micro scale in processes involving particles. However, there is a lack of attention towards chemical reaction fouling, with no publications on this topic [6]. Even though fouling inhibition and reduction options are under consideration for the micro scale [7,8], fouling is not always avoidable. Besides cost and energy efficiency reasons, fouling is also causing issues regarding hygiene and product quality. Therefore, cleaning strategies are necessary, thus a systematic investigation of the cleaning behavior of micro structured equipment is essential. Preliminary studies have already shown first cleaning options and monitoring methods for the microscale [9]. Hence, this contribution presents a systematic investigation of blocking phenomena during cleaning of heat exchangers on the micro scale.

Fundamentals of cleaning

The formation of swellable deposits, such as protein or polymer-based deposits, is a common challenge in the process industry as they are classified as both reaction and particulate fouling, depending on which type is controlling [10, 11]. A frequently used model soil system for this category in macro scale are protein-based deposits such as whey protein isolate [12]. The cleaning of protein-based deposits is initially divided into three phases: the swelling, uniform and decay stages, as shown in Figure 1. This process can be quantified via the cleaning rate according to [13], which is defined as the mass to be cleaned per fouled area and time. As soon as the deposit is brought into contact with the cleaning agent, the cleaning agent diffuses into the deposit, which results in swelling of the deposit. Therefore, the described swelling stage is characterized by an increase in the cleaning rate. Once a constant cleaning rate is reached, the uniform cleaning stage begins, where the deposit is removed

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at the maximum cleaning rate. As soon as only isolated patches are left, rather than a consistent deposit, the cleaning rate drops and the decay stage begins. Cleaning is completed as soon as the cleaning rate reaches a value of 0 $g\cdot m^{-2}\cdot s^{-1}$. This process can also be identified on a micro scale. As the hydraulic diameter decreases, it becomes apparent that the first cleaning stage is critical for the small dimensions of the micro components, since they may still block even during cleaning. This poses the risk of process failure during cleaning regardless of the possibility of cleaning.

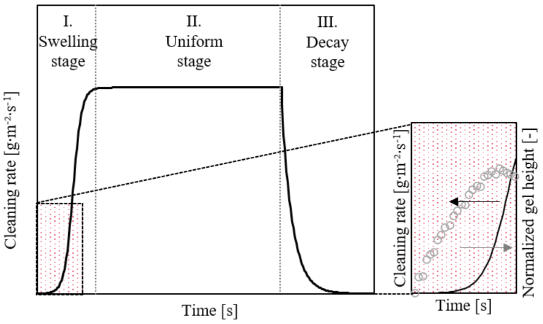

Fig. 1. Cleaning stages of a WPI gel defined by cleaning rate (black line) and the impact of the swelling height (grey circles) on cleaning rate, adapted from [14,15].

The cleaning process and thus cleaning rate, is dependent on the used cleaning parameters. These cleaning parameters are summarized in the so called “Sinner’s circle”, which describes the qualitative impact of the cleaning parameters temperature, e.g. cleaning solution temperature, chemistry, e.g. cleaning solution concentration, mechanics, e.g. flow velocity, and cleaning time on the cleaning efficiency. Consequently, this means that if one parameter is decreasing another parameter needs to be increased, e.g. if the cleaning solution temperature is lower for example the cleaning time needs to be higher, as shown in Figure 2. With regard to the micro scale, the Sinner’s circle with its only four parameters could has its limits. While the well understood relations of the parameters on a macro scale basis lead to effective cleaning, these could be counterproductive for cleaning on a micro scale basis caused by the different geometries. Regarding mechanical impact on a macro scale basis a higher flow velocity and therefore higher Reynolds numbers are always preferred. Due to extensively high pressure drops in micro scale these are not achievable for the latter. With respect to the enhanced heat transfer on the micro scale, high temperatures typically associated with the macro scale may not be necessary for effective cleaning on a micro scale. Concerning the chemical impact, many authors report an optimal cleaning solution concentration for the macro scale, with regards to the shortest cleaning time. In comparison to cleaning options in the micro scale, an optimal cleaning solution concentration could lead to excessive swelling and eventually increases the risk of blocking. The introduction of a scalability parameter, which sets the Sinner’s circle parameters based on the scale of the investigated system, could solve this issue.

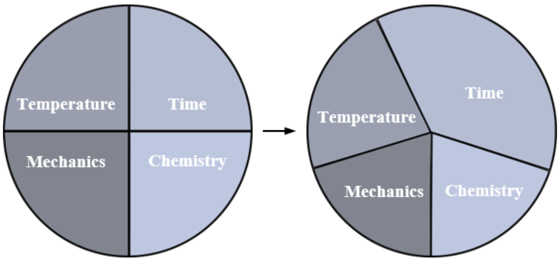

Fig. 2. Qualitative relation of the cleaning parameters summarized in the Sinner’s Circle.

Fundamentals of swelling

As already mentioned, the swelling stage is one of the phases of the cleaning process and especially crucial for the micro scale. The swelling stage consists, based on the cleaning rate, of two parts, as shown in Figure 1. First an induction period, where the protein concentration in solution and therefore the cleaning rate stays constant at nearly zero [15]. Secondly, the induction is followed by a sharp increase in the cleaning rate, which indicates the start of the gel's dissolution. However, the actual swelling of the gel, i.e. the material extension, occurs already during the induction phase [14], which is not visible based on the cleaning rate. As shown in Figure 1, the swelling of the gel proceeds asymptotical until it reaches its maximum value. In comparison to the cleaning rate this maximal swelling height determines the point at which the dissolution of the gel becomes apparent [14]. Hence, the swelling of the gel is mandatory for dissolution and therefore for cleaning.

Regarding the potential risk of blockage due to swelling while cleaning in the micro scale, the material extension during the swelling stage is the most relevant process. Swelling of whey protein-based gels was extensively studied on a macro scale basis, e.g. Saikhwan et al. introduced different types of swelling, characterized by the slope of the height increase. The authors report that whey protein swelling shows a linear height increase until it reaches a plateau [16]. To investigate swelling multiple parameters were taken into account [14,16], e.g. the swelling rate, the swelling time and the extent of swelling. According to the swelling type the swelling rate is determined by the slope of the height increase. The swelling time is defined as the time until the maximal gel height is reached, hence before dissolution starts. The extent of swelling is the dimensionless result of the difference between the gel height at the beginning and the maximal swelling height, as stated in equation (1).

$$s^* = \frac{h_{max}-h_0}{h_0} \qquad (1)$$

In order to prevent process failure and to develop a cleaning protocol for micro heat exchangers, the blocking phenomena during the cleaning of swellable deposits are investigated by quantifying the effect of the cleaning parameters of the Sinner's circle. First suggestions for improvement of a cleaning protocol are presented.

MATERIAL AND METHODS

To characterize blocking phenomena and also options for preventing blockage caused by cleaning, it is essential to understand the underlying causes of this blockage regarding swelling. Therefore, two different evaluation systems are considered: a diffusion-based setup with indentation measurement and a more complex flow system in a test rig, resembling the whole cleaning process in the micro scale.

Model soil system

To conduct reliable blocking and cleaning experiments, especially in micro structured equipment, comparable and well-defined initial conditions, such as deposit height, are essential. However, soil deposits formed under flow conditions tend to deviate significantly even if the process parameters remain constant. Thus, the development of a preparation method for model soils on the basis of a defined soil system is absolutely necessary for comparable cleaning investigations. In this regard a thermally induced colored hydrogel based on whey protein isolate (WPI) is used as a model soil system. The model soil layers are generated on stainless-steel sample plates in a modular aluminum mold, which is designed in order to be able to produce the generated layers with an average height of (0.52 ± 0.03) mm and different defined widths, e.g., 2.0 mm, 4.5 mm, 9.0 mm and 18.0 mm, to resemble different channel configurations. The manufacturing procedure of the model soil system is explained in detail in [9].

Diffusion-based experimental setup

The experimental investigation of diffusion-based swelling, i.e. without an acting flow regime, is carried out in a diffusion cell [17] enabling indentation measurements via a Texture Analyser “TA.XTplus” and an additional moving system “ALIS600” by Stable Micro Systems. The diffusion cell has an inner width of 18 mm and a length of 80 mm, equal to the dimensions of the tested model soil systems of 18·80 mm², to ensure that diffusion of the cleaning solution into the gel is only possible by the upper surface. To investigate swelling under the influence of temperature a closable water bath is used, which can be heated externally by a thermostat. As a measuring probe a stainless-steel cylinder with an outer diameter of 3 mm is used for testing. The tested positions on the gel stayed constant due to the available automatic positioning, as well as the time steps between two measurement points. This enables the time-resolved heights of the gel to be determined as a function of temperatures between 22 °C and 60 °C and sodium hydroxide concentrations between 0 wt%, i.e. pure water, and 2 wt%. Subsequently, these measurement results are characterized with regard to various swelling parameters, which are described in the section *Quantification of swelling*.

Flow-based experimental setup

The experimental investigation of blocking phenomena during cleaning micro structured equipment under the influence of an additional flow is carried out in a rectangular, modular and optical accessible flow channel. The clean flow channel has a height between (0.60 ± 0.01) mm and (1.00 ± 0.01) mm, a width of 18 mm and a length in flow direction of 80 mm, resulting in a micro component in one dimension, with a surface to volume ratio between 4 -20 mm²/mm³. Figure 3 clarifies the design of the different channels with the varying channel heights $h_{ch,end}$ = (1.00 – 0.60 ± 0.04) mm. In combination with the (0.52 ± 0.03) mm high model soil system, the different channel fittings result in channel heights $h_{ch,0}$ between (0.08 - 0.48 ± 0.04) mm at the beginning of each cleaning experiment. Hence, a noticeably high change of the cross-flow section is detectable, which complicates the calculation and definition of the flow velocity, Reynolds number and other cross-flow section related values. Regarding this issue a comparison between cleaning on a micro versus on a macro scale is under consideration, but not yet published. As a first attempt cross-flow section related values are calculated based on the clean state of a micro channel ($h_{ch,end}$) and are used as values for comparison.

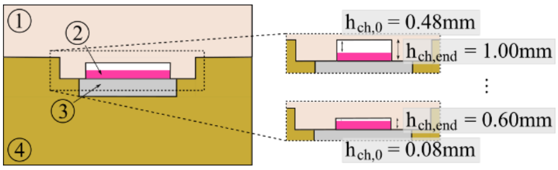

Fig. 3. Cross section of the modular flow channel, with: 1 - lid with exemplary different channel heights $h_{ch,end}$ = 1.00-0.60 mm, 2 - deposit, 3 - sample plate and 4 - base.

The corresponding test rig is operated in single pass mode (“lost cleaning”), shown in Figure 4, i.e. the cleaning solution is only used once. To monitor the progress during the tests, the test rig is equipped with type T thermocouples, two absolute pressure sensors at the channel inlet and outlet for determining the pressure drop, as well as an optical observation via images of the flow channel taken by a commercial digital camera with a macro lens. Based on the optical observation, additional information like surface coverage can be calculated with temporal and spatial resolution. Furthermore, samples of the cleaning solution are collected continuously at the channel outlet over the course of an experiment to determine the protein content of the solution via Bradford protein assay. Based on the time-resolved protein concentration, the cleaning rate can be determined. As already mentioned, the cleaning rate is defined as the cleaned gel mass m per soiled area A_{sps} and time t, as shown in equation 2. The soiled area A_{sps} is calculated by means of the sample plate area A_{sp} times the coverage of the sample plate at the beginning of each trial (DoC_0) as a result from an image analysis. In addition to the pressure drop and sampling option the cleaning process is also tracked via image analysis. The image analysis is performed as follows.

$$R_C = \frac{m}{DoC_0 \cdot A_{sp} \cdot t} \tag{2}$$

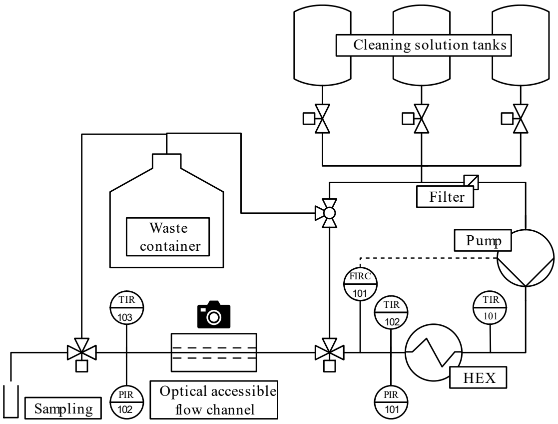

Fig. 4. Flow diagram of the micro cleaning test rig.

All taken images are processed via python image operation using OpenCV, a detailed description of the process is presented in [9]. The processing of the images is used to automatically calculate the coverage of the sample plate and thus determine the removal rate. Later on, the image analysis should be used to detect the position of blockage to map blocking. Accordingly, another output of the image analysis tool is the calculated degree of coverage DoC, which can be derived from the pixel data, with the count of clean pixels (n_{cp}) and not clean pixels (n_{ncp}), using equation 3.

$$DoC = \frac{n_{cp}}{n_{cp} + n_{ncp}} \tag{3}$$

For the investigation of blocking phenomena thin channels with heights smaller than $h_{ch,end} \leq (0.65 \pm 0.01)$ mm are used to provoke blocking. These trials are compared to non-blocked channels during cleaning with channel heights $h_{ch,end} \geq (0.75 \pm 0.01)$ mm, to characterize blocking during cleaning. For comparison, the temperature, sodium hydroxide concentration and flow velocity v_{end} for a channel height are held constant, while only the channel height and therefore the mass flow rate varies. To investigate the cleaning behavior of these partially blocked channels, for channel heights $h_{ch,end} \leq (0.65 \pm 0.01)$ mm the known parameters of Sinner's circle, temperature, mechanical impact and chemistry are varied. Therefore, the temperature is varied between 45 °C and 70 °C, the sodium hydroxide concentration between 0.5 wt% and 1.5 wt%, and the velocity v_{end} between 0.11 ms^{-1} and 0.18 ms^{-1}. In order to investigate the underlying phenomenon of blocking, i.e. the swelling, the effect of an additional flow regime is examined using the same test rig. Therefore, all cleaning parameters are held constant, with an exception of the flow velocity, which is varied between 0.03 mm and 0.16 mm. Finally, these findings can be compared with the results from the "no flow" experiments of the diffusion-based setup, to quantify the impact of the addition of flow on cleaning in micro scale.

Quantification of swelling

As already mentioned, the cleaning and especially the swelling process can be monitored specifically due to pressure drop. Pressure drop is dependent on channel cross flow section and in relation to fouling and cleaning dependent on soil layer height. This relation is used to quantify the unknown soil layer height while cleaning in a micro scale, by means of aluminum based artificial fouling layers resampling the soil layer heights during cleaning. The method described by Spiegel et al. [9] is used to gain a calibration curve which fits the experimental data of pressure drop versus soil layer height. Therefore, artificial fouling layers with heights ranging from 0.0 mm to 0.8 mm, depending on the channel's height, are inserted into the specified micro flow channel. The channel is fastened using a torque wrench set at 60 cNm to ensure uniform starting conditions for all calibration trials. Water is used as a test fluid, as there are only neglectable differences in densities between water and low concentrations of the cleaning fluid sodium hydroxide. All pressure drop values, for each of the utilized sample plates, have been monitored for two minutes and repeated at least twice.

Figure 5 shows an exemplary gained calibration curve for $\vartheta = (43.57 \pm 0.39)$ °C and $\dot{m} = (75.00 \pm 0.81)$ $gmin^{-1}$. Error bars are shown for all measurement points, but are sometimes smaller than the used symbols. In accordance with theory, the pressure drop increases with decreasing cross flow section and therefore with increasing inserted sample plate heights. Even though the shown power function fits the experimental data quite well ($R^2 = 0.878$), the difference between pressure drop values for sample plates thinner than 0.4 mm are small (e.g. $\Delta p_{h\_sp=0.2mm} = (8.15 \pm 0.42)$ mbar vs. $\Delta p_{h\_sp=0.3mm} = (8.69 \pm 0.60)$ mbar), which could result in extensive deviations for calculations by calibration curve for gel heights $h_g \leq 0.4$ mm. Nevertheless, the calibration fits the relevant region regarding swelling quite well. Thus, the calibration method is suitable for quantifying the swelling progress and also the beginning of cleaning.

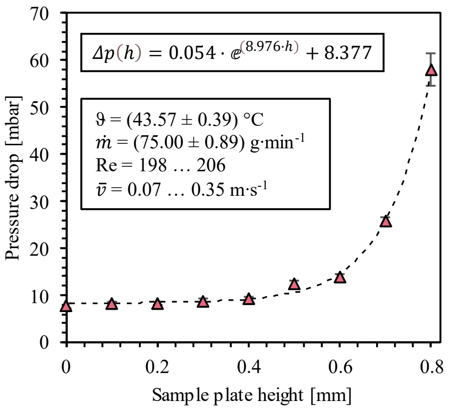

Fig. 5. Calibration curve for pressure drop versus sample plate height, gained from at least three and up to six replicates. Reynolds number and flow velocity vary due to the changeable cross-flow section.

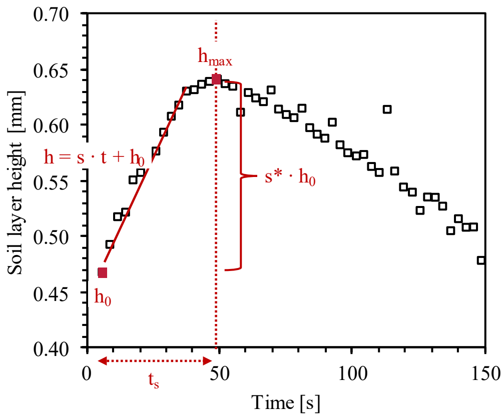

Fig. 6. Calculated height from pressure drop and the related values (s - swelling rate, s* - extent of swelling, t_s - swelling time) to quantify swelling due to height from pressure drop and indentation measurements.

An exemplary calculated height curve of a model soil system during cleaning is shown in Figure 6. The course of the calculated height follows the pressure drop. In the beginning of the cleaning process, the pressure drop increases due to the cross-sectional reduction caused by the swelling of the deposit, therefore the height increase of the gel is also visible. Once the swelling maximum is reached, the pressure drop decreases, thus the gel height decreases and removal starts.

To quantify the influence of the cleaning parameters on swelling, the earlier described values: swelling rate s, swelling time t_s and extent of swelling s* are taken into account. Due to the complexity of the process, all three mentioned parameters are considered and shown exemplarily in Figure 6.

Independently from the gained measurement technique, pressure drop (flow-based setup) or indentation measurement (diffusion-based setup), the three shown parameters could be observed and are applicable, hence are used for the investigations in both setups.

RESULTS AND DISCUSSION

To investigate blocking phenomena in micro scale, first blocking and secondly swelling are described and evaluated. Swelling as the cause for blocking during cleaning, is evaluated due to a variation of the cleaning parameters known from the Sinner’s circle.

Characterization of blocking

To characterize blocking, first blocking and the forms of blocking, as blocked and partially blocked, needs to be defined. Blockage during cleaning occurs, if the cross-flow section is smaller than the swelling height of the gel. Due to imperfections in the gel, especially at the edges of the model soil system, as shown in Figure 7 as a bubbly part of the gel on the right side, the cleaning agent remains able to pass the channel. Therefore, the cleaning agent can continue to circulate through the channel and the deposit remains removable. However, this results in a partial blockage of the channel. As an example, Figure 7 shows the microchannel to be cleaned and an exemplary cleaning sequence of a channel been partially blocked versus a non-blocked channel; the flow direction of the cleaning agent is from right to left; cleaned sections are pictured in black and still soiled sections in white. In the first section on the left, the model soil can be detected over the entire length of the sample plate for both cases. The bubbly and uneven part of the gel in the channel inlet is removed first, without causing any blockage in this area. The cleaning process in a non-blocked channel proceeds uniform with respect to the flow pattern. In contrast the cleaning process of a partially blocked channel shows removal only in the top region of the shown sequence in Figure 7. However, this shows that the cleaning front changed, from the large surface area, in a non-blocked channel, to the side of the gel in longitudinal direction. This results in three different types of removal as shown in Figure 7: the formation of an extended island a.) on one side of

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the channel, b.) in the middle of the channel, or c.) in multiple clusters on both channel sides.

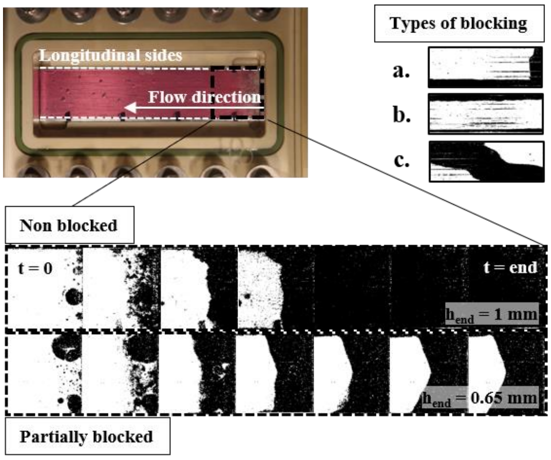

Fig. 7. Top view of a partially blocked versus non-blocked channel due to swelling, with a summary of the observed blocking types. The binary images show soiled parts in white and cleaned parts in black.

Comparison of different monitoring methods

To detect blocking during cleaning in the described test rig the mentioned methods, pressure drop measurement, cleaning rate calculation and coverage detection, are considered. The blocking behavior can be quantified through the degree of coverage. The degree of coverage is normalized by means of the first detected degree of coverage DoC_0 at time t_0, thus all experiments are comparable. As shown in Figure 8 with black non-filled symbols, the degree of coverage of a non-blocked channel ($h_{ch,end}$ = (1.00 ± 0.01) mm) shows for most of the cleaning time only a small linear decrease. This is due to the fact, that the calculation of the coverage is only 2D-based and the information of the deposit height is missing. With the beginning of the last cleaning stage the coverage decreases significantly with a higher linear slope, until no soil is left on the sample plate. In comparison the degree of coverage of a partially blocked channel has only one nearly linear decrease section, which represents the removal of the model soil system in relation to the cleaning area along the longitudinal side of the channel.

As can be seen for partially and non-blocked channels, only removal can be observed due to coverage. Thus, swelling as part of cleaning and cause of blocking cannot be directly measured by means of coverage but can be observed by means of the pressure drop. In Figure 8 the pressure drop is also shown normalized by means of the pressure drop at the beginning of each cleaning trial. For a non-blocked channel, the pressure drop is increasing first due to the swelling of the soil and after reaching a maximal pressure drop, the removal starts, consequently the pressure drop decreases rapidly. Therefore, all normalized pressure drop values above one indicate a layer volume higher than the initial layer volume, while values less than one indicate a layer volume less than the initial volume. In comparison the pressure drop curve for a blocked channel has a similar trend. First, the pressure is increasing like a non-blocked channel. However, due to the smaller dimension, the swelling of the gel is limited by the channel geometry, which leads to a decrease in the slope of the pressure drop. After that maximum pressure drop is reached and remains nearly constant for a certain time, the pressure drop decreases and cleaning continues in the longitudinal direction of the channel. Note that the absolute pressure drop values for a partially blocked ($\Delta p_{max} \approx$ 600 mbar) in comparison to a non-blocked channel ($\Delta p_{max} \approx$ 50 mbar) are one magnitude higher. Therefore, the normalization removes this difference in the magnitudes. Hence, partial blockage is evident in the pressure drop primarily through a higher absolute pressure drop. However, it is harder to differentiate a partially blocked channel from a non-blocked channel using pressure drop measurements, because it is an integral measurement and lacks spatial resolution.

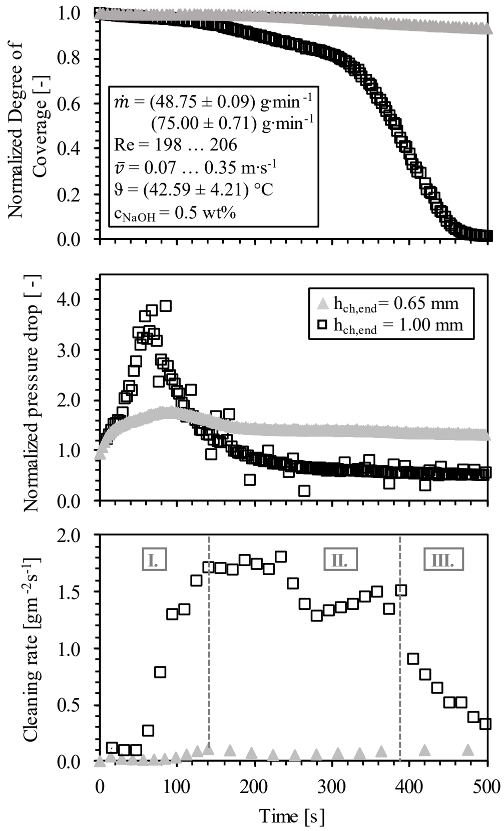

Fig. 8. Application and comparison of different monitoring methods for cleaning in micro scale for a non-blocked (squares, unfilled) versus a partially blocked channel (triangles, filled). Process parameters of Reynolds number and flow velocity are defined for a non-blocked channel and vary due to the change of the cross-flow section between the given values.

For the cleaning process of a non-blocked channel the cleaning rate shows the same three stages as known from the common macro scale, as shown in Figure 8 with the numbers I-III. The mean cleaning rate gained from the uniform stage, defined as a parameter for the overall cleaning performance, reaches comparable values as known from the macro scale. However, a comparison of the cleaning efficiency in both scales, taking mass transfer into account, is under consideration. In comparison, cleaning in a partially blocked channel can also be identified through protein quantification, resulting in a lower cleaning rate. This confirms the findings obtained by means of pressure drop and coverage that cleaning is not prevented and takes place slowly.

Cleaning of partially blocked channels

In order to be able to detect the differences in cleaning behavior, the cleaning processes in a non-blocked channel and a partially blocked channel are compared to each other. The different channel configurations have been realized by means of the described channel inserts of different heights. All other process parameters, such as temperature, cleaning agent concentration and flow velocity, remain constant. To monitor cleaning of partially blocked channels the pressure drop is less suitable for considerations. The surface of the micro channel is soiled just from one side. As shown in Figure 3, only the bottom of the channel is soiled. When it comes to blocking the removal is uneven, as shown by the different types in Figure 7. Due to the integral pressure drop measurement, removal, as a local phenomenon, is hard to detect. Similar results can be observed for non-blocked channels, where other monitoring methods are preferred for cleaning time detection [9]. Due to the local resolution and high accuracy, the degree of coverage offers a feasible option for monitoring the cleaning of partially blocked channels.

Effect of partially blockage on cleaning

Figure 9a. shows the cleaning process of non-blocked channels with channel heights at the end of cleaning of $h_{ch,end}$ = (1.00 ± 0.01) mm and $h_{ch,end}$ = (0.75 ± 0.01) mm with unfilled symbols and the two partially blocked channels with channel heights at the end of cleaning of $h_{ch,end}$ = (0.65 ± 0.01) mm and $h_{ch,end}$ = (0.60 ± 0.01) mm with filled symbols. For non-blocked channels, as mentioned before, initially no major change in the coverage can be seen until a large part of the gel depth has been removed. With the beginning of the removal of the final layer the cleaned plate starts to appear underneath. At this point, a linear removal rate can be observed, shown in Figure 9b Ia. In contrast, the partially swollen channels show a different cleaning behavior, with the removal rate remaining constant from the start of cleaning, as shown in Figure 9b, marked with IIa. As shown in Figure 9b IIb. a higher removal rate may occur at the beginning of cleaning due to an uneven distribution of the model soil system at the channel inlet, resulting in a removal rate peak even though the channel is blocked. However, the described value of the removal rate i is not greater than i = -$2{\cdot}10^{-3}$ s^{-1}. Thus, both cases show constant phases in the respective removal rates, allowing partially blocked and non-blocked channels to be compared and differentiated. These phases can be identified via the removal rate derived from the rate of change from the degree of coverage.

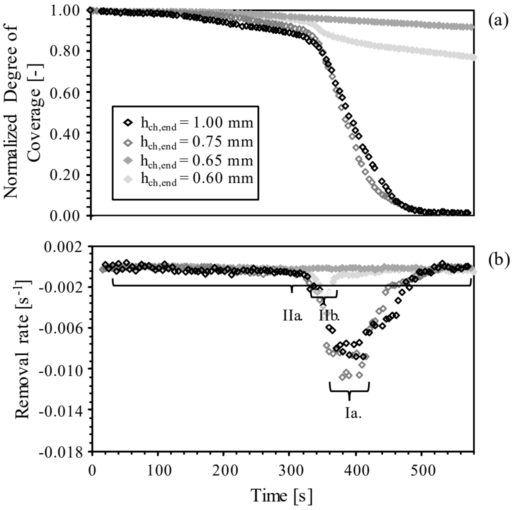

Fig. 9. Difference in cleaning between partially blocked (filled symbols) and non-blocked channels (unfilled symbols) due to (a) normalized coverage and (b) removal rate.

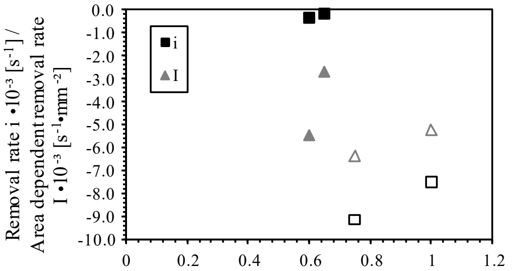

Fig. 10. Time dependent removal rate i and cleaning area dependent removal rate I in comparison to channel height and thus a comparison between partially blocked (filled symbols) and non-blocked channels (unfilled symbols).

When comparing the cleaning process of a partially blocked channel with a non-blocked channel, the constant removal rate of -$8.31{\cdot}10^{-3}$ s^{-1} for a non-blocked channel is over 30 times higher than for a partially blocked channel with a constant removal rate of -$0.26{\cdot}10^{-3}$ s^{-1}. Due to the cleaning front change the area of the cleaning surface is reduced by a factor of 12 - 23 from about 1,440 mm²

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to about 117 - 63 mm², while the mass to be cleaned remains identical. Conversely, this means that the removal rate is reduced by a factor of about at least 12 - 23. If the removal rate is adjusted accordingly for the cleaning surface, the removal rate for a partially blocked channel is in the same range as for a non-blocked channel, as shown in Figure 10. However, it could be assumed that the normalized cleaning rate for a partially blocked channel is comparably higher. This could be due to the smaller flow region caused by the blocked part of the channel and the resulting increase in flow velocity. At the same time, the mass flow rate remained constant.

Cleaning options of partially blocked channels

The high likelihood of blockages due to the small size of micro components necessitates the need for cleaning options. Therefore, the first investigated cleaning option for the described partial blockage is cleaning with the same parameters, regardless of the mentioned lower removal rate.

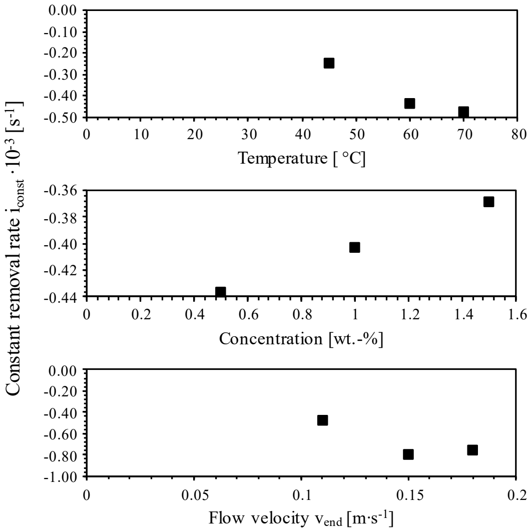

Fig. 11. Effect of cleaning parameters on the cleaning performance of partially blocked channels.

The cleaning agent concentration was varied between 0.5 wt% and 1.5 wt%, the cleaning agent temperature between 45 °C and 70 °C, and the flow velocity for a clean channel at the end of the cleaning process v_{end} between 0.11 ms^{-1} and 0.18 ms^{-1}, which corresponds to Reynolds numbers between 201 and 554. Due to extensively higher pressure drops in micro scale, in comparison to the macro scale, only laminar flow regimes can be achieved in most of the applications. The constant removal rates of the parameter variations are shown in Figure 11. Please note that these are only single trials. The shown results are derived from the rate of change from the degree of coverage (Figure 9). As the temperature increases within the considered range, the removal rate also increases. While this is known from other cleaning tests, it is also confirmed here for partially blocked channels. As known from the macro scale, the removal rate should tend to decrease with increasing cleaning agent concentration over 0.5 wt% [18]. Similar results can be seen for partially blocked channels. As the flow velocity increases, the removal rate also increases, as also known from non-blocked channels and the common macro scale.

Due to the shown significant low removal rates it is necessary to evaluate options to prevent blockage from occurring.

Effect of cleaning parameters on swelling

To investigate the effect of the cleaning parameters, e.g. cleaning agent concentration, temperature and flow velocity, on the initial swelling during cleaning, following parameter variations were carried out.

Diffusion-based impact of swelling

First of all, the temperature and sodium hydroxide concentration of the cleaning solution is varied in the diffusion-based setup, to explore the impact on swelling in a less complex system. The temperature during the trials has been varied between 22 °C and 60 °C, as being relevant cleaning temperatures known from the macro scale. As the temperature increases, the swelling rate increases as well, as shown in Figure 12. That is not surprising, as the molecular movement into the gel also increases as the temperature rises. Accordingly, the respective swelling time is shorter as the temperature increases. Thus, indicating that the dissolution starts earlier, as the reaction to dissolve the protein chains in the gel takes place faster due to the higher reaction temperature and thus reaction rate, thereby favoring the dissolution. An exception to that is the water-based solution, as the swelling time stays constant, while the swelling rate increases. In fact, water does not cause dissolution of the gel, resulting in a constant gel height and therefore a swelling time for about the whole testing time. The extent of swelling slightly decreases with increasing temperature, with the already mentioned exception for water. In contrast other authors demonstrated that the swelling ratio is not affected by a higher process temperature [19]. These contrasting observations may be attributable to the slightly different operating conditions, as no equilibrium state may have been reached here and dissolution could be observed. However, regarding the investigated process conditions during cleaning in the micro scale, the values shown are indicative even without a state of equilibrium. Accordingly, an increase in the cleaning temperature leads to faster swelling and increases the blocking potential. Consequently, increasing the temperature, which is normally useful for cleaning, proves to be

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potentially disadvantageous in the case of swelling handling regarding cleaning of micro components.

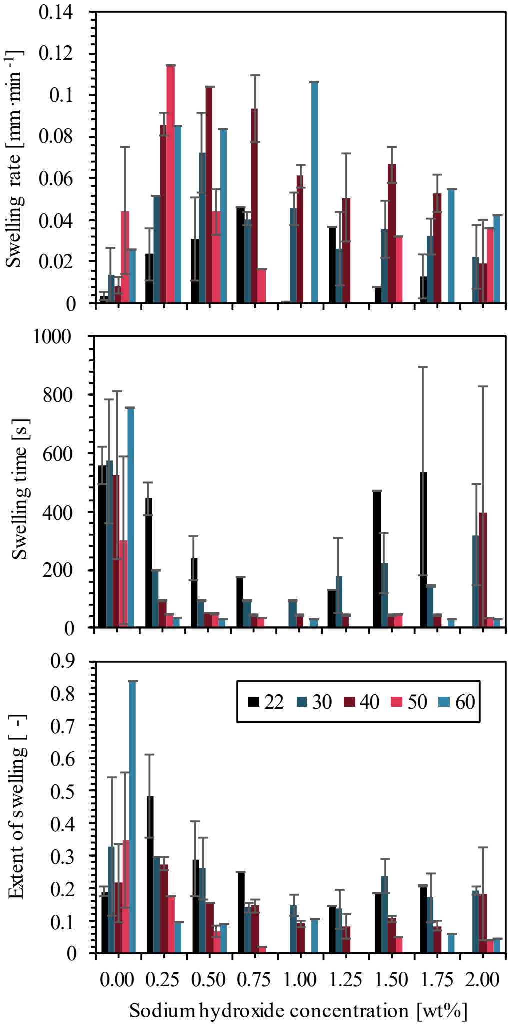

Fig. 12. Effect of cleaning parameters on the swelling in a diffusion-based setup, without an additional flow regime. Different colors indicate temperatures between 22 °C and 60 °C. If a column or an error bar is missing no data were available. Error bars indicate the deviation of at least two and up to five replicates.

Also, the sodium hydroxide concentration is varied between 0.0 wt%, meaning pure water, and 2.0 wt%. As the concentration increases from 0.0 wt% to 0.5 wt%, a high increase in swelling rate is detectable, as being suggested due to the fact, that around 0.5 wt% an optimal concentration for cleaning WPI is assumed [20] and swelling is a key process before dissolution [14]. With increasing concentration above 0.5 wt% the swelling rate decreases. This could have been advantageous for micro scale but accordingly the regarding swelling time increases with increasing concentration and thus with decreasing swelling rate. This also results in only small differences between the extents of swelling with varying sodium hydroxide concentrations. It could be assumed, that a sodium hydroxide concentration of 1.0 wt% has the smallest extent of swelling, as shown in Figure 12, and therefore could be considered as advantageous for cleaning in the micro scale. However, no significant trend exceeding the limits of deviation is detectable, resulting in the need for further investigation of the considered hypothetical correlation.

Flow-based impact of swelling

The effect of flow velocity on swelling and dissolution is considered for one parameter set of sodium hydroxide concentration and cleaning solution temperature. With an increase in flow velocity the swelling rate increases, as shown in Figure 13. Regarding the change from no flow to a flow velocity of 0.03 ms^{-1}, it is not following an expected linear pattern. This could be due to the dissolution setup not being operated in micro scale, meaning having a comparably endless amount of cleaning solution volume available, thus excluding scaling effects.

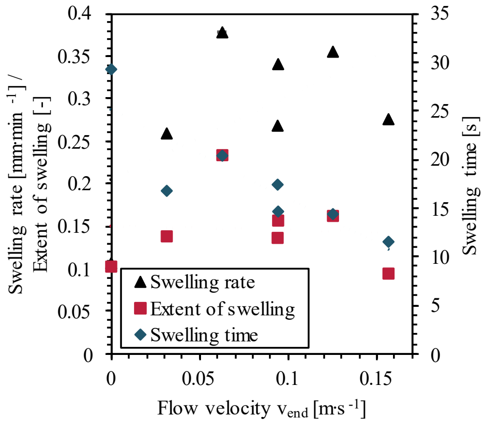

Fig. 13. Differences in swelling parameters under flow conditions. Constant cleaning parameters c_{NaOH} = 1.0 wt%, ϑ = (57.25 ± 1.26) °C and channel configurations $h_{ch,end}$ = (0.75 ± 0.01) mm and w = 18 mm.

As a first approximation the swelling time decreases by a linear trend with increasing flow velocity. An increasing flow velocity is typically beneficial for WP-dissolution and therefore the overall cleaning process, which leads not only to a decreased cleaning time, but also to a decreased swelling time. The extent of swelling shows with increasing flow velocity only a non-significant relation, represented by the linear regression which indicates a slope nearby zero. Nevertheless, up to a velocity of 0.13 ms^{-1} it could be assumed that with the addition of a flow and an increase in flow velocity the extent of swelling increases as well. Even though these are mostly single trials, the shown results could lead to the conclusion that an

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increase of velocity causes a higher risk of blockage. However, further trials and replicates need to be done to break down the impact of flow velocity on swelling.

CONCLUSION

The investigation of the blocking phenomena in micro scale shows that partially blockage of flow channels can be detected during cleaning and can be cleaned. The results show that parameter settings usually effective for cleaning on a common macro scale, like higher flow velocity or higher temperature and ideal cleaning agent concentration, actually hinder the cleaning process for micro components. This is particularly true when the initial channel height is smaller than the potential swelling height of the deposit. For micro scale cleaning a suitable increase in the cleaning parameters of the Sinner's circle known from the macro scale obstructs or even prevents the cleaning process of the micro component, as the initial swelling and partial blockage of the micro-component prohibits effective cleaning. This leads to the conclusion that a new approach must be adopted when planning cleaning protocols for micro components. Consideration should be given to overcome the static Sinner's circle and, for example, using a variable cleaning agent concentration to provide the appropriate cleaning agent concentration for the particular cleaning stage. Moreover, the goal is to determine if a specific combination of cleaning solution concentration, temperature and flow velocity can minimize deposit swelling and, in turn, decrease the risk of partial blockage of the micro component during cleaning. The high potential for blockage in microchannels, attributed to their small dimensions, necessitates a thorough characterization of blocking behavior. This should involve analyzing the surface to volume ratio, comparison of the scalability between the macro and micro scale and determining the optimal time for cleaning micro components to prevent blockages during the process.

NOMENCLATURE

A	Surface, mm
c	Concentration, wt%
DoC	Degree of coverage, %
h	Height, mm
i	Normalized removal rate, s^{-1}
I	Area dep. norm. removal rate, $s^{-1}\cdot mm^{-2}$
$\dot{m}$	Mass flow, $g\cdot min^{-1}$
n	Quantity, -
R_C	Cleaning rate, $g\cdot m^{-2}\cdot s^{-1}$
s	Swelling rate, $mm\cdot min^{-1}$
s*	Extent of swelling, -
t	Time, s
v	Velocity, $m\cdot s^{-1}$
w	Width, mm
Δp	Pressure drop, mbar
ϑ	Temperature, °C
$\bar{v}$	Mean velocity, $m\cdot s^{-1}$

Subscript

0	Start time (zero)
ch	Channel
const	Constant
cp	Cleaned pixel
end	End time
g	Gel
max	Maximum
NaOH	Sodium hydroxide solution
ncp	Not cleaned pixel
norm	Normalized
s	Swelling
sp	Sample plate
sps	Sample plate soiled

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